DEVELOPMENT OF NEXT GENERATION SOLID LUBRICANTS

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ABSTRACT

Present generation precision thin-film solid lubricants include heritage products of MoS_2 and Lead (Pb) applied via physical vapour deposition (PVD). Whilst well established, there is a desire to further improve the tribological properties of these coatings and anticipate any potential future legislative restrictions on the availability of Pb-based lubricants.

Considering these technical and commercial requirements, ESTL has developed a family of next generation Mo-based solid lubricants, utilising a variety of dopants, interlayers, and production process updates. Tribological tests show significant improvements in vacuum life, and component (angular contact bearing) test campaigns demonstrate success and compatibility with appropriate self-lubricating cage materials.

1 SOLID LUBRICANTS FOR SPACE

Effective lubrication is essential for the long-life operation of mechanical interfaces, acting to minimise adhesion, friction, and wear of the contacting surfaces. This is typically achieved through the separation of surfaces in relative motion by imposing a third body with low resistance to shear such that the two surfaces do not encounter severe wear. This third body lubricating layer can consist of a variety of materials, including solid or fluid lubricants.

Space mechanisms are no different, requiring adequate lubrication to achieve high efficiency (i.e. low and repeatable friction) over their desired operational lifetimes. This becomes more pertinent as longer and more demanding missions produce ever greater requirements of space engineering. Lubricant selection commonly reduces to a simple choice between a low volatility fluid (oil or grease) and a thin film low-shear strength solid lubricant. In general, solid lubricants are selected when the following requirements are encountered.

- <u>Operation at high temperatures</u> – Solid lubricants such as Physical Vapour Deposition (PVD) MoS_2 are capable of operating at elevated temperatures (≥260°C), together with appropriate cage materials in angular contact ball bearings [1]. Such temperatures would preclude the use of fluid lubricants due to their increased vapour pressures and evaporation rates.
- <u>Operation at low temperatures</u> – PVD MoS_2 can also operate at cryogenic (≤20K) temperatures [2]. Fluid lubricants are precluded at these temperatures due to the viscosity-related torque increases, which become prevalent once the temperature drops much below 0°C.
- <u>Avoidance of contamination</u> – For applications with significant contamination risks (for example optical elements located close to lubricated components) the increased outgassing and evaporation of fluid lubricants can be a concern (with the risk increasing as the temperature of operation increases). In general solid lubricants display much lower volatilities, and so are safer to select in these instances.
- <u>Accelerated testing</u> – Solid lubricants are generally considered to be less susceptible to the influence of acceleration (or much easier to predict in the case of load increases), as the complications of specific film thickness (Λ) changes encountered for fluids are not applicable to solids. As a result significantly higher acceleration rates can be confidently achieved [3].

In practice the most common solid lubricants used for space are MoS_2 and Lead, deposited in controlled precision thin films (~1µm) via PVD techniques such as sputtering. These lubricants are presented in more detail below.

Other lubricants are occasionally employed, but much more infrequently. Silver-based coatings may see use where electrical resistance must be minimised, but their frictional behaviours at nominal temperatures are sub-optimal. Other coatings are available, such as WS_2- or DLC-based, but these are typically limited by shorter operational lifetimes and quality control issues (in the case of the former) [4], or lower TRL (in the case of the latter).

1.1 MoS_2 as a Space Lubricant

Molybdenum disulphide (MoS_2) is a Transition Metal Dichalcogenide (TMD) with a lamellar structure, where each layer consists of Mo atoms covalently bonded to S atoms, and each layer weakly bonded by van der Waals forces (Fig. 1). These weak forces yield low shear between layers and therefore low friction upon relative motion between contacting surfaces is maintained, in the absence of moisture. Production of MoS_2 has existing and established procedures and long heritage within the space mechanisms industry.

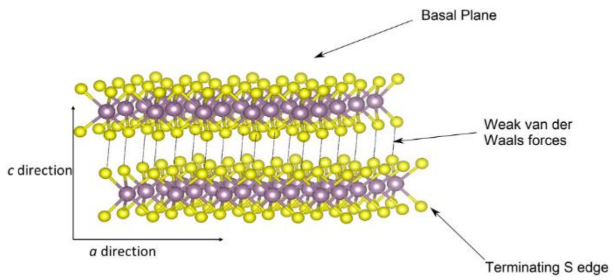

Figure 1. MoS_2 layer structure

The key strengths of present generation/conventional MoS_2 coatings are long operational life and exceptionally low coefficient of friction (CoF) under vacuum (<0.02 depending upon operating conditions), with low friction maintained over a wide range of temperatures (see above), translating to low bearing torque. However, when operated in laboratory air the coatings adsorb water, increasing their shear properties which in turn causes the friction to increase (by up to an order of magnitude). Furthermore, the coating oxidises and, as a result, wears more rapidly than would be the case in vacuum. Thus operation in air reduces the subsequent in-vacuum life of the coating [5] and, although some experimental evidence exists that a limited degree of moist air operation may not be detrimental to the coating (and in fact in some cases may be advantageous [6]), this behaviour has hitherto nearly always precluded the testing of components and mechanisms in air when lubricated by conventional PVD MoS_2 products.

1.2 Lead as a Space Lubricant

Lead provides low shear due to the high density of dislocations within its Face Centred Cubic (FCC) structure. This shear strength is somewhat higher than for MoS_2 however, resulting in ~2.5-3x higher torque at bearing level. Lead can provide exceptionally long lifetime in rolling bearing applications, particularly when used together with an appropriate lubricating cage. Like MoS_2 it is a mature product with significant heritage within the space mechanism industry. Lead is also generally more resilient to operation in a moist air environment, with the recommendation from ESTL being to not exceed 10^5 bearing revolutions in air at low speed.

However, a significant concern with Lead relates to its toxicity, and as a result it has been identified by REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals) legislation of EU and UK law as a Substance of Very High Concern (SVHC), in effect the first step to potential prohibition of lead within the EU and UK. Though prohibition, either in widespread use or for space is not yet imminent, it is considered by the REACH Lead Task Force that a reasonable worst-case (earliest) timeline will include a sunset date of 2028 [7].

2 NEXT GENERATION LUBRICANT REQUIREMENTS

ESTL has been a trusted supplier of PVD MoS_2 and Lead for the space mechanism (and terrestrial vacuum) industry for several decades, with ESTL lubricants being employed within many hundreds of flight applications achieving TRL9. However, given the above, present generations of both PVD MoS_2 and lead films might in some respects appear sub-optimal with respect to the evolving technical requirements and commercial needs of today's space mechanism industry.

For example, for some classes of mechanism which would have been typically fluid lubricated in the past (especially for commercial clients), the AIT costs and timescales associated with cage impregnation, application of creep barriers or anti-migration protocols for 1-g storage are problematic. In other cases, increased lubricant and cleanup solvent costs or hardware delta-qualification costs driven by lubricant formulation or solvent cleaning changes may be opportunities for change. Such AIT, environmental and qualification-driven issues might be avoided by selection of a robust, low torque noise and long-life solid lubricant solution

These requirements were reviewed via an anonymous targeted survey of (predominantly) European users of solid lubricants, including design authority organisations, bearing manufacturers, sub-assembly and component/equipment suppliers, plus a review of ESMATS papers and past ESTL test data. The key output from this review is presented in Fig. 2, in which the achieved lifetimes of angular contact bearing pairs (of different sizes and operating in different conditions) are plotted against contact stress, following the general stress-life relationship [8].

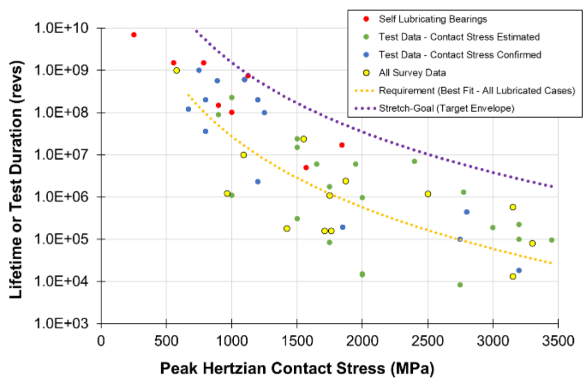

Figure 2. Life vs. stress of solid lubricant bearing applications

In addition, this survey highlighted how the market for solid lubricants may comprises broadly two types of customer.

- Performance-Driven Users – Those requiring high life and/or high stress
- Cost-Driven Users – Those requiring a low cost solution, with robust and proven products, ideally with reduced measures to protect the lubricant during AIT ensuring low cost.

These observations confirmed the need for improvement in the solid lubricant lifetime but clarified that increased cost would (generally) not be acceptable by the industry, and that alignment with existing processes and practices (i.e. minimal deviation from the existing flight-heritage ESTL coatings and processes) would be strongly desired.

Increased robustness during AIT is traditionally interpreted to mean the lubricant is less susceptible to increased wear when operated in moist air environments and maintains its ability to reach subsequent in vacuum life target without impact from any AIT operation in moist air. However, robustness can also be achieved by significantly increasing the available lifetime of the coating itself under vacuum (i.e. "headroom"), such that any reduction in life from elevated wear during AIT test in a moist environment is mitigated.

3 NOMENCLATURE OF DEVELOPED COATINGS

This paper presents the cumulation of multiple development activities into next generation solid lubricants performed by ESTL to achieve the above objectives, covering multiple individual development activities overseen by ESA.

Taken together, a total of 82 independent variants of thin film solid lubricant coatings were formulated, developed, deposited and tested (predominantly at tribometer level) by ESTL as part of these activities, in many cases under parallel iterative loops. Many coating formulations did not show tribological improvements over the existing heritage coatings, were deemed too expensive to be practical, or displayed other behaviours which excluded them from further study. Explored formulations included the use of chemical dopants, adhesion-improving interlayers, and a wide range of production process parameter changes (some minor, some considerable). For brevity, only the details of successful formulations (i.e. those viable for productisation) are presented here.

These selected coatings are assigned a three-digit designation, and this nomenclature is used throughout the remainder of this paper.

- MoS_2-101 – ESTL's flight heritage product employed on multiple TRL9 applications.
- MoS_2-201 – Identical composition to the heritage product, but ammended deposition process to improve coating performance.
- MoS_2-202 – Surface fuctionalised (SF) refinement of improved deposition process [9].
- MoS_2-301 – Improved formulation featuring antimiony trioxide (Sb_2O_3) dopants within the coating, plus ammended deposition process.
- MoS_2-302 – SF refinement of 301.
- MoS_2-402 – Tantalum (Ta) doped formulation, applied via surface functionalised improved process with metallic interlayers.
- MoS_2-502 – Non-metallic dopants introduced to fundamtentally modify the coating structure, applied via surface functionalised improved process.
- MoS_2-Star – Combination coating of multiple dopants (produced via a monolithic target), interlayers, and deposition process ammendments.

4 DEVELOPMENT CAMPAIGNS

Depositions of the above next generation coatings were produced used the same ESTL deposition facilities as our heritage product, including levels of cleanliness, sample preparation and inspection, and QA procedures (where applicable to the formulation itself). Where the deposition process itself was amended, geometric trials were performed using realistic components to ensure consistent and precision control of the applied coatings in an identical manner to the heritage product.

Physical assessments of the coatings were performed, including adhesion trials, morphological checks (including studies of amorphous and crystalline structures of the coatings), chemical structure and stoichiometric studies, thermal and humidity cycling, static load tests, and solvent compatibility trials, drawing from relevant ECSS standards where applicable. Production cost calculations were also performed, taking into account any all cost-non-neutral requirements (QA, hardware, maintenance etc.) for scaling-up of trial coatings to production level. Where technical performance or results of commercial assessments were unsatisfactory, the coatings were rejected for subsequent study.

4.1 Tribometer Test Campaigns

The tribological performance of the next generation coatings was assessed via a Pin-on-Disc (PoD) tribometer, operating under vacuum conditions (Fig. 4). Tests were performed under identical conditions to aid in comparison, with the Hertzian contact stress selected being quite high at 3.00GPa to reduce the duration of

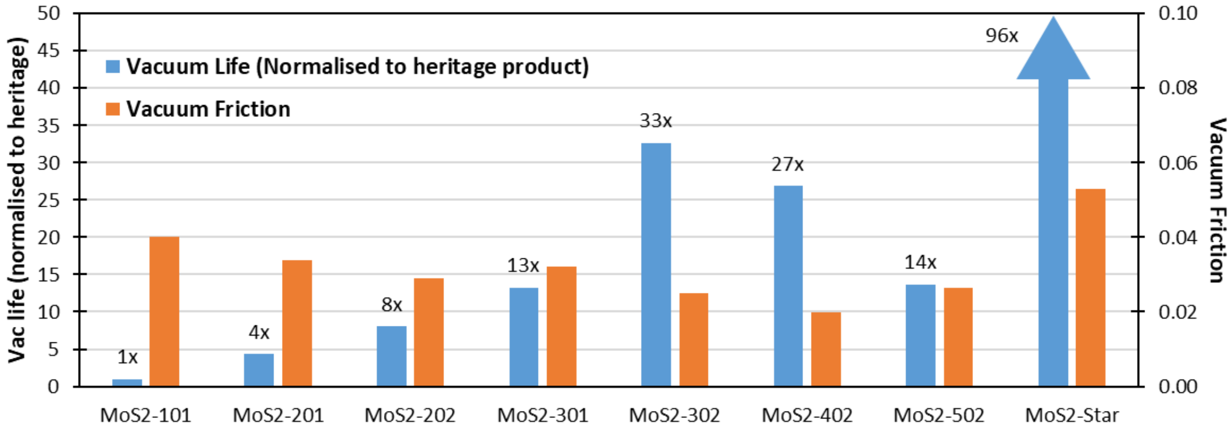

Figure 3. Comparative performance of next generation solid lubricants via vacuum PoD

each test to an acceptable level. Care was taken to ensure accurate results, including consideration and accommodation of known behaviours of PoD life test distributions, and maturation effects for Mo-based lubricants [10].

Figure 4. Vacuum PoD Tribometer at ESTL

Average in-vacuum coating lifetime and mid-life friction results from this extensive PoD test campaign are presented in Fig. 3, again with the comment that only the successful formations are included. Lifetimes are presented normalised against the heritage ESTL product to aid in comparison. These tribological results were very encouraging, with extensions in life demonstrated for next generation coatings, with no associated loss in the steady-state friction performance.

- The MoS_2-2xx variants showed a reasonable life increase over the heritage product, with no change in the applied coating itself (or anticipated production cost). The SF application process (MoS_2-202) demonstrated the best performance [9], and was selected for more detailed assessment at component level.
- The chemically doped coating varients (MoS_2-3xx, 402 & 502) all showed a further improvement over the 2xx variants, with the SF deposition improvement also being applicable to the Sb_2O_3 containing coating. These coatings were also selected for assessment at component level.
- MoS_2-Star provided exceptional performance, with the longest lifetimes seen in any tribological trials. However, the formulation of this coating also introduces a cost increase (due to the nature of the dopants included), which is likely acceptable only for those users demanding the very highest lifetime performance. Multiple additional campaigns were performed to identify and demonstrate low-cost methods for introducing these high-cost dopants into the coating, but all trials were unsuccessful in reproducing the exceptional performance achieved with the monolithic deposition target. As a result, though the proces is considered to be frozen, this coating formulation was not selected for subsequent component level trials and so should be considered as lower TRL. If a particularly high-demanding application is identified, MoS_2-Star could be adopted however.

Limited test campaigns were also performed using the Spiral Orbit Tribometer (SOT) on some coating variants, but these were intended only to demonstrate that the selected next generation coatings could be successfully applied onto balls as well as flats and were not comprehensive enough to allow a full comparison of performance against the heritage coating. Instead, these selected coatings were progressed onto trials at component level utilising angular contact bearings.

5 COMPONENT TESTING

Bearing tests were performed on pairs of FAG-7004C type bearings (~31mm PCD), with the balls and raceways lubricated with comparable and representative thicknesses of next generation solid lubricant. All bearings were fitted with a complementary inner race riding cage, manufactured from the recently developed TS8591 self-lubricating material [11]. All tests were performed at relatively high preload of 160 ± 5N, providing a peak Hertzian contact stress of 1210/1040 MPa at the inner/outer raceways respectively, and room temperature, unless otherwise stated.

Assessments were predominantly performed using the new Multi Chamber Rig (MCR) at ESTL, allowing for parallel testing of bearings under identical thermal vacuum conditions. Rotation speeds of 1750 rpm were used for long-running testing, with periodic slow speed “torque reversals” (several revs CCW and CW respectively) used to characterise the torque performance. Tests were halted once a significant increase in mean or peak torque was observed, characteristic of a transition from MoS_2-dominated lubrication to cage-dominated lubrication. Some selected testing was also performed using the Advanced Bearing Test Rig (ABTR) to confirm the in-situ film-thickness progression for the MoS_2-201, -202 and -502 variants during test, as well as some more detailed testing to characterise the behaviour of the coatings.

Figure 5. The Multi-Chamber-Rig (MCR) in operation in the ESTL cleanroom

5.1 MoS_2-202

ABTR testing of MoS_2-202 lubricated bearings (featuring TS8591 cages) was performed at a lower preload of 48 ± 3N (~840MPa peak at the inner raceway). Results showed low mean and peak torque of <1 and <2 mNm respectively achieved after ~5-million revolutions in vacuum, with this low torque maintained for the full 80-million revolutions of the test. Bearing shaft displacement was observed during this initial operation, attributed to a removal of ~50% of the available as-deposited MoS_2-202, with further slow shaft displacement observed during the remainder of the test supporting the argument that MoS_2-202 was still present within the contact after 80-million revolutions [9].

At the higher preload of 160N, a lifetime of 50-million revolutions was attained under vacuum, with a mean torque of 4mNm. Beyond this point the torque was elevated to ~15mNm, indicative of cage-dominated lubrication (i.e. not necessarily a failure of the MoS_2). This test was repeated with 10,000 and 100,000 revolutions in moist air prior to vacuum, with 36- and 33-million revolutions achieved respectively in vacuum before transition to cage-dominated lubrication.

One test was performed at -40°C (with TS8591 cages), achieving a mean torque of ~5mNm following 145-million revolutions at low temperature. Identical tests performed with Vespel-SP3 cages demonstrated elevated torques immediately upon cooling, attributed to the known adverse moisture-absorbing properties of the polyimide material cage, and not due to the MoS_2-202 coating [12].

A test at room temperature was performed utilising MoS_2-202 lubrication of the balls only (i.e. no lubricant applied to the raceways of the bearings), plus a TS8591 cage. This test again was highly successful, with a mean torque of ~3mNm maintained for ~75-million revolutions before an increase in torque to a cage-dominated regime (Fig. 6).

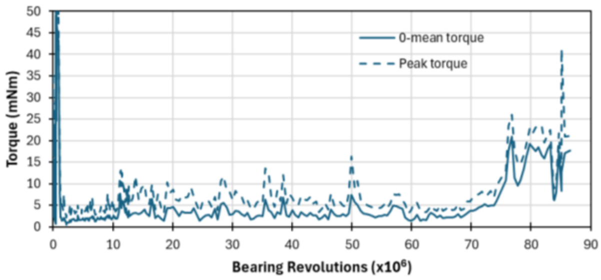

Figure 6. Vacuum torque behaviour of bearing pair lubricated by MoS_2-202 applied only to bearing balls

Taken together results demonstrate the capability of MoS_2-202 to provide long life even at high contact stress and following some considerable period of in-air operation.

5.2 MoS_2-301 / MoS_2-302

The behaviour of MoS_2-301 at bearing level was assessed using the ABTR, with bearings preloaded to 48 ± 3N (~840MPa peak at the inner raceway). and fitted with TS8591 cages. Results revealed low mean torque <2mNm for 90-million revolutions before a transition to cage-dominated lubrication (also indicated by a rate change in the shaft displacement behaviour) [13].

MoS_2-302 (applied to balls and raceways) was assessed at a higher preload of 160N (~1210MPa peak at the inner raceway), providing torque of 1mNm for 30-million revolutions under vacuum. This was followed by a transition period, where the mean torque increased to

~9mNm, which was maintained for a subsequent 100-million revolutions (Fig. 7).

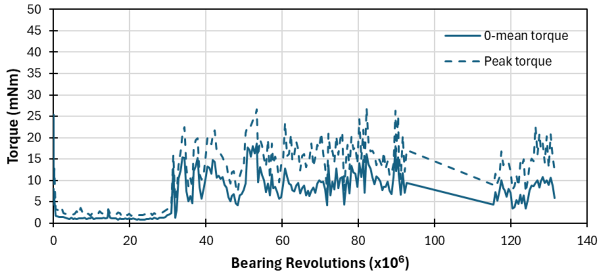

Figure 7. Vacuum torque behaviour of MoS_2-302 lubricated bearing pair

5.3 MoS_2-402

Testing is ongoing on bearings lubricated with MoS_2-402. Results will be presented once these tests conclude.

5.4 MoS_2-502

ABTR testing of MoS_2-502 (also at ~1210MPa peak Hertzian contact stress at the inner raceway) showed effectively no impact of operation over the temperature range -60 to +80°C, and consistent torque behaviour up to 2000 rpm (with elevated torque noise at higher speeds likely due to rig resonance issues, rather than being indicative of the coating itself). Moist air dwell tests showed no significant detrimental impact on the subsequent behaviour of the lubricant coating, with low torque obtained within a few hundred revolutions once motion was recommenced, increasing with increased dwell time. Limited operation in moist air up to 160,000 revolutions at 500 rpm resulted in elevated torque, but low torque was re-established when returning to vacuum after 500-5000 revolutions (depending upon the in-air actuation duration).

MCR testing was performed on bearings (at ~1210MPa peak at the inner raceway) lubricated with MoS_2-502 applied to the balls and raceways, with two identical tests achieving ~70 and ~90-million revolutions at 3-4 mNm mean torque, before a rapid increase indicative of transition to cage-dominated lubrication. Tests were also performed whereby the lubricated bearing pair was operated in moist air (55 ± 10% RH) under the same preload for 100,000 revolutions prior to the vacuum lifetest. Under these conditions one bearing pair showed a transition to cage lubrication after ~70-million revolutions, whilst an identical test was halted at 140-million revolutions without showing clear evidence of transition (Fig. 8).

These results are very encouraging and suggest that some significant period of in-air running of MoS_2-502 is possible whilst still achieving long life at low torque under vacuum, potentially due to the increased "headroom" effect described above.

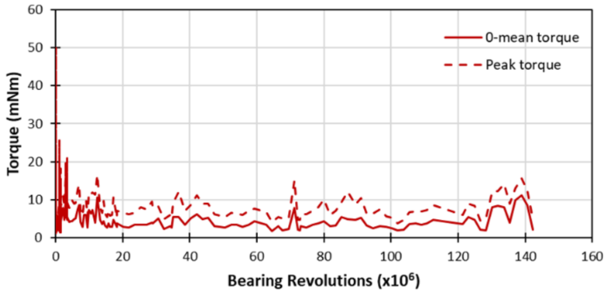

Figure 8. Vacuum torque behaviour of MoS_2-502 lubricated bearing pair following 100k revs in-air

5.5 Post-Test Inspections

Post-test inspections were performed of all bearings, paying particular attention to the condition of the lubricant and any produced transfer film, in compliance with ECSS requirements for Qualification Testing Success Criteria [14].

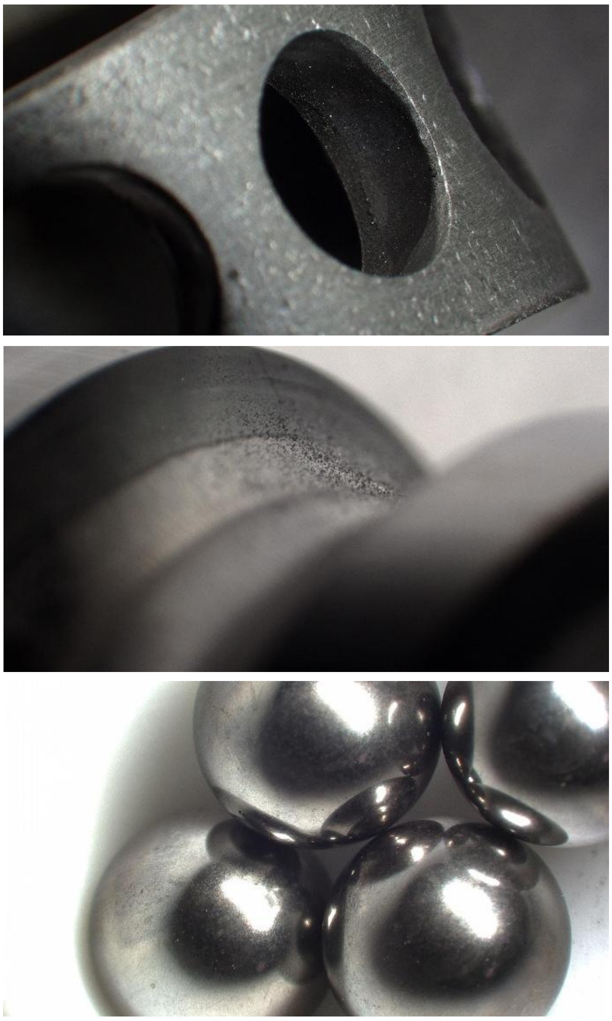

Figure 9. Example cage wear, transferred cage material on inner raceway, and balls following 140-million revolutions (MoS_2-502 test)

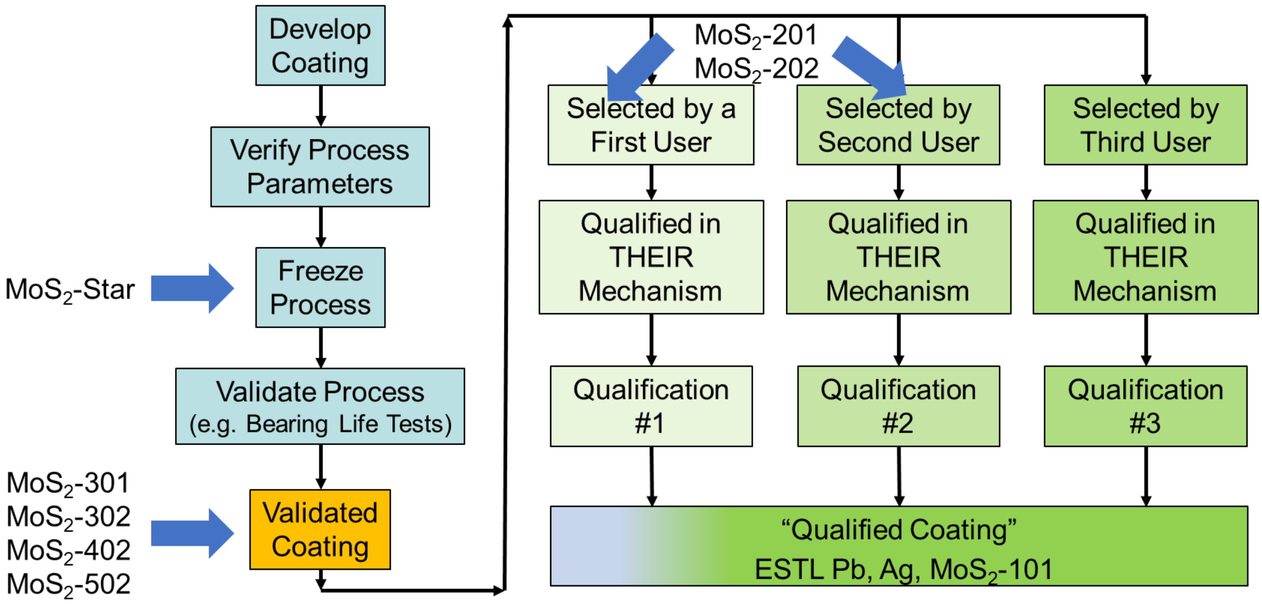

Figure 10. Progression of next generation coatings through verification and validation to inclusion in mechanism qualification

In all cases the bearings appeared good, with some dusting of lubricating material across the contacting elements of the bearings, evidence of transferred cage material, and no evidence of unacceptable steel wear. Some wear was observed within the cage pockets, but this is necessary for successful lubrication via the double transfer mechanism [15], and was deemed to be of an acceptable level given the duration of the test.

These inspections confirmed that the bearings had transitioned to a TS8591-dominated lubrication regime, and suggest that, should the elevated torque and torque noise be acceptable for an application (recalling the relatively high preload used for these bearing tests), extended duration of these next-generation lubricated bearing is entirely possible.

6 CONCLUSIONS AND NEXT STEPS

ESTL has successfully identified, formulated, developed, and demonstrated a next generation of precision thin-film solid lubricants for space and vacuum applications, offering significant improvements over its own current heritage products, without compromising on friction coefficient. Angular contact ball bearing tests were successful, and the coatings have been shown to be appropriate for use with the newly developed SLM cage material TS8591. Long lifetimes were achieved in vacuum at high stress, and in many instances successful vacuum operation following considerable periods of moist-air running were demonstrated.

Following these successful test campaigns, ESTL is now able to offer these next-generation coatings to the space mechanisms and vacuum industry communities.

6.1 Effective TRL of Developed Coatings

Formally according to ECSS, a lubricant is treated as a material (i.e. *raw, semi–finished or finished substance (gaseous, liquid, solid) of given characteristics from which processing into a component or part is undertaken* [16]) and therefore cannot be progressed to the highest TRL (i.e. flight qualified), as these are validated only, and only the employing mechanism can be qualified to higher TRL.

MoS_2-201 and -202 have already begun to see the first applications for space and terrestrial vacuum users (though at the time of writing these coatings are being employed in various qualification programmes and have not yet flown). MoS_2-402 has been baselined as the preferred lubricant for a European mechanism development project, but this has not yet progressed to qualification testing.

Taking these scenarios into account, the effective TRL (i.e. gaining TRL from the employing application) presented below can be considered for the next generation coatings presented within this paper (Tab. 1). All opportunities to further develop the TRL of these next generation coatings through selection and implementation in appropriate mechanism hardware should be taken.

Table 1. Effective TRL of next generation coatings

Coating	Vacuum life extension (PoD)	Effective TRL
MoS_2-101	--	9
MoS_2-201	4x	6/7
MoS_2-202	8x	6/7
MoS_2-301	13x	3/4
MoS_2-302	33x	5
MoS_2-402	27x	5
MoS_2-502	14x	5
MoS_2-Star	96x	3/4

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