POST TEST INSPECTION OF LUBRICANTS – GUIDELINES AND LESSONS LEARNT

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ABSTRACT

For a mechanisms development / qualification campaign to be successful, ECSS mandates a post-test assessment and health check of the tribological surfaces. However, the defined success criteria are somewhat non-quantifiable and require a degree of subjectivity in their assessment. This paper discusses the common failure modes of space mechanism lubricants and presents experimentally verified techniques for health assessments of these lubricants, providing guidance and support to the mechanism engineer, enabling more objective post-test inspections.

1 ECSS DEFINITION OF FAILURE AND SUCCESS

The general concept of failure has multiple meanings, ranging from operable but unsatisfactory performance to complete loss of function / inoperability. Given that it is completely possible that "unsatisfactory performance" as defined in a certain application might be deemed entirely acceptable to another, the concept and definition of failure (and success) is considered as a system property.

For a spacecraft mechanism undertaking a qualification campaign, ECSS provides guidelines as to this definition. This includes a requirement for a disassembly and inspection of the tribological elements of the mechanism, and verification of the status of the lubricants and contacting surfaces, with respect to the identified success criteria (Section 4.8.3.3.17 within [1]). Essentially, whilst ECSS fundamentally pertains to the mechanism itself, the success of the mechanism requires that the lubricant is identified to be functional at the end of the test. However, this presents an immediate difficulty for the mechanism engineer as these criteria require, at best, some degree of subjectivity in their assessment, and at worst cannot be demonstrably achieved. Examples include;

- "***No direct contact*** *between metallic parts in relative motion identified in the interface of solid lubricated contact surfaces*", is not practically achievable from a tribological viewpoint as some degree of metallic contact and wear is always present, even when the coefficient of friction (CoF) is low.
- "***No chemical deterioration*** *beyond the* ***specified limits*** *of fluid lubricants*" and "***The amount and size of wear products*** *conforms to contamination and overall mechanism* ***performance requirements specified***", requires a specification and quantification of acceptable degradation and wear limits, beyond which is practically definable.
- "*Deterioration torque or force performance is less than or equal to 50% of the values measured at the* ***beginning of qualification tests***", does not adequately define what is meant by the beginning of the test, and takes no consideration of run-in, or environmentally dependent lubricant behaviours.

It could (perhaps provocatively) be argued that it is impossible for ANY qualification campaign to fully comply with the ECSS requirements for test success, exactly as presented, without some degree of subjectivity. It is also noted that this scenario is not unique to ECSS, with similar ambiguity existing in the parallel NASA documentation for qualification testing which requests "*successful tests will not have any anomalous conditions such as abnormal wear, significant lubrication breakdown, or excessive debris generation*" [2].

Whilst early detection and non-destructive techniques do exist for the assessment of lubricant health, including torque-based methodologies [3], these techniques are somewhat speculative in nature and are yet to be proved or employed at application level. They certainly are not mature enough to provide a replacement for the requirement of a post-test strip down and inspection of the lubricant and contacting surfaces. Instead, the post-test inspection of tribological surfaces should be done in such a manner, and using appropriate methodologies, to allow for as meaningful and quantifiable assessment as practicable possible, and the degree of subjectivity employed should be as little as possible.

1.1 Other Definitions

Beyond the ECSS definition of qualification success, intrinsically linked to lubricant life and status), the term failure may be applied to a range of other conditions encountered during the mechanism development and qualification campaign, including:

- Unacceptable or unpredictable torque noise, or an increase in the mean- to peak-torque ratio

(often indicative of a change in the lubricant behaviour).

- The onset of abrasive, lumpy debris during material wear testing, often with similarly poor definitions of acceptability.
- Sustained elevation of the cofficient of friction, often used in tribometer tests. Typically a failure criteria of CoF $\geq$ 0.30 is used, which is close to the value at which the maximum shear forces will reach the surfaces of a contact resulting in plastic deformation, therefore providing a meaningful definition of the failure of the lubricant.
- Fatigue failure in bearing applications. This is rarely (if ever) seen in space applications as fatigue typically occurs significantly beyond the lifetime of the lubricant itself.

These definitions may have parallels with the ECSS post-test inspection criteria, and consideration of the potential lubricant failure modes and behaviours should be made when performing the post-test strip down and inspection.

1.2 Extension of Life

As the use of spacecraft in telecom, Earth Observation (EO), navigation and defence applications becomes more prevalent there is a driving need to extend mission or service life, often at minimal cost. An attractive route to achieve this is through the extension of the qualified lifetime of existing developed hardware, and demonstration of success of the critical tribological elements when subjected to extended operation. As such there is great desire to identify the magnitude by which a lubricant has passed a defined qualification campaign and demonstrate (ideally quantifiably) the degree of degradation/health of a lubricant, beyond the mere identification of acceptance required by ECSS.

This paper will present the common root causes of lubricant degradation leading to spacecraft mechanism failure, and the post-test methodologies which may be employed to investigate them, with a focus on quantifiable techniques which require reduced subjectivity (where possible). Discussions are separated by lubricant type.

2 FLUID LUBRICANTS

Fluid lubricants for space applications are typically selected for their long chain polymers and subsequent low volatility in a vacuum environment. Whilst an understanding of lubricant loss via evaporation can be calculated from the operating parameters of the application (albeit conservatively [4]), such long chain polymers are also highly susceptible to degradation under shear. Shear forces within the tribological contact will act to break down the polymer chains, both increasing the volatility of the fluid (resulting in some identifiable species), and increasing the viscosity of the fluid. This shear-induced tribo-chemical fluid degradation is reasonably well explored for both families of space lubricants; PFPEs [5] and MACs [6], and is dependent upon a multitude of external factors.

For PFPEs, this rate of degradation is accelerated by the presence of reactive elements within the contact (and similarly combated by the removal or passivation of these elements), known as Lewis acids. Concerns have also been raised regarding auto-catalytic degradation of the lubricant, whereby some degree of initial operation results in formation of catalysing compounds within the fluid, which may theoretically act to degrade the lubricant during a subsequent dwell period [7] (although experimental evidence of this behaviour is very limited). The degradation rate of MAC fluids is slower than for PFPEs, due in part to the lack of a Lewis acid during their breakdown [8].

Regardless of the root cause, the consequence of this degradation of the fluid lubricant is a rapid increase in CoF, often in a distinct “hockey-stick” shape (Fig. 1), resulting in an increase in torque or reduction in gear efficiency and potential substrate wear at application level. This CoF increase is also often accompanied by a temperature increase, further exacerbating the rate of lubricant degradation.

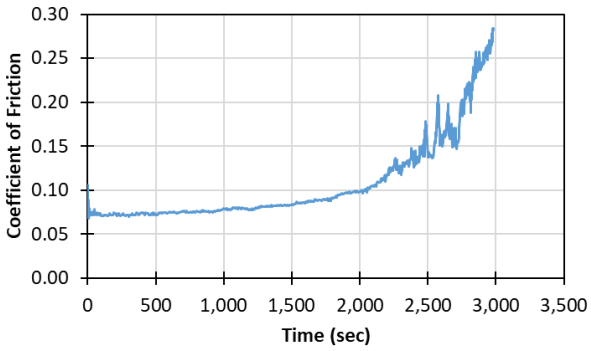

Figure 1. Typical CoF progression of fluid lubricant

The degraded fluid lubricant will look discoloured, often darker brown or black in colour, typically more viscous or dried out, and potentially more crystalline (Fig. 2). However it can be hard to visually interpret the extent of degradation, and can be difficult to differentiate between a dry or thicker lubricant as a result of evaporation / separation (particularly where elevated temperatures have been experienced), as a result of tribo-chemical degradation, or as a result of a high volume of metallic debris within the fluid (or a mixture of all three). In general, a visual inspection may be good in identifying that something has occurred, but poor at quantifying the magnitude of the degradation, its cause, or how much potential life remains in the fluid.

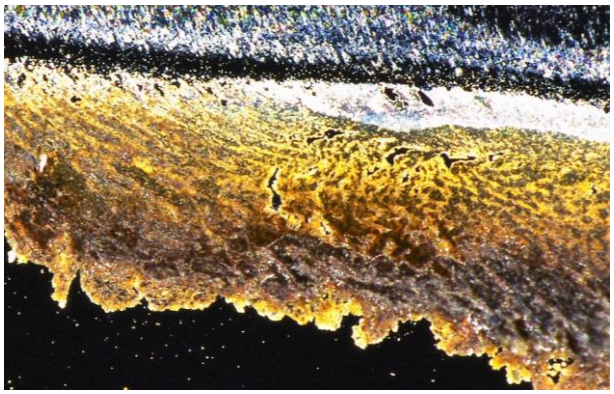

Figure 2. Degraded MAC lubricant

One proven methodology for such assessment is to extract a small amount of oil or grease from the mechanism application and test the lubricant using the Spiral Orbit Tribometer (SOT), comparing the attained lifetime against fresh undegraded samples. Should the sampled lubricant show a statistically significant early failure in comparison to the control, it can be reasonable stated that the sample has degraded. This methodology can also be used to provide some understanding of the magnitude of degradation.

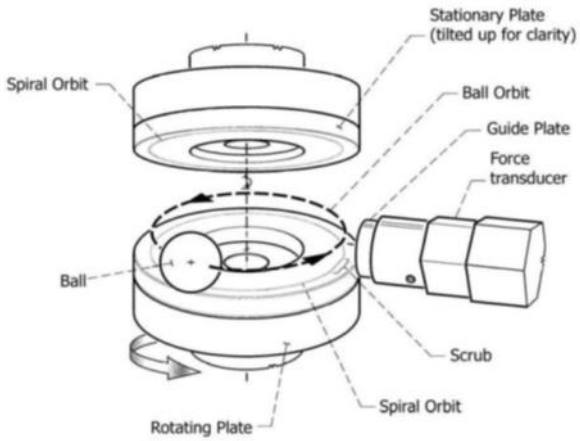

Figure 3. Spiral Orbit Tribometer (SOT)

2.1 ASAP-IMS Mass Spectrometry

Due to their molecular complexity and relationship between molecular structure and physical properties, a preferred method of post-test assessment of space fluid lubricants is the coupling of an Atmospheric Solids Analysis Probe (ASAP) ionisation source, together with High Mass Ion Mobility Spectrometry (HM-IMS). This technology allows for rapid characterisation of a fluid lubricant sample over a large m/z range (typically 1250), allowing for identification of initial degradation products (expected to be quite large in m/z), in both the positive and negative ion spectra. Lubricants are typically extracted from the application via a capillary tube and inserted directly into the mass spectrometer, with only minute quantities of lubricant required. ESTL has recently acquired such an assessment tool in the form of a RADIAN ASAP, allowing for fingerprinting of fresh and degraded lubricant to be performed.

Figure 4. RADIAN ASAP

This methodology was experimentally assessed using multiple PFPE lubricant sets;

- Fresh “undegraded” lubricant.
- Thermally degraded lubricant, exposed to >9 months of baking at 140°C under vacuum whilst mixed with catalysing FeF_3 powder.
- Grease extracted from a client’s APM actuator support bearings. These bearings had completed their life test of ~145-million revolutions, and post-test optical examinations had determined that the front bearing appeared to have degraded lubricant but was still able to rotate quite smoothly by hand (Fig. 5). The rear bearing also appeared to contain degraded lubricant; however, when rotated manually the bearing appeared “notchy” suggesting a greater level of lubricant failure.
- Grease extracted from the gearbox of ESR Technology’s Motorised Umbilical Separation Device (MUSD) [9] following the 3.5-million revolution lifetest. The gearbox was not disassembled, allowing for demonstration of the cabability to extract lubricant from a complex mechanism without the need for disassembly.

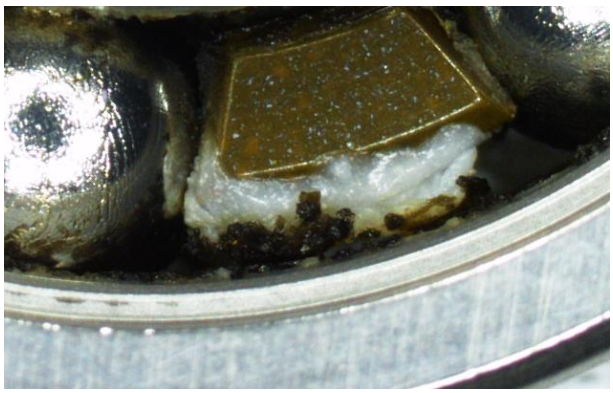

Figure 5. APM actuator front support bearings with discoloured PFPE grease

MAC lubricants were also assessed but for brevity are not reported here.

Results showed clear and repeatable evidence of fluid lubricant degradation, with key species identified in the positive and negative ion spectra (Fig. 6), and these markers being the same for the thermally, and tribo-chemically degraded fluid [10]. These species were more pronounced in the more degraded fluid samples.

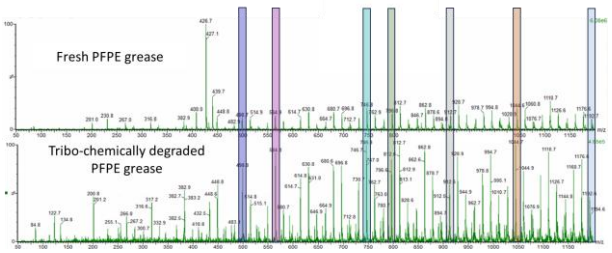

Figure 6. Negative ion spectra of PFPE greases assessed via RADIAN ASAP spectrometry

Furthermore, through the application of statistical comparison of key degradation elements (Principal Component Analysis (PCA)), a progression of the lubricant from fresh through to fully degraded can be plotted. This observation suggests a methodology by which a lubricant sample can be assessed using the RADIAN ASAP and mapped along the progressive scale of degradation to provide analysis of the health of the lubricant (Fig. 7). An activity to quantify this progression, via periodic sampling of a fluid lubricated angular contact bearing test operating under boundary conditions, is currently underway at ESTL.

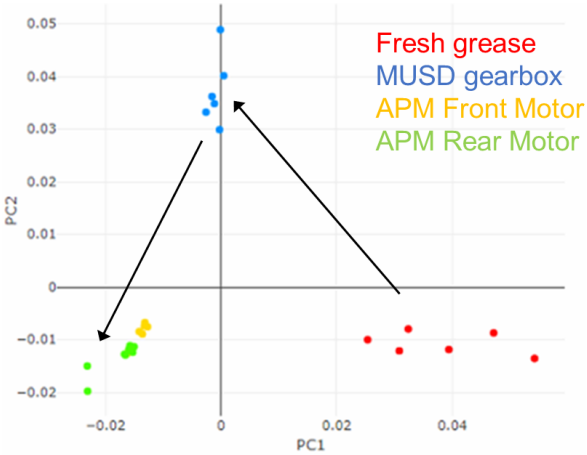

Figure 7. Negative ion PCA showing lubricant degradation progression

2.2 Other Fluid Lubricant Analysis Tools

Other chemical analysis tools are also occasionally proposed to identify lubricant degradation which, although useful, display some limitations.

Fourier-Transform Infrared Spectroscopy (FTIR) can be used to consider the status of a lubricant at the end of life, but it requires more lubricant than can easily be non-intrusively extracted from a typical space component. In addition, FTIR analysis of degraded PFPE oils typically identifies only the presence of a carbonyl group at a wavenumber in the region 1800-1900cm^{-1} [2]. Due to the singular presence of this marker, FTIR analysis runs the risk of being incorrect.

Residual Gas Analysis (RGA) is commonly used as an in-situ tool for the identification and analysis of volatile gasses during fluid lubricant degradation, commonly employed during tribometer-level tests [7]. However, it is generally inapplicable as a post-test inspection tool and, with a typical maximum m/z of 200, limited in the range of degradation products it can detect.

Gel permeation chromatography (GPC) measures the diffusion of molecules through a medium to determine the molecular size distribution. Whilst theoretically useful for identifying degraded components within a fluid, the volumes required for the test are impractical as an in-situ post-test inspection tool.

X-ray Photoelectric Spectroscopy (XPS) is an analysis tool which provides information on the top ~2nm of a sample surface, including contaminant layers, oxides, carbon, and sulphides. Such contaminants can be misleading as they are not the products of tribological interactions, and as such most XPS instruments are fitted with an ion-etch facility which can be used to remove post-test contaminants thus exposing the true tribological surface. Whilst not applicable for assessments of the lubricant itself, this technique has been employed to determine the presence of catalysing elements (FeF_3) for PFPE oil degradation on steel surfaces [11].

Ultrasonic reflectometry can be used to measure film thickness (and therefore depletion) in-situ and, via formation of a quasi-static standing wave within the fluid, an in-situ viscosity measurement can also theoretically be made [12]. Successful trials of ultrasonic inspection of fluid films on space oils have been performed at tribometer level by ESTL utilising a 400 x 800µm sensor [13], but have not been implemented further.

Atomic Force Microscopy (AFM) involves the use of a tapped or rastered probe across a dry or fluid medium. This probe can be controlled to apply a shearing force and used to assess the mechanical and micro-viscosity changes within a lubricant as an indication of degradation [14], though to date there have been no application trials of this technique to space oils.

3 THIN FILM SOLID LUBRICANTS

Thin film solid lubricants are often selected for applications where the use of a fluid is precluded, either due to application temperature ranges, contamination concerns, or to allow for accelerated testing. Typically, these include MoS_2 or Lead (Pb), applied via Physical Vapour Deposition (PVD) onto the tribological elements of the mechanism, such as the balls and raceways of the bearings.

Failure of these lubricants is generally attributed to wear and removal of the material via the action of shearing, with this wear rate dependent upon several factors (including, but not limited to, contact stress, temperature, surface roughness, substrate hardness, and operational environment). Therefore, the most intuitive post-test assessment methodology would be an inspection of the surface to identify the presence, and extent, of the lubricating film in the contacts. Visual / microscope inspections can be very misleading in this manner, as it is often extremely difficult to optically identify and quantify the presence of a thin film, and so some chemical identification tool is preferred.

3.1 X-ray Fluorescence (XRF)

XRF is used extensively for quantitative analysis of solid film thickness and elemental analysis. Although the technique does not have imaging capabilities, it is rapid, non-destructive, and highly accurate with the intensity of the signal proportional to the number of atoms within the assessed area. The technique can detect Mo and S elements within a coating (including deconvoluting between these elements), as well as Lead, but is not capable of detecting elements such as Sodium (Na), Aluminium (Al) or Silicon (Si) below 16 amu. Careful calibration of the XRF against a controlled sample is required for quantified thickness measurements and, for MoS_2, care must be taken to ensure the ratio of Mo to S within the coating matches that within the calibration standard, including any Mo present within the substrate. Spot sizes to 50 x 50µm can individually assessed, allowing for a coarse mapping of the coating thickness over the surface of a tribological component.

This methodology was trialled at ESTL via a Pin-on-Disc (PoD) test campaign, where PVD MoS_2 was initially deposited onto 52100 steel substrate discs [15]. Coatings were then sheared under vacuum for varying magnitudes of operation; some halted once the lubricant had fully failed (identified by a rapid increase in CoF), some halted once the CoF had begun to display elevated noise, and the remainder of tests halted whilst the CoF still remained low and steady. On completion of each test the thickness of MoS_2 remaining within the wear track was assessed over a minimum of four independent XRF scans (Fig. 8).

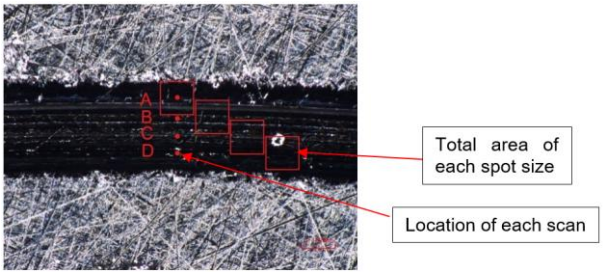

Figure 8. Wear track and XRF scan regions on MoS_2 lubricated PoD disc post-test

This XRF study showed removal of the MoS_2 from the contact, with the proportion of MoS_2 remaining (from the original applied thickness determined from outside the wear track) being lowest for tests displaying a CoF increase. A minimum thickness of 9% MoS_2 was measured in this region, suggesting a threshold below which the MoS_2 can no longer adequately protect against steel-on-steel asperity contact and wear (Fig. 9). Higher thicknesses were observed on the edges of the wear tracks, showing the displaced lubricant accumulating away from the contact zone where it can no longer be replenished.

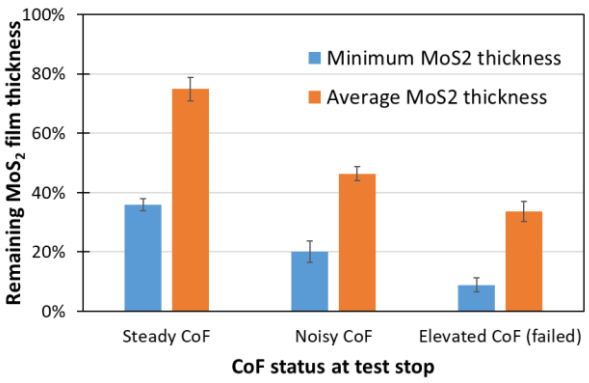

Figure 9. Minimum and average MoS_2 thicknesses measured from PoD wear scars

A complementary campaign was performed utilising MoS_2 lubricated test balls, rolling against unlubricated 440C under vacuum on the SOT. During these tests the ball was periodically removed from the SOT, and the MoS_2 film thickness assessed via XRF [16]. Results demonstrated that the initial thickness of applied MoS_2 was very quickly depleted in all cases, with the majority of operation being achieved on a significantly thinner layer (Fig. 10.). This rate of MoS_2 removal was observed to be dependent upon contact stress, but rather independent of surface roughness (within the range assessed) (Fig. 11). It is proposed that the rate of MoS_2 removal, and the threshold thickness below which the coating can no longer provide adequate lubrication, is a system property of the contact in question.

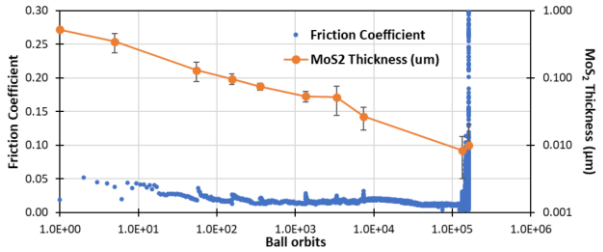

Figure 10. MoS_2 thickness progression on the SOT

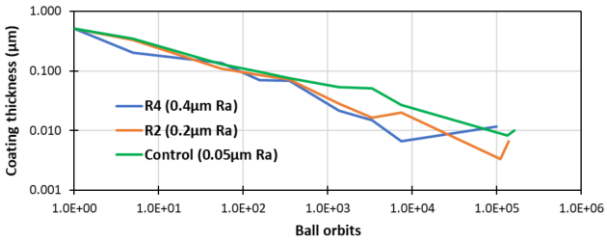

Figure 11. MoS_2 thickness progression as a factor of surface roughness

Although not explicitly studied in such detail, it is proposed that a similar scenario exists for lead lubrication, where extended SOT lifetimes have been observed for coatings measured to be <0.01µm on the originally lubricated ball [17].

3.2 Other Solid Lubricant Analysis Tools

Although XRF measurement of the remaining coating thickness is a preferred methodology for assessment of solid lubricants, other techniques should also be considered.

Scanning Electron Microscopy (SEM) is a useful inspection technique where information on the morphology, distribution, and wear of a lubricant film can be explored. Typically coupled with Electron Dispersive X-ray spectroscopy (EDX) this provides the ability to measure and map the elemental make-up of the contact where scuffing or adhesive wear has taken place and visualise the distribution of lubricant. However, SEM-EDX is generally considered to be non-quantitative (though semi-quantitative resolution of Mo and S peaks are possible), and the physical restrictions of the facility often necessitate destructive sectioning of the components.

Profilometry (contacting and non-contacting) can also be used to build a picture of the tribological surfaces in question. These are useful in identifying the onset of micro-polishing or wear of a metallic substrate, indicative of lubricant failure, and can be used to provide context into the magnitude of "acceptable" wear according to ECSS [1]. If higher resolution is required, AFM may also be considered.

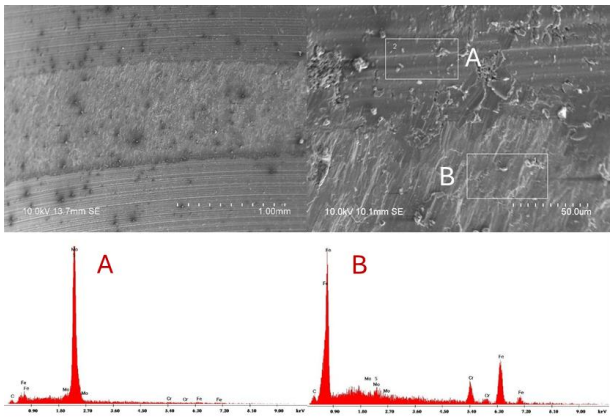

Figure 12. MoS_2 wear track under SEM and EDX

Raman Spectroscopy is an analysis tool used to differentiate structures containing the same atoms but in different arrangements. For MoS_2 this allows for non-destructive identification of crystalline and amorphous structures within the coating, with the general observation that the former provides significantly longer operational lifetime of the coating, as well as information on dopants, contaminants, and the density of the coating. Tribological trials performed by ESTL demonstrated that towards the end of life the crystalline MoS_2 structure reorientates itself somewhat to be more amorphous [15], but this behaviour cannot readily be quantified into a post-test methodology and is restricted by the fact that Raman struggles to provide accurate information on coating films <0.1µm. Therefore, Raman is more applicable as a quality control method during the application process, rather than a post-test investigation tool. Raman cannot be applied to pure metals such as Lead. X-Ray Diffraction (XRD) offers similar capabilities to analyse the crystalline structure of MoS_2 but, with a typical sampling depth of 1-2µm, is likely not applicable to post-test coatings which are considerably thinner.

Direct measurements of adhesion are occasionally requested as part of the specification plan for the development of new lubricants, either taking the form of a gross tape-pull test, or a more detailed scratch test. Opinions differ on the benefit of adhesion testing as anything more than an indicator of bulk failure of the deposited coating, and there is no strong correlation between the critical load of a coating as measured in a typical scratch test and its tribological performance. As such scratch testing is not considered an appropriate post-test analysis tool.

One common failure mode for MoS_2 is excessive operation in a moist air environment, resulting in the elevated formation of MoO_3 on the surface, which produces a higher wear rate and depletion of the applied layer. It is occasionally proposed to characterise the magnitude of this layer via some chemical analysis tool

such as XPS. Whilst technically possible, analysis of transition metals (such as Molybdenum) is extremely challenging via XPS due to the wide range of oxidation states, requiring reference data in order to accurately deconvolute similar peaks [18]. If unknown compounds or oxidation states are present, inappropriate peak fitting can lead to inaccurate quantification

4 TRANSFERRED SOLID LUBRICANTS

The discussion above pertains mainly to failure of the original as-deposited lubricant film. In practice the failure of many long-life lubricated components (for example an angular contact ball bearing fitted with a self-lubricating cage) may not be solely related to the loss of the original coating. Instead, the failure of the bearing may be related to the status of the transferred film, deposited onto the balls and raceways via the double transfer mechanism (Fig. 13). Self-Lubricating Materials (SLMs) are also commonly used in bushing and gear applications, though here lubrication is not necessarily provided by a transferred film and instead may simply be a consequence of the reduced CoF resulting from the polymer material in relative motion to a harder metallic substrate.

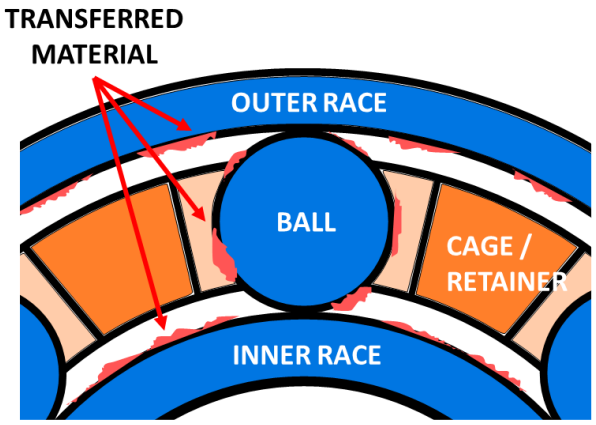

Figure 13. Double transfer mechanism for SLM cages

Definition of failure in cases utilising SLM lubrication can be difficult, as bearings lubricated in this way can operate for 10^8 to 10^9 revolutions whilst still displaying acceptable torques [19]. Where failure does happen, gross or local depletion of the SLM transfer film may occur such that the asperity contacts are not sufficiently well prevented and gross wear of the bearing substrate occurs. It is tempting to consider some closed-loop mechanism whereby an initial depletion of a transferred lubricant film results in an elevated CoF (and/or temperature), therefore increasing the wear rate of the material, replenishing the transferred lubricant film. However past experimental campaigns at ESTL (using the SOT) have failed to provide direct evidence of this mechanism, and instead appear to show that the thickness of the transferred film (on a single ball) exists in a bistable state with rapid transitions (i.e. either high or low thickness, with no measured values in-between) and no correlation between film thickness and CoF [20]. Within a more complex application such as an angular contact bearing the behaviour may be multifaceted, but this campaign was enlightening in its demonstration that film thickness alone cannot be used to provide a quantifiable assessment of transfer film life.

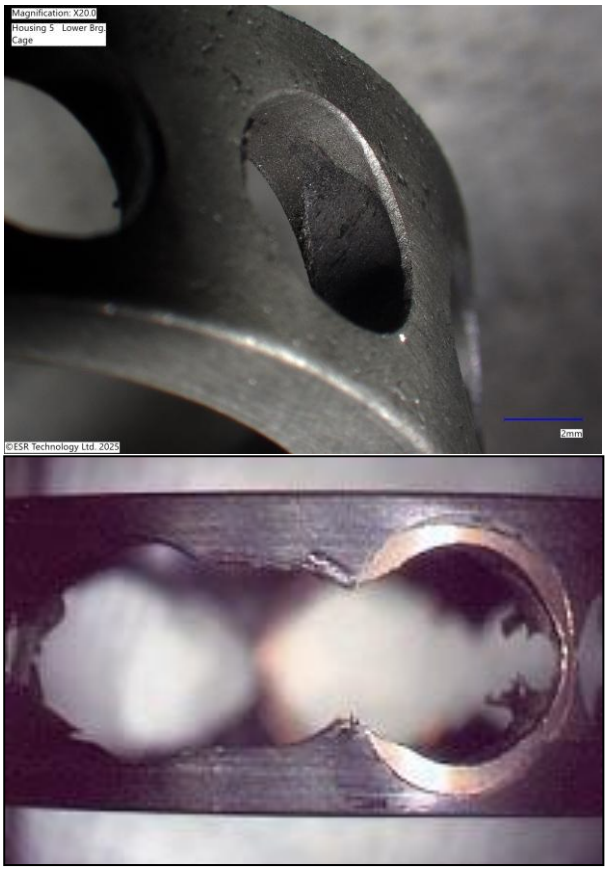

Figure 14. Acceptable (upper) and unacceptable (lower) cage pocket wear

Another approach is to consider the magnitude of SLM wear, as an unacceptably high build-up of transferred material or wear debris may result in choking of the bearing, leading to unacceptable torque noise for the application. Removal of material from the cage pockets will ultimately also result in lack of control of the ball positions, increasing the susceptibility to ball crowding, cage hang-up, vibrational noise, and other contributors to torque noise (Fig. 14). Quantification of “acceptable” cage wear is very difficult however. Previous studies performed by ESTL have focused upon mass loss of the SLM source, with presumably an optimal mass loss (or wear rate) releasing enough, but not too much material to the contacts. The limitation of such mass-based methods is that they are sensitive to the occurrence of a reverse transfer mechanism, where wear particles or transfer material may be transferred back onto the cage, (and are likely very application-specific). To overcome this, a

more stringent mass measurement process may be needed, or otherwise it might be more appropriate to quantify material loss based on wear scar volume.

Ultimately optical microscopy (together with SEM-EDX where allowable) proves to be the most effective methodology for post-test assessments of SLM lubricated contacts. Direct imaging of bearing raceways allows the compositional makeup of the surface to be determined, showing where the originally deposited film remains, and where transfer film material has been deposited upon the surface. Analysis of wear debris is also possible via SEM, but care must be taken during the rinsing/washing stages of sample preparation, as information on its distribution and adherence can easily be lost.

5 CONCLUSIONS

Quantitative identification of the health of a post-test lubricant remains a challenge for the mechanism engineer and, particularly when considering the criteria defined by ECSS, some element of subjective analysis is unavoidable. Nevertheless, recent experimental studies at ESTL can provide guidance on how best to perform this review. For fluid lubricants, High Mass Ion Mobility Spectrometry (HM-IMS) such as the RADIAN ASAP is the preferred methodology, with the ability to both identify failure as well as provide information on the health of the lubricant. For as-deposited films (such as MoS_2), XRF-based inspections of the remaining film in the contact would be preferred, with the understanding that the threshold below which the coating can no longer provide a lubricating film is likely a system property of the contact itself. In instances where SLMs are included, identification of transfer film health is much more difficult, and direct SEM-EDX analysis of the tribological contacts is likely necessary.

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