LESSONS LEARNT FROM LUBRICANTS' TESTING AT ESTEC MATERIALS' LABORATORIES

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ABSTRACT

This paper summarizes the lessons learnt so far from the application of various advanced techniques for characterising space lubricants:
- vapour pressure characterization testing, a method developed to increase the accuracy of evaporation and contamination analysis by measuring the outgassing of contaminants and evaporation of bulk material.
- tests for outgassing properties according to ECSS-Q-ST-70-02C, an in-vacuum screening method for the selection of space materials,
- surface creep characterisation,
- rheology, to characterize the basic properties of the lubricants under different conditions (temperature and relative humidity)
- triborheology with several surfaces and geometries relevant to space applications.

Examples of applications of these techniques to support design phases as well as failure investigations that occurred in space projects are included.

INTRODUCTION

Trade-off between dry and wet lubricants for space mechanisms is often based on a number of criteria which vary from life time, operational temperature ranges, optical payload contamination risks. While dry lubricants performances are more stable in temperature and their usage in the vicinity of sensitive devices is preferred, wet lubricants exhibit more benign friction conditions at tribological surfaces with a resulting longer life within their application thermal ranges. Therefore oil and grease lubricants are often preferred by mechanism's designers, who must ensure that, on one side, the right quantity of fluid remains available at end of life and that, on the other side, the lubricant losses are not posing any degradation risk to nearby sensitive devices. In this sense, evaporation rate prediction of wet lubes becomes a very relevant analysis in combination with labyrinths modelling. One of the most driving characteristic to be considered for the prediction is represented by the vapour pressure of the fluids and more specifically its dependence versus temperature and thermal history. Very often, vapour pressure values reported on lubricants data sheets are not based on actual measurements or physical characterisation along the thermal range, and the validity and applicability have been often questioned. In the recent years, ESA/ESTEC has developed a new in-house capability of measuring the vapour pressure of fluids at different temperature and taking into consideration the thermal history of the fluids themselves, allowing for a better understanding of the evaporation and outgassing phenomena and therefore more accurate predictions. Complementary facilities have been also acquired or developed, which support a wider spectrum of tests of lubricants performances.

VAPOUR PRESSURE CHARACTERIZATION

Most vacuum lubricants display simultaneously outgassing of contaminants and evaporation of the bulk material during flight. Contamination analyses based on vapour pressure only will greatly underestimate the mass loss from as received (i.e. not baked) lubricant, as the vapour pressure values provided by the manufacturer are usually estimated based on the molecular weight of the base oil only. Outgassing of the molecular organic contamination (MOC) and the bulk evaporation scale differently with geometry. Re-emission of outgassed MOC from sensitive surfaces is much faster than the emission from the source [1-3]. For bulk evaporated molecules, the difference between re-emission and emission times are considered negligibly small with some deviations due to the chemical affinities of the target surfaces. Therefore, contamination analyses based on outgassing data only may severely under- or over-estimate the MOC levels. ESA-developed test methodology is based on the fundamental scaling differences in the molecular transportation mechanism of the contaminants during bulk evaporation and outgassing. The test is a combination of kinetic testing [1], bakeout [3] and vapour pressure (p_v) measurements by decreasing the temperature of the test material in a controlled manner until the detection limit of the system is reached. The procedure to separate the outgassing of the MOCs and the bulk evaporation is shown in Table 1.

Table 1 Dynamic outgassing and vapour pressure combined test procedure.

Step	*Detail*
1	Standard kinetic test up to the maximum specified temperature (typically maximum non-operational value for the specific application)
2	Extend the duration of the step at maximum temperature until the deviation from linearity of the TML is less than 1%/h (with a minimum of 3 days).
3	The rate at the end of this step is considered to be rate associated with bulk evaporation value at this temperature.
4	The temperature is lowered in 24 h steps with 12.5 °C decrements until the mass loss in the 24 h steps drops below the detection limit.

Figure 1 is an example measurement obtained by following the procedure in Table 1.

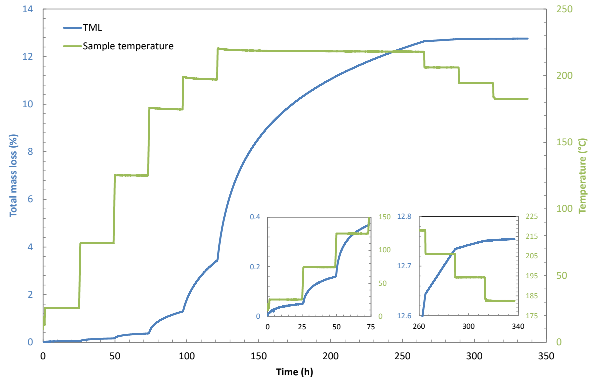

Figure 1. A vapour pressure test conducted in the VBQC chamber (see kinetic test ref). Insets show details of steps I to III and VII to IX.

The analysis of the data starts with Steps 3 and 4. From the linear mass loss rate, the vapour pressure is calculated with the Langmuir equation:

$$\phi_m = (p_v - p_p)\sqrt{\frac{M_M}{2\pi RT}}$$

The temperature dependence follows from the Clausius-Clapeyron relation:

$$\ln\frac{p_2}{p_1} = \frac{L}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

The latent heat of evaporation is obtained from an Arrhenius plot (see Figure 2). As discussed, for most materials, the apparent vapour pressure (p_v) and latent heat of evaporation (L_{app}) depend strongly on the thermal history of the sample. To demonstrate the impact of thermal history, a vapour pressure test was performed where the L_{app} was calculated both at the temperature increase and at the temperature decrease. The difference between the calculated L_{app} results of the test is demonstrated in Figure 2.

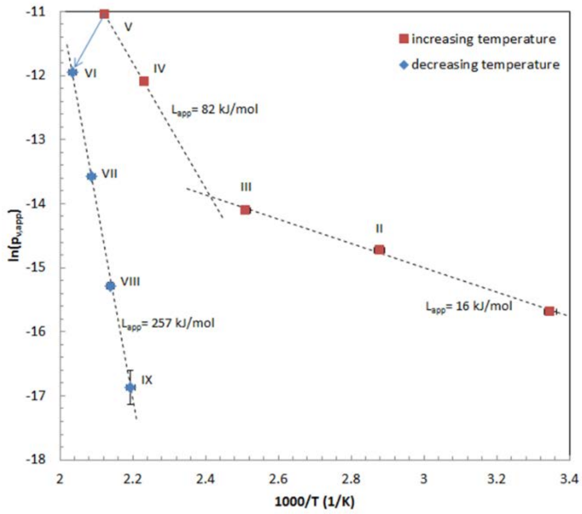

Figure 2. Arrhenius plot, $\ln(p_v)$ vs 1000/temperature in Kelvin plot: red dots indicate the L_{app} during temperature being increased and blue dots demonstrate the L_{app} during temperature being decreased.

The next step consists in subtracting the bulk evaporation contribution from observed total mass loss during the kinetic testing and to correct the measurement for small temperature drift, as the temperature dependence of vapour pressure is most of the time not negligible.

From the mathematical treatment of outgassing data the acceleration factor, the apparent activation energy and the residence time–temperature dependency coefficient involved in the outgassing process were obtained from Figure 1.

The long-term outgassing prediction of the TML, based on the accelerated time approach of [1], is shown in Figure 3 for several constant temperatures ranging from 25 °C to 75 °C.

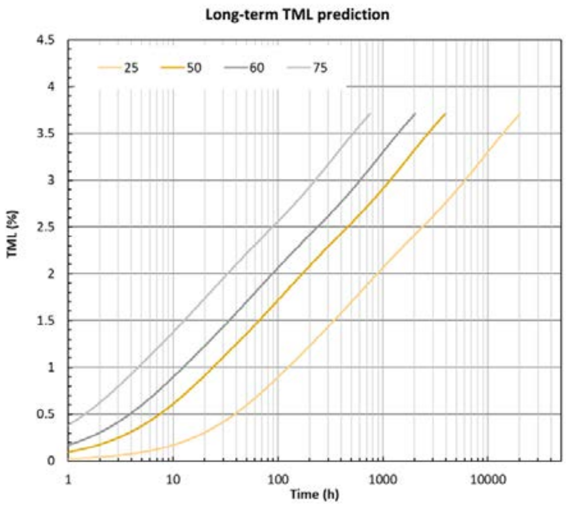

Figure 3. Long-term TML predictions at the indicated temperatures.

As a result of this non-trivial test method both outgassing and vapour pressure analysis can run on parameters obtained from corrected data. The outcome are the mathematical parameters needed for molecular simulation to run long-term prediction and vapour pressure value at some reference temperature with latent heat parameter.

RHEOLOGY AND TRIBORHEOLOGY

The Materials and EEE components laboratory in ESTEC provides extensive support for testing of lubricants for space mechanisms applications. The TA Instruments Discovery Hybrid Rheometer (DHR-2) allows to obtain rheological and viscoelastic properties of viscous materials using several cone-plate or parallel plate configurations. In particular, sample viscosity and stress can be obtained through flow seeps under a wide range of angular velocities. Values of shear storage and loss modulus and tan delta can be obtained for fluids.

All these tests can be performed under complex environmental conditions. Firstly, samples can be tested under a wide range of temperatures. Using a Peltier Plate setup, all these measurements can be performed in the temperature range [-40, +200] °C. With the Environmental Test Chamber (ETC), connected to a liquid nitrogen dewar, a larger range of temperatures can be achieved, between -160°C up to 550°C. Secondly, a humidity chamber can be installed around the sample holder, to test the lubricants under relative humidity conditions between 0% RH and 90% RH.

Figure 4. Left: Parallel plate inside the humidity chamber. Right: Parallel plate configuration mounted on ETC.

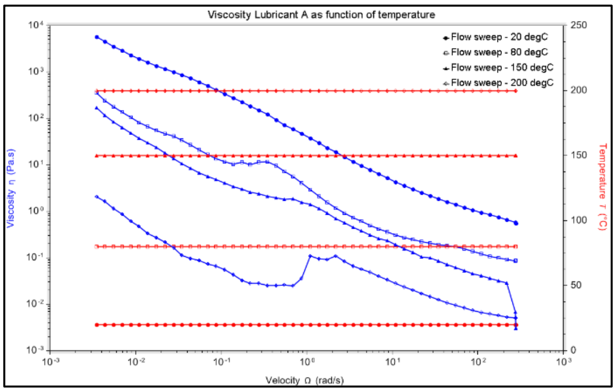

Figure 5. Measured viscosity of a space-grade lubricant as a function of angular velocity and temperature.

Moreover, a tribology accessory extends the capabilities to measure the coefficient of friction between two given surfaces. The following configurations from stainless steel are available at the moment: ring on plate, 3 balls on plate, and 4 balls. It is also possible to obtain Stribeck curves (see Figure 6) using substrates representative of the targeted application in a particular space mechanism.

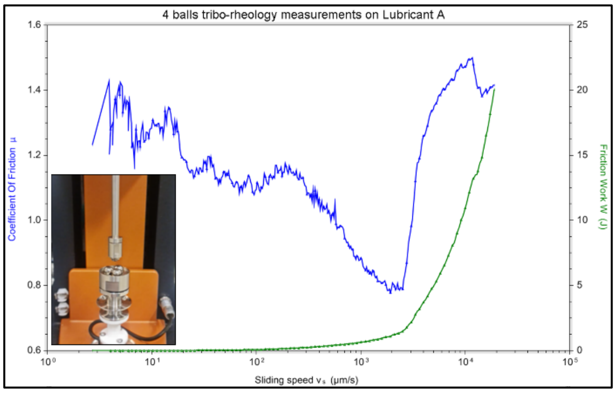

Figure 6. Stribeck curve and friction work of 4 stainless steel balls coated with space-grade lubricant.

Fast, low-cost screening approach to testing of space lubricants could be implemented at ESTEC.

PERSPECTIVE APPLICATIONS

Influence on space optics and other sensitive surfaces

Outgassing of lubricants drive the amount of lubricant needed at beginning of life: despite the loss of mass throughout the lifetime, enough lubricant must remain until the end of life in order to guarantee the expected mechanism functional performances. Standard tests such as the methods described in ECSS-Q-ST-70-02C are important to quantify the lubricant's outgassing properties and the overall potential to release contaminants and to release mass. Nonetheless, from a system's perspective, the full contamination assessment requires the evaluation of the performance-sensitive surfaces and how these are impacted by the presence of the released contaminants. More than quantifying the potential to outgas (from a *source* perspective), the tests described below directly evaluate the system's contamination impact (from a s*ensitive surface* perspective).

Of particular interest are sensitive surfaces located near the lubricant. These can be optical elements such as lenses, mirrors, diffusers, windows, filters or other sensitive elements such as radiators or solar cells, among others. The sensitive surfaces are the ones whose acceptable performance losses drive the contamination requirements.

Many different aspects influence the effects of contaminants on a surface. Ranging from material-related characteristics (several physical and chemical properties of all involved elements), the environment characteristics (such as the temperature profile of both source and surface, partial pressure, ultra-violet radiation), to the actually intended use of the surface (wavelength range, operation concept). Since a standard approach may not cope with such wide range of variables in most cases, dedicated tests are set-up to evaluate the effects of a particular contaminant on a specific surface of interest depending on the characteristics of a specific mission. In such tests the evaluation of the performance is also performed *ad-hoc:* for example transmittance measurements of filters and windows, reflectance measurements of mirrors etc.

The optical measurements allow the evaluation of different effects such as changes in scattered light, changes of polarization, overall decrease of throughput caused by absorption of light on the contamination deposit. In some cases, the contaminated surfaces may also see an apparent increase of throughput (e.g. when the contaminant acts as an anti-reflective layer).

Figure 7 illustrates an example of a contamination test performed with a mix of organic outgassing sources (adhesives). The surface being measured is a diffuser. Total transmittance was measured as a function of contamination amount (up to ~800 ng/cm^2) and UV radiation. The degradation is clearly seen as the increased transmittance loss throughout the test. Due to the general lack of literature data, the authors of this paper also encourage other organizations and industries to perform such evaluations and to propose additional characterizations methods and techniques.

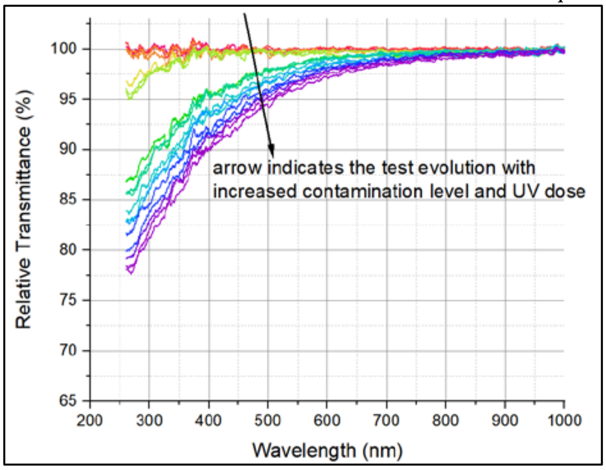

Figure 7. Relative transmittance as a function of wavelength measured on an adhesive-contaminated diffuser.

Currently, as wet lubricants are avoided close to sensitive surfaces, the actual test data may justify their application in case the optical performance remains within acceptable limits. After all, basing contamination requirements on actual performance losses can drastically affect future programs and will allow case-specific requirements which could be more relaxed (or perhaps more stringent) than historically employed ones.

Interaction with regolith simulants

The Apollo missions to the Moon were affected by the presence of dust in lunar environment. In particular, operation of lubricants and seals in space mechanisms was impaired [4-6]. Lunar dust penetrates space mechanisms (Figure 8) as a result of electrostatic levitation which is connected with natural day-night balance in lunar environment. Plume of lunar dust may be created during landing, rover operation and impact of micrometeorites.

The presence of lunar dust might adversely impact space mechanisms causing abrasive wear problems, as well as issues with the actuation mechanism due to variable dynamic/static friction coefficient. Current strategies to deal with these problems include the use of antiabrasive coatings such as diamond-like carbon (DLC) coatings [4].

Model studies can be undertaken using pre-defined amounts of regolith simulants and simple laboratory setup for triborheology.

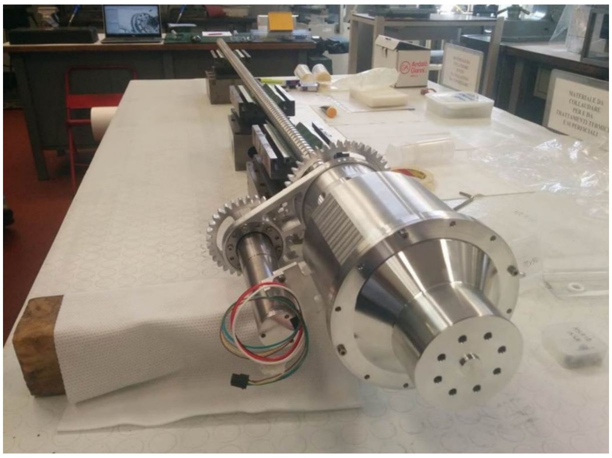

Figure 8. A drill designed to penetrate 1–2 m into the lunar surface is envisaged by ESA to fly to the Moon's south pole on Russia's Luna-27 lander in 2020. Credit: ESA/Finmeccanica.

CONCLUSION

To sum up, ESTEC laboratories have been involved in failure investigations and qualification activities for space lubricants. The available techniques include in particular vapour pressure characterization and rheology / triborheology.

Future directions of testing will include impact of lunar / martian dust on rheological properties of lubricants which is of strategical importance for future long-term lunar and martian missions. Moreover, the growing requirements of optical space missions require more extensive testing of impact of lubricants on optical payloads, therefore the in-house facilities will be also extended in this direction.

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