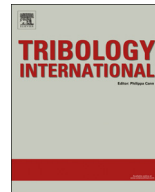




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Film thickness and friction behaviour of thermally aged lubricating greases

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ABSTRACT

Non-additized batches of lubricating greases were subjected to an artificial thermal aging procedure. The fresh and aged samples were then tribologically characterized through film thickness measurements and Stribeck curves, over a large range of entrainment speeds on a ball-on-disc test rig at different operating temperatures, ensuring fully flooded conditions. The results were analysed at the light of the physical and chemical changes regarding the fresh greases.

The film thickness increased for all greases after the thermal aging. The film thickness plateau at low speeds also changed and for certain greases it disappeared under the same range of entrainment speeds. The friction behaviour also changed considerably given the changes to the lubrication regimes for the same speeds.

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1. Introduction

The grease aging in rolling bearings is characterized by permanent changes in its properties. According to Cann et al. [1], these changes depend mostly on the grease formulation (base oil viscosity and nature, thickener type), on the bearing geometry, material and type, on the bearing house and also on the rolling bearing operating conditions (mainly speed and temperature) and running time. Wear debris and water contamination have also been reported to promote grease aging [2–5].

It is known that quite frequently a large majority of the rolling bearings fail primarily due to lubricant failure than by surface fatigue [6]. However, it is still very hard to accurately estimate grease life based only on the bulk properties of the fresh greases. Different aging mechanisms are known to occur with grease when lubricating a rolling bearing, depending on the operating conditions. The aging can generally be grouped in mechanical and chemical changes. It is known that during operation at high

temperatures the grease is continuously stressed thermally and mechanically, leading to chemical and physical changes. However, the way these changes affect the lubricity and the capacity to maintain a lubricating film are still unknown. Nonetheless, it is frequent for rolling bearings to fail primarily due to lubrication problems than by surface fatigue [6].

The mechanical aging, which is not the scope of this work, promotes oil separation and deterioration of the grease thickener matrix [6]. The balance between the oil loss and the degradation of thickener material by shear can lead to grease softening or hardening (reduction or increase in consistency, respectively).

On the other hand, the chemical/thermal aging of greases is still poorly studied. The oxidation of both thickener and oil can happen (as shown in a previous work [7]) and it was found that this type of aging can also lead to grease softening or hardening depending on the grease formulation (thickener nature, base oil viscosity/nature) [8]. The oxidation is known to promote reaction/consumption of additives, acid formation, thermo-oxidative degradation of thickeners, polymerization of base oils, thermo-oxidative degradation of the base oils and also to promote the formation of varnish and sludge [6,8,9]. However, the ways these changes modify the tribological behaviour are unknown.

This paper intends to show how the thermal aging affects lubricating greases' properties and how these changes interfere with the film thickness and friction behaviour of the aged greases.

Abbreviation: ATR, Attenuated Total Reflectance; COF, Coefficient of friction; E, Ester based oil; FTIR, Fourier-Transform Infra-red Spectroscopy; LiX, Lithium complex thickener; MIN, mineral base oil; PAO, Poly-alpha-olefin base oil; PP, Polypropylene thickener; SEM, Scanning Electron Microscopy; SRR, Slide-to-roll ratio (%)

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Notation and Units

h_0	central film thickness (m)
h_{pl}	central film thickness in the plateau region (m)
H_{0c}	central film thickness calculated according to Hamrock and Dowson (m)
H_{exp}	central film thickness measured under moderate to high speeds (m)
σ	composite roughness of the surfaces (m)
U	dimensionless Speed parameter (-)
W	dimensionless Load parameter (-)
G	dimensionless Material parameter (-)

η	dynamic viscosity (Pa s)
U_0	entrainment speed (m/s)
R_x	equivalent radius in the rolling direction (m)
C_0	influence of ellipticity (-)
ν	kinematic viscosity (cSt)
F	normal load (N)
T	operating temperature (°C)
α	pressure-viscosity coefficient (1/Pa)
σ_1	roughness of the ball's surface (m)
σ_2	roughness of the disc's surface (m)
Λ	specific film thickness (-)
S_p	Stribeck modified parameter (-)

2. Methods and materials**2.1. Tested greases**

Four lubricating greases were tested in this work: M2, M5, MLI and MLI_M, specifically manufactured for this work by Axel Christiernsson. The greases' main properties are shown in Table 1.

The polymer greases M2 and M5 were formulated with the same poly-alpha-olefin (PAO) base oil. Grease MLI was formulated with a mixture of two different grades of PAO and 5% ester (PAO+5%E), added to facilitate the saponification reaction. Finally, grease MLI_M was formulated with a base oil of mineral (MIN) nature.

Regarding the thickener, greases M2 and M5 were formulated with polypropylene (PP). Greases MLI and MLI_M were formulated with Lithium Complex (LiX) soap thickener. None of these greases is additized. However, grease M5 was formulated with an elastomer (as co-thickener), which works as a viscosity improver of the oil bleed by the grease under work allowing to control the oil bleeding rate. This elastomer also contributes to improve the mechanical stability of the grease [10]. Grease MLI_M was formulated with 1.7 % of PIB (Polyisobutylene) which acts like a viscosity improver.

2.2. Aging process and evaluation methods

The aging process was performed on a sample of each fresh grease. The sample was manually spread over a steel disk forming a layer thickness of approximately 1 mm. The process was intended to replicate the aging in grease lubricated rolling bearings and the grease interaction with the steel surface. Despite the layer thickness could have a considerable influence on the overall aging of the grease sample, it was found that for this aging time and temperature, 1 mm was enough to obtain an homogeneous sample of aged grease. Similar aging methods have been used by other authors [9,3,11], using grease layers of different thickness.

Both the polymer and the LiX thickened greases were thermally aged in the oven for ten consecutive days (240 h) at 120 °C which is the maximum working temperature of the polymer greases, indicated by the manufacturer for continuous use (the corresponding temperature for the LiX thickened greases is 150 °C). The temperature of the oven was adjusted manually through a potentiometer, using a thermometer for the atmosphere temperature evaluation. Although there was no forced convection mechanism, the oven had a breathing hole on the top, allowing the chamber atmosphere to be refreshed. Care was taken to keep the temperature constant at all times.

After the aging process, the disc was removed from the oven to the room temperature and the grease was immediately collected in a container to avoid further aging. The aged samples will be

Table 1

Tested greases' properties.

Grease reference		M2	M5	MLi	MLi _M	Units
Thickener type		PP	PP	LiX	LiX	-
Base oil nature		PAO	PAO	PAO+5%E	MIN	-
Thickener content		13	13	17.5	10.6	%
Elastomer content		0	2.6	0	0	%
Worked penetration (ISO 2137)		269	249	276	279	10 ⁻¹ mm
NLGI		2	3	2	2	-
Storage modulus ^a G'	80 °C	21 347	29 810	22 285	15 820	Pa
Loss modulus ^a G''		4596	6029	7102	1861	Pa
Base oil viscosity (ASTM D445)	40 °C	48.0		178.7	153.3	mm ² /s
	100 °C	8.0		21.4	15.7	
Bleed oil viscosity (ISO 12058)	40 °C	49.3	779.1	135.2	159.9	mm ² /s
	100 °C	8.3	93.3	18.9	16.6	

^a values measured at the LVE region, under a frequency of $\omega=1$ rad/s.

referred to with the suffix "a" for aged. These samples were evaluated through FTIR and rheology measurements. The thickener morphology was also investigated through scanning electron microscopy (SEM).

The FTIR spectra were obtained on an Agilent[®] Cary 630 FTIR device, using an ATR (Attenuated total reflectance) accessory. The full wavelength range (650–4000 cm⁻¹) available for this device was tested. The samples were analysed through direct comparison of height of the characteristic oxidation peaks between the samples spectra. All the spectra shown in this work were taken directly from the device's software without smoothing and a very good reproducibility was achieved. All spectra were normalized to the same peak's height at 1460 cm⁻¹ [9], allowing the comparison between samples.

The rheology measurements were performed on a Physica MCR 301 rheometer, using a smooth plate-plate geometry PP50 ($\phi=49.998$ mm). No rough of sand-blasted plates were available. The storage and loss moduli were measured in the linear viscoelastic region, under a frequency of 1 rad/s and at 80 °C, one of the temperatures at which the film thickness and friction tests were conducted. A gap of 1 mm between plates was chosen and a pre-shear procedure (see Appendix A) was applied to each sample before the main test.

On the other hand, the dynamic viscosity of the bleed-oils was measured using the same plate-plate geometry but with a 0.1 mm gap. The rotational speed for these measurements ranged from 10⁻⁴ up to 3300 rpm. Please refer to [7] for more information regarding the rheology measurements.

The analysis of the grease micro-structure was performed using a scanning electron microscope (SEM) operating under a high vacuum environment (FEI Quanta 400FEG ESEM/EDAX Genesis

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