



Article Influence of Rheological Properties of Lithium Greases on Operating Behavior in Oscillating Rolling Bearings at a Small Swivel Angle

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Abstract: In this study, the behavior of greases during oscillating bearing operation with a small oscillation angle and high frequency was investigated. This mode of operation entails demands on the lubrication system that differ significantly from those for continuously rotating bearings. In order to determine the variables influencing the suitability of a lubricating grease for small angle oscillating operation, the grease samples were examined with particular regard to their rheological properties. The focus of this investigation was to find a relationship between the rheological parameters and the real behavior in the bearing. Therefore, rheological and physical parameters, which influence the longterm structural changes and lubrication conditions, were identified. For this purpose, the viscosity was measured over a wide shear-rate range. The storage and loss modulus, the work of deformation, and the adhesion force jump are also determined. Afterward, rotational transient flow measurements were performed. These allowed us to analyze the development of the shear stress over time, at a constant shear rate, and to examine the internal friction behavior by evaluating the energy density. Subsequently, grease-lubricated four-point bearings were used in component tests, while the frictional torque was measured. These bearings operated in oscillating motion. Moreover, the yield point of mechanically aged greases was measured and compared with that of fresh greases to examine the influence of the oscillating operation on the lubricant condition. Finally, correlations between grease composition, rheological measurements, and component tests were investigated. Thereby, parameters influencing the frictional behavior of greases in rolling bearings during oscillating operation at small swivel angles were identified.

Keywords: grease; lubrication; friction; tribology; rheology; oscillation bearing

1. Introduction

Lubricants are used in various areas of mechanical engineering to reduce friction and wear. Due to technological progress in the industry, higher speeds and/or loads are demanded, while simultaneously increasing the longevity of these systems [1]. Additionally, the need for completely automated systems with low maintenance requirements is constantly increasing. Thereby, the demand for the miniaturization and minimization of machine complexity makes it extremely difficult to integrate complex lubrication systems such as circulation oil lubrication. The use of grease as a lubricant offers the possibility of complying with cost reduction, resource efficiency, and competitiveness. However, grease behavior in tribological contacts has not been fully understood up to this date.

Due to the high complexity of the lubricating grease composition and its frictional behavior, various studies have investigated the processes within the structure. For this purpose, Li et al. examined [2] the temperature dependence of viscosity, storage, and loss modulus on the rheological behavior in general. Delgado et al. [3] used a statistical approach and a subsequent investigation to take a closer look at the influence of base oil



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). viscosity and soap concentration on the rheological and mechanical properties of lubricating greases. Couronne et al. [4] proved with their research that the soap-base oil interaction has a significant influence on the rheological and tribological properties of lubricating greases. According to Xu et al. [5], the initial lubricating film is proportional to the thickener concentration. Thus, a lower overall grease viscosity due to less soap content leads to a thinner lubricating film. Lovell et al. [6] pointed out that the frictional torque of rolling bearings operating under small angular rotation increases with the increasing lubricant viscosity. Moreover, Dresel [7] stated the thickener does not exclusively work as an oil reservoir but is also directly involved in lubrication processes of the tribological contact area. For this purpose, the thickener structure must first be changed by applying shear stress. This process is referred to as lubricant degradation or lubricant wear. According to Kuhn [8], the exceeding of critical energy levels is responsible for initiating this structural adaptation. Cousseau et al. [9] investigated a dependence of rheological properties and frictional torque and additionally grease wear. A correlation between low base oil viscosity, poor contact filling, and low rolling moment was found. In another work [10], the influence of grease aging and thickener at different lubrication conditions on lubricant film thickness was investigated. Important factors in this context are the thickener type and concentration. In addition [11], a comparison of experiments and grease properties was made to evaluate the performance of the greases. Among other things, the rheological behavior of greases was evaluated with sweep and flow tests. It was found that the interaction between thickening agent and base oil has a strong influence on the frictional torque. Furthermore, the rolling frictional torque depends on the viscosity of the lubricating oil at the operating temperature and on the refilling of the contact. Goncalves [12] considered thickened greases in terms of their thickener content and its effect on rheological properties. The focus was on the storage and loss modulus values in the linear visco-elastic (LVE) range. It was demonstrated that the formulation changes the rheology and imparts different properties to the greases. The observed behavior showed that the storage and loss moduli (G' and G'') increase with the thickener content. Adhvaryu et al. [13] defined this degradation as a reduction or damage to the tribological lubrication properties. Yuxin et al. [14] confirmed this by performing rheometer measurements at room temperature to analyze the behavior of a lithium grease under shear stress. In contrast to that, a study by Zhou et al. [15] showed that shear degradation may also cause positive effects such as increased oil mobility due to reduced yield stress; increased fiber size, which increases the film thickness; and the ability to release more oil that would, in turn, improve replenishment. However, negative effects on the performance of rolling bearings are also referred to. Cyriac et al. [16] provide research on the grease film thickness in a rolling elastohydrodynamically lubricated rolling contact under fully flooded contact at medium speeds in order to identify the influencing parameters. Evidence of the presence of thickening agents in the contact emerged. An increase in effective viscosity is associated with the presence of thickening particles in the inlet of the contact. An analysis of the relationship between the effective viscosity and the concentration of thickener particles showed that not all thickener particles migrate through the contact.

By carrying out long-term bearing tests and analyzing frictional torque, Kuhlmann [17] developed a test specification for greases in line-contact bearings under isothermal operating conditions at low temperatures. According to his study, the bearing service life is largely determined by the lubricant and its properties, such as oil release behavior, dynamic viscosity, base oil, additives, and thickener when grease-lubricated rolling bearings are used. Especially in oscillating applications, bearings usually operate at a speed which aggravates building up an elasto-hydrodynamic lubricant film and favors lubricant degradation due to non-stationary kinematics [18]. In contrast to continuous rotation, the processes in bearings during oscillatory motion have not been sufficiently investigated so far. Up to this date, suitable lubricating greases for use under such conditions are selected based on complex and expensive long-term tests. By selecting a grease based on its rheological–physical parameters, cost-intensive test runs might be avoided. Furthermore, a deeper understanding of the relation between rheology and practical application may also allow us to increase the grease's operating life and, thus, bearing life.

As part of the research project [19], greases have already been investigated in oscillating bearing operation with swivel angles of 30° and 90° . This allowed predictions to be made based on rheological properties for defined operating conditions. For oscillating applications with low load (<2 kN), the used grease should have sufficiently high oil release capacity (>3%), the smallest possible storage modulus (<25 kPa), a moderate yield point (shear deformation between 30% and 40%), and a low base oil viscosity (50 cSt). Additionally, better results could be achieved with greases based on synthetic oil. The results of bearing testing for the grease samples regarding wear and damage to the bearing raceway surfaces are displayed in Table 1. A constant and low curve of the frictional torque and low bearing damage after testing are considered to be good results. It was found that Grease 1 and Grease 5 show the best behavior when operating with a swivel angle of 30° and an oscillation frequency of 1 Hz under a load of 2 kN, meaning that bearing damage after each test was relatively low compared to tests with other greases. In contrast to that, Grease 2, Grease 4, and Grease 6 did cause significant wear in the bearing's raceway surfaces. Thus, they were considered as not being suitable for the investigated operating conditions. An increase of the oscillation angle from 30° to 90° , while the frequency and load remained constant, led to lesser bearing damages for all greases. This may be due to overlapping of the rolling elements' movement ranges and, thus, better lubricant distribution. When increasing the oscillation frequency from 1 to 5 Hz, an overall increase in occurring bearing damages was detected. This effect was also observed for every grease. If the frequency and the swivel angle are increased to 5 Hz and 90°, while keeping the load at 2 kN, all greases show good results regarding the wear of the bearing raceways, except for Grease 4, Grease 2, and Grease 6. However, it was suspected that very small swivel angles could favor problems in terms of bearing lubrication, such as starvation.

Grease Sample	NLGI	Base Oil	Base Oil Viscosity in cSt	30° 1 Hz 2 kN	30° 5 Hz 2 kN	90° 5 Hz 2 kN	90° 1 Hz 2 kN
Grease 1	1	MIN	50	+	_	+	+
Grease 2	2	MIN	50	_	_	_	+
Grease 3	1	MIN	100	+	_	+	+
Grease 4	2	MIN	100	_	_	+	+
Grease 5	1	PAO	50	+	_	+	+
Grease 6	2	PAO	50	_	_	_	+

Table 1. Results of the investigations according to DGMK project no. 810 [19].

"+" means relatively low wear; "-" means relatively high wear.

David et al. [20] investigated the behavior of frictional torque in grease-lubricated rolling bearings rotating in oscillatory motion with small angles. They found that the frictional torque steadily increases over continuously repeating cycles and suggested that this may be due to a combination of grease degradation and starvation caused by the small rotational motion. This was confirmed by Wandel et al. [21], who compared bearings operating under small oscillation angles including a relubrication cycle with a larger oscillation amplitude of 60° every 100th cycle with bearings operating under the same conditions without a relubrication cycle. It was found that including a relubrication cycle significantly lowers bearing damages. Thus, it was suggested that, next to load, oscillation frequency, and amplitude, the oil release ability of a grease and oil mobility within a bearing are crucial parameters when operating under small swivel angles. These parameters are strongly dependent on the lubricant viscosity.

For this purpose, relations between rheological properties and bearing application at these angles were investigated in this work. The focus is to make a lubricant selection based on characteristic grease parameters possible and examine the problem of starvation.

2. Materials and Methods

2.1. Test Greases

In this study, lubricating greases with various parameters from DGMK810 [19] were further investigated. The selection is limited only to lubricants with a thickener based on lithium. Thus, the influence of different thickener types on the behavior of greases is eliminated. The chosen grease samples differ in their properties, such as soap content, NLGI class, base oil type (base mineral oil MIN and base synthetic oil PAO), base oil viscosity at 40 °C, and rolling stability (according to ASTM D1831), as well as the additive package used. Information about the thickener content in percent by weights is provided by the grease manufacturer. Based on these differences, a conclusion can be drawn about the behavior-influencing parameters. In total, six greases were examined within this work. An overview and more detailed information about the individual properties of the chosen lubricating greases are given below in Table 2.

Table 2. Individual properties of the examined lubricating greases.

Grease Sample	Thickener (%)	NLGI	Base Oil	Base Oil Viscosity in cSt	Roll Stability (Difference 0.1 mm)	Oil Separation Rate [22]
Grease 1	Li (7.9)	1	MIN	50	106	2.342%
Grease 2	Li (9.5)	2	MIN	50	87	1.339%
Grease 3	Li (8.06)	1	MIN	100	135	1.549%
Grease 4	Li (9.48)	2	MIN	100	105	3.020%
Grease 5	Li (7.5)	1	PAO	50	75	5.608%
Grease 6	Li (10.5)	2	PAO	50	98	1.103%

All greases contained the same additive package, which was a combination of specialized synthetic sulfur, high-pressure components, corrosion, and wear inhibitors. Thus, the friction was reduced, wear was decreased, and welding of metal surfaces was prevented. By using the exact same additives in all greases, any influence on the test results due to differences of additives could be excluded.

2.2. Rheological Tests

The respective rheological measurements were performed three times, from which the mean value was determined for further comparisons.

2.2.1. Dynamic Viscosity

Dynamic viscosity is one of the key properties of a lubricant regarding tribological contact areas. It fundamentally influences friction and, therefore, the occurring wear of contact partners. This comes from the percentage of thickener in the contact which is nearly zero at high speeds and up to 50% at low speeds [23]. The viscosity is determined by means of a rotational test under isothermal conditions at temperatures of 20 and 80 °C. These temperature levels were specified in DGMK project no. 810 [19] and further investigated in this study in order to extend the experimental database for oscillatory bearing motion. For this purpose, an Anton Paar rheometer type MCR 102 (Anton Paar Germany GmbH, Ostfildern, Germany) is used, applying the Searle method with a continuously rotating measuring body. The tests are performed with a CP25-1/TG cone/plate system (Anton Paar Germany GmbH, Ostfildern, Germany). The cone diameter is d = 25 mm, and the taper angle is $\alpha = 1^{\circ}$. In order to investigate the viscosity of the greases within a wide range of shear rates, the minimum is set to $\dot{\gamma}_{min} = 0.001 \text{ s}^{-1}$, and the maximum is set to the limit of the measurement system of $\dot{\gamma}_{max} = 17,500 \text{ s}^{-1}$.

2.2.2. Rational Transient Flow

Frictional behavior and, thus, longevity of tribological contacts is determined by either solid friction, fluid friction, or a combination of both. In order to take a closer look at fluid

friction, which occurs inside the lubricant, rheological energy densities were investigated. It represents the energy expenditure required to deform a certain lubricant volume [24]. The rotational transient flow measurements of the model greases were carried out on an MCR-102 rheometer (Anton Paar Germany GmbH, Ostfildern, Germany) by using a PP25 plate–plate system (Anton Paar Germany GmbH, Ostfildern, Germany). The measurement gap was set to 1 mm, and all tests were performed at 20 and 80 °C for a duration of 2700 s while applying a constant shear rate of 1000 s^{-1} . After reaching the testing temperature, and previously to applying the shear stress, the greases were left to rest for 15 min in order to relax their structure. By integrating the $\tau(t)$ against the t curve from the point of maximum shear stress, t_{max} , to the end of measurement, t, the rheological energy densities are obtained [25].

$$e_{\text{Rheo}} = \dot{\gamma} \cdot \int_{t_{\text{max}}}^{t} \tau(t) dt$$
(1)

In Equation (1), $\dot{\gamma}$ represents the applied constant shear rate, while $\tau(t)$ represents the measured shear stress.

2.2.3. Viscoelastic Properties

The viscoelastic properties of a grease provide information about its behavior under shear stress and, more precisely, the resistance of the lubricant's structure toward shearing. Moreover, it allows an investigation of the deformation required to degrade the grease structure. These viscoelastic properties are determined with an amplitude sweep test, using a plate/plate measuring system. Thereby, the oscillation amplitude is varied at a constant oscillation frequency [26]. The tests are performed by specifying the shear deformation and increasing deformation amplitude with time. This allowed a higher reproducibility of the measurement results outside the LVE range compared to tests with shear stress specification. Shear deformation is determined by the displacement of the upper plate and height of the measuring gap.

The amplitude tests were performed under isothermal conditions at 20 and 80 °C, using a PP25/TG measurement system (Anton Paar Germany GmbH, Ostfildern, Germany). According to DIN 51810-2 [22], the constant angular frequency is specified as $\omega = 10 \text{ s}^{-1}$, and the shear deformation is increased logarithmically, from $\gamma = 0.01\%$ to $\gamma = 100\%$.

2.2.4. Tack Test

The tack test is used to investigate the cohesive forces within the grease sample and the adhesion forces between the grease and the surface of the measuring system. Thus, the tackiness of the sample can be determined. It describes the ability of substances to form a bond with measurable strength between the contact partners at low contact pressure and short contact time [27]. In particular, the resistance of an adhesive substance to the separation of the bonded surfaces was examined [28].

The tests were performed at 20 and 80 °C for each sample by the Tribology Research Center (TREC) of the University of Applied Sciences Hamburg, Germany, using an Anton Paar MCR 302 rheometer (Anton Paar Germany GmbH, Ostfildern, Germany).

The positioning speed was set to $v_1 = 0.01 \text{ mm/s}$ in order to position the system exactly to a gap height of $d_T = 2 \text{ mm}$, while avoiding overloading the specimen. This position was held for one second. Subsequently, the measuring system stroke was executed. Therefore, the constant stroke speed was set to $v_2 = 0.1 \text{ mm/s}$. The stroke limit was set to the maximum possible value of the measuring system $d_T = 10 \text{ mm}$. A load cell was used to measure the perpendicular force, F_N , of the grease sample that opposes the stroke movement of the upper measuring element due to adhesion and cohesion. A maximum of the perpendicular force, F_N , represents the internal cohesion of the specimen structure, whereas a minimum of the perpendicular force, F_N , represents the adhesion to surfaces. As long as the measured perpendicular force, F_N , shows negative values while gap height increases, the lubricant sample forms threads, which counteract the stroke movement of the

upper measuring body. As soon as the perpendicular force, F_N , becomes zero, the grease threads are torn off.

2.2.5. Squeeze Test

The squeeze test allowed us to draw conclusions about the grease behavior during displacement in the rolling bearing and, therefore, required energy expenditure. For this purpose, a defined volume of grease was applied to a measuring gap between two measuring bodies. The upper measuring body could be moved vertically and was positioned to provide contact between the specimen and both measuring bodies. Subsequently, the sample was loaded with a compressive force or velocity profile until a defined perpendicular force or gap height was reached. The perpendicular force and gap height were measured continuously throughout the whole process. As a result, the gap height, d, is typically plotted vs. the perpendicular force, F_N . Afterward, the area under the curve $d(F_N)$ was calculated. This area under the graph is an indication to the energy required to deform the grease and can be interpreted as an indicator to churning losses in real bearings. Here, the grease is squeezed through the cage's pocket and the rolling element, as well as between the cage and its guiding raceway.

The tests were carried out for each grease sample at 20 and 80 °C. The gap height at the beginning of the measuring phase was set to $d_S = 1$ mm, since this is the point at which initial contact between the measuring system and the test sample occurs. In the loading phase, the perpendicular force, F_N , that squeezes the grease sample was linearly increased from zero to a maximum of 30 N. This value was selected as the maximum in order to prevent the gap height from dropping to zero while testing soft samples. Otherwise, the measuring system could be damaged.

2.3. Tribological Tests

The respective tribological measurements were performed three times, from which the mean value was determined for further comparisons.

2.3.1. Ball-Bearing Test

The ITR-Bearing Test [29] consists of two specific test housings, which are horizontally directed and connected by a vertical shaft. At each end, a four-point angular contact ball bearing of type QJ212-TVP is mounted, as illustrated in Figure 1.



Figure 1. Schematic view of ITR-Roller-Bearing Test (**a**); test-arrangement, movement, and load direction four-point angular contact ball bearing (**b**).

Both test housings and, consequently, the test bearing are axially loaded under dynamic conditions +/-23.3 kN by means of a hydropulser. By using a modified crank drive, which is connected to the shaft and driven by a three-phase motor, a swing movement, $\Delta \varphi$, of the shaft and rolling elements (balls) is achieved. Tests were conducted at room temperature. The temperature of both bearings and the test shaft was measured by using thermoelements (Pt 100), and the results depended on the absolute temperature of the test housing and heating due to friction. Besides the temperature, the frictional and swing torques of each test bearing were separately measured. The measuring signals of temperature and frictional torque were continuously measured by an NI data acquisition system and postprocessed through a bespoke MATLAB R2020a code.

2.3.2. Experimental Conditions

The ITR-Bearing Test was conducted under the following experimental conditions listed in Table 3:

Experimental Parameter	Parameter Value
Amplitude pulser-load (sine-wave shape)	\pm 23.3 kN (5 rolling elements)
Max contact pressure	around 3000 MPa in inner ring
Frequency of swing	7 Hz (length of a test: 43 h)
Number of load cycles	1.1 million
Operating temperature	20 °C
Filling degree of grease	Full filling
Swing angle	$\pm 4.6^{\circ}$

Table 3. Experimental conditions of Ball-Bearing Test.

2.3.3. Measurement Technique

The axial load (F_A), swing movement of shaft, movement of hydropulser, frictional torque, and temperature of bearing can be measured either continuously or intermittently. After the completion of the test, the bearing-damage depth was measured.

The axial load (F_A) was measured by a load cell, which was mounted on the top of the housing. The frictional torque of the bearing was measured by a torque-strain gauge, which is defined by the strain of shaft. During a test run, the frictional torque was continuously recorded. Due to the oscillatory motion, the friction torque curves show a sinusoidal form. These signals were subsequently processed and plotted as a function of time. Therefore, the absolute values of all points within a single cycle were determined. To obtain the characteristic value of a cycle, these absolute values were then averaged. Additionally, a microscopic examination of the bearing raceway surface and grease condition after each test run was conducted to compare the lubricating performance of all greases with each other.

The measurement of depth failure succeeds by using the computer-controlled formmeasuring device of type "MMQ 400" from Mahr GmbH, Göttingen, Germany. For the measurement, a clamping block was used to hold the bearing's inner ring, which was placed on a measuring board. This instrument could not measure the depth's area; only the damage's depth per scanning path could be measured. The device has a measuring head, which has a sensor pin. Before each measurement, the ring was automatically centered. The damage's depth per inner ring was measured in three scanning paths (s_1 , s_2 , and s_3) at distances of 1.5, 3.5, and 5 mm from the upper edges along the circumference of the inner ring. They were scanned at an arc length of 12 mm of the inner ring (Figure 2).



Figure 2. Schematic representation of three scanning paths $(s_1, s_2, and s_3)$ for damage's depth measurement.

3. Results and Discussion

3.1. Dynamic Viscosity

By evaluating the viscosity curves, a statement about a grease's shear stability behavior can be made. For a fresh grease, the thickener is formed by long intermeshing fibers. When subjected to shear stress, these fibers may break and shorten, resulting in the grease's viscosity being reduced. According to Cann and Lubrecht [30], this thickener fiber breakage would occur by increasing the operating time during which the grease is subjected to a constant shear. On the other hand, shear thinning occurs when these thickener fibers are broken down due to the increasing shear rate. Both methods would lead to the same output, which is the decrease in the lubricant's viscosity. In this work, the word shear stability would be used to describe any decrease in the grease's viscosity due to shear (either by applying a constant shear rate for a long time or by applying an increasing shear rate). The shear stability of a grease directly affects the availability of free oil, which is crucial for maintaining and replenishing the lubricating film in the tribological contact area. A grease with high shear stability slowly releases its base oil, which may favor starvation. In contrast, a grease with low shear stability quickly releases its base oil. This may result in premature grease dry-out and, thus, shorter lifetime. Furthermore, the operating temperature influences the viscosity and replenishment of the lubricating film. Therefore, a decreasing temperature leads to an increased viscosity. This increases the starvation probability [31]. Consequently, a balance between the operating temperature and the shear stability has to be found. This can be achieved by carefully selecting the right thickener fiber size or the base oil viscosity.

Figure 3 shows the measured viscosity curves of the lubricating greases in a log–log diagram, which also makes small changes in viscosity at lower shear rates visible. By comparing both Grease 1 with Grease 2, and Grease 5 with Grease 6, it can be observed that an increasing soap content causes a major increase in grease viscosity at low shear rates. This viscosity can also be changed by altering the used manufacturing process, as mentioned by Cousseau et al. [10]. From $\dot{\gamma} = 1 \text{ s}^{-1}$, this viscosity difference between the greases steadily decreases, while the shear rate increases until the viscosities, η_E , of all greases finally show values of similar magnitude located in a range from 0.275 to 0.3 Pas.

The viscosity curves of Greases 1 and 2 at 20 and 80 °C show an increasing influence of temperature on the grease viscosity with increasing soap content. This behavior can only be observed with mineral-oil-based greases. In contrast to that, grease samples with synthetic base oils show an inverse correlation. This is additionally illustrated by a comparison of the curves of Greases 5 and 6 at 20 and 80 °C. In total, Greases 5 and 6 have the lowest and highest shear stability, respectively. This can probably be attributed to the difference in the thickener content. Figure 3c,d shows noticeable local minima of the curves at 80 °C which are followed by temporary increases in grease viscosity. This may be caused by interactions between additives and the other lubricating grease ingredients. Another possible explanation for this behavior could be a too-low base oil viscosity due to high temperature, which results in increased oil bleeding/oil loss [30]. For further experimental results see Appendix A.



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Figure 3. Viscosity shear rate behavior (base oil viscosity, 50 cSt (**a**) and 100 cSt (**b**)): temperature effect on viscosity for synthetic-oil-based (**c**) and mineral-oil-based (**d**) lubricating greases.

3.2. Rational Transient Flow

The measured frictional energy density results are displayed in Figure 4 and Appendix B. The calculated values for the rheological energy densities for each grease at the testing temperatures of 20 and 80 $^{\circ}$ C are shown in Figure 5.

By comparing the transient flow curves shown in Figure 4, it may be seen that lubricating greases exhibit higher values of residual shear stress with increasing soap content. That is only observed at 20 °C. At the 80 °C temperature, the residual shear stress of Greases 1, 3, and 5 does not follow the same trend. In Figure 5, a significant temperature dependency of the rheological energy densities is clearly visible. An increase in temperature generally leads to a reduction in energy density. This originates from the change in the grease structure. Since rheological energy density is understood as a measure of the energy required to shear a lubricating grease, it fundamentally influences grease behavior in tribological contacts.



Figure 4. Transient flow curves of all greases at 20 °C.



Figure 5. Energy densities, e_{Rheo}, of all greases at 20 and 80 °C.

3.3. Viscoelastic Properties

The storage modulus represents the structural strength of a grease. Thereby, a low storage modulus indicates a weakly cohesive structure. This may be advantageous regarding lubricant film replenishment and friction behavior at high shear rates. Moreover, according to References [5,31], greases with a low storage modulus can be used in applications with low loads, although this was not tested in this work. In contrast, greases with high storage modulus are characterized by strong structural cohesion, which results in an improved load-carrying capacity. However, it also limits the ability of replenishing the lubrication film [5,31]. In addition to the thickener amount, Cousseau et al. [11] have concluded that the thickener type can influence the load carrying capacity of the grease; however, this was not tested in this work.

A low shear deformation at the yield point (crossover point) favors low frictional torque at startup of a machine. According to Reference [19], multiple greases with different flow limits were tested, and the starting torque was found; greases with high flow limits resulted in higher starting torques compared to those with low flow limits. Moreover, fresh greases were tested against samples that were pre-sheared. In this case, the fresh samples (which had higher flow limits due to fresh thickener) suffered from higher starting torques in comparison to their pre-sheared equivalents (in which the thickener fibers were broken down due to the pre-shearing step). However, the risk of starvation is increased since the grease may easily flow out of the bearing (in case of a poorly sealed bearing). The data that were obtained are displayed below. Figure 6 shows the storage and loss moduli of the grease samples at the beginning of the LVE range. The curves of G' and G" for lubricating greases based on synthetic oil and mineral oil are depicted in Figure 7, Figure 8 and Appendix C respectively. The comparison of shear deformation values at the crossover-point is depicted in Figure 9.



Figure 6. Storage (a) and loss (b) moduli within LVE range.



Figure 7. Measurement curves from amplitude test for Grease 1.



Figure 8. Measurement curves from amplitude test for Grease 5.



Figure 9. Crossover point.

The storage modulus increases with increasing the soap content up to 9.48%, as can be seen with Greases 2, 4, and 6. Comparable results were also reported by Cousseau et al. [9]. As for Greases 1, 3, and 5, they have comparable storage moduli, despite having different base oil viscosity. From that, one can see that, starting from a certain thickener content, the storage modulus is influenced. The thickener content has no significant influence on the relative decrease with temperature. Generally, the storage modulus of all examined greases drops with the increasing temperature.

For the loss modulus, the same correlation can be observed. In general, higher values of the storage modulus G' generally go along with higher values of the loss modulus G", regardless of the temperature. It is noticeable that grease 6 displays the highest storage and loss moduli values at all temperatures due to the fact that this grease contains the highest proportion of thickener.

When looking at the curve of Grease 1 (as in Figure 7), an increase of the loss modulus, G", occurs when leaving the LVE region. According to Mezger [26], the increasing loss modulus values are caused by relative movements within the grease structure. Thus, this behavior indicates initial irreversible changes of the grease structure. This does not cause the structure to collapse abruptly when leaving the LVE region. At first, microscopic cracks form within the structure. When the yield point is reached, they have grown into macrocracks, which arise throughout the entire shear gap. The structure of the sample is then weakened, and the grease liquefies.

By comparing the shear deformation values at the yield point, it is evident that, at this point, Grease 4 has the lowest shear deformation value at 80 °C. The crossover point between the storage and loss moduli increases with increasing the temperature from 20 to 80 °C. This was observed for all tested greases. Moreover, a higher temperature causes viscous behavior to occur at lower deformation values. These values also indicate that, with increasing soap content, the yield point is reached at higher deformations. This can be explained by the increasing structural strength with increasing thickener content. However, a high storage modulus, G', does not necessarily result in a high yield point [32].

3.4. Tack Test

In the tack test, the measured adhesion force jump represents the tackiness of a grease. The greater the adhesion force jump, the higher the tackiness. High tackiness results in a high ability to adhere on surfaces, such as the rolling elements or the raceway of a bearing. This may improve lubrication of the tribological contact and, thus, reduce friction. Since tackiness also indicates strong cohesive forces within the grease structure, extremely high tackiness may cause clumping and, therefore, rising temperature due to increased friction.

Figure 10 shows that the thickener concentration of a grease has a significant influence on adhesive behavior. In contrast to Grease 5, Grease 6 causes a higher perpendicular force in terms of magnitude. Thus, the structural strength (cohesion) and the tackiness to surfaces (adhesion) increase with the increasing soap content. This behavior can also be observed with mineral-oil-based greases (Greases 1–4). However, it is noticeable that the



threads of Grease 6 break ($F_N = 0N$) much earlier than those of Grease 5. This effect does not occur to the same extent with mineral-oil-based greases.

Figure 10. Results for greases at 20 °C (**a**) and 80 °C (**b**). Influence of soap content on tackiness of synthetic-oil-based (**c**) and mineral-oil-based (**d**) grease. Influence of base oil viscosity on tackiness. (**e**) Influence of temperature on tackiness (**f**).

The influence of increasing temperature has the opposite effect to the influence of the soap content and, thus, leads to decreasing tackiness. This can be seen from the fact that increasing the temperature leads to a decreasing discrepancy between the maximum and minimum of the perpendicular force, F_N (adhesion force jump), for all greases examined, as shown in Figure 11. Furthermore, the thread length decreases with increasing temperature, indicating a proportionality between decreasing thread length and decreasing storage



modulus, G'. By looking at the curves of Grease 1 and Grease 5 at 80 °C, the effect of the decreasing thread length due to the increasing temperature is clearly visible.

The difference between the maximum and minimum of the perpendicular force, F_N , is shown below in Figure 12. It may be observed that this adhesion force jump decreases with increasing temperature. In addition, the thread lengths of the analyzed grease samples tend to be lower at higher values of the adhesion force jump. Grease 6, for example, exhibits the highest values of the adhesion force jump at 20 and 80 °C, while showing the lowest thread length.



Figure 12. Influence of temperature on adhesive force jump (absolute values).

3.5. Squeeze Test

The measurement curves of all examined lubricating greases can be divided into three sections. The first section shows a progressive reduction of the gap height. Here, the structure of the specimen deforms elastically without reaching the yield point. During the transition from the first section to the second one, the gap height decreases noticeably, since nearly stationary flow begins. Now viscous behavior dominates, and the test medium partially is squeezed out of the measuring gap. In the third section, the perpendicular force, F_N , continues to increase, while only a degressive reduction of the gap height, d_S , is observed. This indicates that the grease remaining in the measuring gap is undergoing a work-hardening process. The measurement curves of the greases are shown in Figure 13. By comparing Grease 5 and Grease 6 at 20 $^{\circ}$ C and 80 $^{\circ}$ C, it can be seen that the soap content has a significant influence on the grease's behavior under compressive force. As the thickener content increases, the deformation work required to change the grease structure also increases. It is noticeable that a proportionality between increasing deformation work from the squeeze test and increasing storage modulus, G', from the amplitude tests is clearly visible. At 20 °C and F_N = 30 N, Grease 6 exhibits the largest remaining gap height, d. This indicates an increased tendency to work-harden with increasing soap content. In addition to that, a decrease of deformation work due to increasing temperature can be observed. The influence of an increasing temperature thus counteracts that of an increasing soap content. This behavior is also visible with mineral-oil-based greases.

Figure 11. Normal force at 20 °C and 80 °C.



Figure 13. Comparison of all greases at 20 $^{\circ}$ C (**a**); influence of temperature and soap content on forming work (**b**).

Figure 14 shows the absolute values of the work of deformation, where it can be seen that the relative drops in the work of deformation from 20 to 80 °C increase with increasing the soap content in the grease. For example, Greases 2, 4, and 6 have the highest soap content, and so they suffer a drop in the work of deformation of 46%, 43%, and 42%, respectively. On the other hand, Grease 5 had the lowest thickener content, and so it experienced the smallest reduction in the work of deformation when the test temperature was increased from 20 to 80 °C.



Figure 14. Influence of temperature on forming work (absolute values).

Figure 15 shows the comparison of storage modulus, adhesive force jump, and forming work at 20 °C. Here, for all greases, increasing the storage modulus would consequently increase the adhesive force jump and the forming work.



Figure 15. Comparison of storage modulus, adhesive force jump, and forming work.

3.6. Bearing Test

Figure 16 displays the frictional torque curves of all greases; the shades around each curve represent the standard deviation of the repeated three tests for each grease. It seems that friction torque curves correlate directly with the greases' composition.



Figure 16. Frictional torque curves.

A comparison between the friction energy densities and the frictional torque at the end of the test is illustrated in Figure 17. It can be observed that an increasing frictional torque tends to be related to an increasing frictional energy density for five greases. Grease 6 is an exception. A grease's frictional behavior is strongly dependent on the occurring interaction between the thickener and the base oil [11]. It is possible that the frictional behavior at a very high thickener content and low base oil viscosity is determined by these interactions rather than energy density.



Figure 17. Comparison of frictional energy densities and frictional torque.

As an illustration, Figure 18 shows the condition of Grease 4 before testing compared with its condition after dismantling the bearing at the end of the test, respectively. The average depth of the bearing raceway damage is depicted in Figure 19. Based on the results plotted in Figures 19 and 20, the greases that resulted in a constant and low friction torque during the bearing tests showed no damage. This is true for all greases with a base oil viscosity of 50 cSt with a thickener content up to 9.5% (Greases 1, 2, and 5). The PAO-based Grease 5 (with a thickener content of 7.5%) showed a higher friction torque at the beginning of the test compared to Grease 1. This indicates a worse starting behavior when the grease is still fresh that would decrease with time as the test progresses. Generally, large fluctuations and an overall increase of the frictional torque are an indication of severe damage to the bearing raceways, as illustrated by the experiments regarding Greases 3, 4, and 6. For low base oil viscosity, the frictional torque increases by increasing the thickener content. On the other hand, higher base oil viscosity results in higher frictional torque during the bearing test.



Figure 18. Fat sample Grease 4 at the beginning of test (**a**) and after end of the test (**b**) (detailed image of steel ball bearing).



Figure 19. Average depth of damage.

On the contrary, Cousseau et al. claimed in their work [11] that the bleed oil properties and the thickener/base oil interaction are predominant factors regarding frictional behavior of a grease-lubricated bearing. According to another study on the frictional torque by the same research group [9], the highest frictional torques are measured at the lowest speeds (100 rpm). Here, mineral greases generated the highest frictional torque among the tested greases, followed by ester-based greases and polymeric greases. At medium speeds (2000 rpm), mineral greases generated 26% and 65% higher torques than ester and polymeric greases, respectively. When the speed was increased up to 5500 rpm, mineral greases followed the same trend and generated the highest torques and consequently the highest operating temperatures. The results for the highest frictional torques for mineraloil-based greases are also shown in the measurements carried out here.

Despite the fundamental differences between the work presented here (which was obtained through oscillating tests) and that of Cosseau et al.'s research group (which was carried out under rotating conditions), their results confirm what is seen here--that a direct relation between the rheological properties of grease and the generated frictional torque is very difficult to establish since there exists no available model for such correlation. A reduced base oil viscosity would decrease the lubricant film thickness, which is equally dependent on the contact replenishment factor. This again confirms that a direct relation between the grease's rheological properties and the generated frictional torque is hard to model. It is well understood that two greases might generate different frictional torques despite having similar base oils, due to having different replenishing factors. As shown in [9], greases with low base-oil viscosities generate a low rolling torque, which might further decrease if the grease's formulation generated a better contact replenishment. For example, greases with lithium and ester-bases oils generate significant starvation [4] where greases with high flow index have higher frictional torque than those of lower flow indexes. PAO-based greases will generate lower friction than mineral oils. These findings can be confirmed on the basis of the friction torque measurements carried out.



Figure 20. Damages in rolling bearings with various greases (right column shows each failure of lower ring).

In Figures 21–23, by comparing the fresh and used greases after the riffle test, the yield point is observed to shift toward lower shear deformation. This points out that mechanically aged greases reach the yield point earlier than fresh equivalents. Moreover, there is a correlation between NLGI class and deformation at the yield point. Greases with a low NLGI class reach their yield point earlier. On the other hand, no correlation between the yield point and the base oil viscosity, as well as its type, can be observed.













4. Conclusions

In this study, the influence of rheological grease properties on the operating behavior of greases in an oscillating shear field at a small swivel angle was investigated. Rotational tests were carried out in order to determine dynamic viscosity over a wide shear-rate range and time-dependent shear stress. Moreover, oscillation measurements were performed. Thereby, the yield point, storage modulus, and loss modulus of the lubricating greases were examined. In addition to that, tackiness and the energy required to displace the grease within the rolling bearing were derived by executing rheometer measurements with a normal force buildup.

An evaluation of the rheological measurements (Figure 15) showed a proportional relation between a grease's storage modulus, its adhesion force jump from the tack test, and its forming work measured in the squeeze test. This allows us to make statements about the adhesion ability and displacement behavior of a lubricant by only performing the oscillation test, and it reduces cost, time, and effort. The tack and squeeze tests may then be neglected for a rheological classification when performing an oscillation test.

The rheological properties that are determined in rheometer tests, such as dynamic viscosity, storage modulus, yield point, adhesion force jump, and forming work, are insufficient indicators regarding the lubricating grease performance in oscillating bearings at small swivel angles, since a correlation cannot be demonstrated. However, it was shown that a thickener concentration may be used to describe the frictional behavior of a grease for the operating conditions considered in this study. Thereby, a correlation between higher thickener content and increasing frictional torque was pointed out. This also applied to an increasing base oil viscosity, which was reflected in increased frictional torque values. The same conclusion could be drawn for the friction energy density, which has shown a direct correlation to the actual bearing behavior, except for Grease 6. However, at an oscillation frequency of 7 Hz, the base oil viscosity and thickener concentration exert predominating influence on frictional torques.

In DGMK project no. 810 [19], it was pointed out that pre-sheared greases with high oil release rate, low NLGI class, and low base oil viscosity may ensure better lubrication performance at large oscillation angles and low frequencies. Similar results could be derived from rheological investigations. These correlations were demonstrated at swivel angles from 30° to 90° and oscillating frequencies up to 5 Hz. However, these correlations could not be confirmed at a smaller oscillation angle of 9.2°. This may also be explained by the higher frequency of 7 Hz, which changes the time interval for the released oil to reflow into the contact area and thus worsens lubrication performance. Consequently, it is assumed that more complex rheological relations influence the lubricating grease properties under such operating conditions.

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	Decrease (%)				
Grease Sample	Shear Rate $\dot{\gamma}_{min}$ = 0.001 1/s	Shear Rate $\dot{\gamma}_{max}$ = 17,500 1/s			
	20–80 °C	20–80 °C			
Grease 1	48.5	79.5			
Grease 2	62.9	72.9			
Grease 3	46.5	81.7			
Grease 4	40.9	80.1			
Grease 5	79.3	72.9			
Grease 6	59.9	67			

Appendix A

Appendix B







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