Susanne Hahn*
Simon Feldmeth
Frank Bauer

Assessment of the Lubricity of Grease-Sealing Rotary Shaft Seals Based on Grease Properties

Grease-lubricated sealing systems have an increased risk of starved lubrication. For this work, the lubricity of 23 greases in a rotary shaft sealing system was evaluated with a new test and evaluation method. The lubricity was then correlated with rheological and other grease properties. These grease properties are either available by the data sheet or can be measured with low effort. The results of the correlation allow a preselection of greases which are expected to lubricate rotary shaft seals well. This can support manufacturers and users in considering the lubrication of the sealing system early in the development process.

Supporting Information available online

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1 Introduction

Grease-lubricated systems have several advantages over oil-lubricated systems. They can be designed to realize a lifetime lubrication and do not require additional components like oil pumps. When sealing grease with a rotary shaft seal, the seal is usually lubricated with a small amount of the same grease that it is meant to seal. This grease is usually optimized to lubricate other tribological systems as bearings or gears [1]. However, a grease optimized for the other tribological contacts does not necessarily have to meet the requirements of the seal.

A damaged seal cannot restrain lubricant (or parts of the lubricant, e.g., bled oil) from leaking out. It also can only partly prevent pollution particles from entering the sealed area. Additionally, a starved lubricated seal itself produces particles of elastomer abrasion or oil carbon that additionally can disturb tribological contacts. The abrasion particles emerge on the air side of the sealing contact as well as on the fluid side, making the damage clearly visible to all, even if no lubricant leaks. In special applications as washing machines, the seal is to retain poorly lubricating fluids as water. Leaking water would destroy a subsequent bearing easily resulting in a total failure of the washing machine. In this case, grease can significantly enhance the lubrication of the sealing lip and serve as additional barrier to withhold the water.

The application limits of grease-sealing rotary shaft seals are significantly stricter than for oil-lubricated rotary shaft seals [2, 3] and starved lubrication occurs more frequently [4]. However, there is much less research on the sealing of grease. The investigation on grease usually concentrates on the lubrication of highly loaded, non-conforming, hard tribological contacts as they occur in bearings or gears. Consequently, there exists little knowledge about the correlation between grease properties and starved lubrication in sealing applications. This limits the possibility to consider the lubrication of the seal early in the grease-selection process. The limited research on the sealing of grease concentrates on the configuration and functionality of the sealing system [5–9] and its surroundings [10] or on a variation of single aspects of the grease [11]. The observations were mainly made on the basis of few prototype greases or on theoretical considerations. Therefore, they are only transferable to a limited extent.

Sommer [11] not only showed that thickener particles are present in the sealing contact, but also, that their shape and size strongly influence the coefficient of friction and the wear of the seal. He also found a correlation between a low minimum coefficient of friction and low wear with a low reduced viscosity (the contribution of the thickener type to the grease viscosity). A low consistency and a low thickener concentration lead to a better lubrication of the sealing contact. However, starved lubrication was also observed with greases with NLGI 1 grade [11] and semi-fluid greases [10]. Baart et al. suspect an influence of oil bleeding on the lubrication of axial seals and of the base oil viscosity (in form of a temperature depending viscosity) on the film thickness in an axial sealing contact [12, 13].

For this work, 23 different greases (described in Sect. 2) were characterized rheologically (Sect. 3) and their lubricity in a sealing system was examined on a test rig (Sect. 4). Subsequently, the rheological properties and other grease properties were correlated with the lubricity of the grease (Sect. 5). The aim of this correlation is to estimate the lubricity of a grease based on grease properties, that either can be assessed with limited effort on a rheometer or are available as established grease properties.

Susanne Hahn, Simon Feldmeth, Dr.-Ing. Frank Bauer
susanne.hahn@ima.uni-stuttgart.de
University of Stuttgart, Institute of Machine Components (IMA), Research Department Sealing Technology, Pfaffenwaldring 9, 70569 Stuttgart, Germany.

2 Analyzed Greases

For this work, the properties of 23 greases were correlated with their lubricity in a rotary shaft sealing system. The greases have an NLGI grade between under the classification (< 000) and 2–3. They possess different thickeners (Fig. 1), base oils, and they have different amounts and types of additives [14].

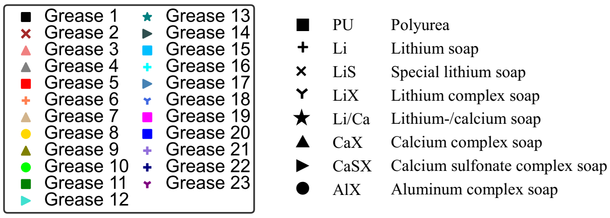

Figure 1. Symbols and thickeners of the greases.

The 23 greases are commercially available greases and not specially designed prototype greases. This makes it more difficult to retrace single influences on exact grease properties as the formulation is not exactly known. However, it makes the correlation between starved lubrication and grease properties independent of individual grease components, such as thickeners. It bases on measurements and can therefore be applied directly to a variety of greases almost regardless of their components.

The 23 greases have eight different thickeners that can be categorized into four thickener groups according to their basic substance:

- aluminum complex soap (AlX): 2 greases
- calcium soaps (Ca): 7 greases, including CaX and CaSX
- lithium soaps (Li): 7 greases, including LiS and LiX
- polyurea (PU): 6 greases

Grease 13 has a thickener combination with lithium-/calcium soap and can therefore not be clearly categorized.

In the following diagrams, each of the 23 greases is displayed with the same symbol and color. The form of the symbol indicates the thickener type as illustrated in Fig. 1.

3 Rheological Measurements

The rheological measurements provide a data basis to correlate the grease properties with the lubricity rated with the test and evaluation method, described in Sect. 4.

This section gives a brief overview of the rheological measurements carried out on the rheometer MCR 302 of the company Anton Paar and of the measuring procedures used.

3.1 Shear Viscosity

Greases are viscoelastic, non-Newtonian lubricants, whose viscosity depends, e.g., on the shear rate and must be measured specifically for individual shear rates. The shear rate at the edge of the depot area between the sealing lip and the counterface on the fluid side next to the sealing gap, illustrated in Fig. S1a in the Supporting Information, is up to several thousand s^{-1} [14, 15]. To represent the shear rate in this area, the shear viscosity is not only measured at the shear rates recommended by DIN 51810-1 [16] (500, 1000 s^{-1}) but also at significantly higher shear rates (10 000, 17 500 s^{-1}) as they occur in the depot area of the sealing system. The shear rate in a sealing gap, with a gap height of about h[1] ≈ 1 µm, may exceed several million s^{-1} [14]. The rheological behavior of greases at very high shear rates in narrow gaps, as in the sealing gap, was investigated on a specially designed rheometer by Sommer [17] and is not part of this publication.

The shear viscosity in form of the shear viscosity at the beginning, η_A, and the end, η_E, of a shear period as well as the relative shear viscosity change between beginning and end of the shear period was estimated basing on DIN 51810-1 [16]. To cover a wide range of conditions, the shear viscosity was measured not only at four shear rates (500, 1000, 10 000, 17 500 s^{-1}) but also at four temperatures (25, 60, 80, 100 °C). For the measurements a cone-plate system with a diameter of 25 mm and a cone angle of 1.0° was used. The grease sample was trimmed at a gap height of 0.062 mm before the final gap height of the measurement, h = 0.052 mm, was set.

Before starting the actual measurement, the sample was heated to the test temperature with a heating rate of 2 K min^{-1} and tempered at this temperature for another 10 min. To reduce the measurement effort, all four shear rates at the same temperature were determined according to the measuring process displayed in Fig. S1b, with the same sample. The acceleration to and measurements of the next higher shear rate (t_4 and t_5) were directly started after a rest time of 1 min (t_6) as it is displayed in Fig. S1b. A comparison between this collective measurement and individual measurements of a single temperature/shear rate measurement point is shown in [18]: the deviation is less than 13 % for η_A and less than 7 % for η_E. The measured values are displayed in [14].

As illustrated in [14, 15, 18], the shear viscosity of some greases shows a sudden drop during the measurement. This is caused by an ejection of grease out of the measuring gap and a therefore smaller volume of the sample that requires less torque to be sheared. This ejection of grease mostly occurs at low temperatures and high shear rates. Starting with the first drop of viscosity, the measured values are invalid and no longer included in the further evaluation. As the ejection of grease is repeatable in most cases, some shear viscosities cannot be measured for each grease.

3.2 Pull-off Tackiness Measurements

There is no standard definition or a standardized measurement method for the "tackiness" of a grease. Lugt et al. [1] describe the tackiness of a grease as its ability to "stick"s and name three forms of tack: cohesion, adhesion and autohesion of a material to itself. Harmon et al. [19] indicate that tackiness, as ability to form strings, and adhesion, as ability to stick to a surface, are frequently confused. A common definition of tackiness is

1) List of symbols at the end of the paper.

known by Gay and Leibler [20]: "A substance appears sticky when some work is required to remove one's finger from it." Achanta et al. [21] define tackiness as ability to form strings or threads before separation. Georgiou et al. [22] agree with that and differ between tackiness as ability to pull strings (according to the referenced "finger test") and adhesion as pull-off force to separate an indenter from grease. Achanta et al. [21] measured the pull-off force using an approach-retraction test method with a ball on which a grease substrate is pressed. When a specific load is reached, the substrate is removed from the ball, while the required normal force is measured.

The approach of Georgiou et al. [22] used a Falex tackiness adhesion analyser (TAA) to indent a 3-mm ball into a napkin filled with grease and subsequently retract it evaluating both, the pull-off force (as adhesion) and the thread formation (as tackiness). They found no correlation between adhesion and tackiness. Harmon et al. [19] developed an approach-retraction tackiness test on a tribometer to evaluate tackiness regarding the lubrication of wheels of a train. They compressed a specific amount of grease with a defined load before an upper specimen was retracted with a defined retraction speed. They varied different parameters, e.g., the compressive force, the speed or the amount of grease and compared the results to larger scaled wheel-rail grease pickup tests showing a correlation between both test methods.

The aim of the tackiness measurements in this work was the correlation with the behavior of the grease in the depot area between the sealing lip and the counterface on the fluid side of the sealing gap. There exists close to no normal force onto the grease in this area. For this work, a pull-off test with the rheometer was developed, using a parallel-plate setup with a diameter of 25 mm. The grease is put onto the lower plate and compressed by the upper plate with a speed of 0.2 mm s^{-1}, before it is retracted again, forming the typical strings that indicate the tackiness. There are several possibilities to put the grease on the plate. It was decided to put the grease on the lower plate using a spatula instead of a syringe to avoid a preshearing of the grease while application. To always measure a fully filled gap with the same amount of grease, the grease was trimmed at the gap height h_0, before retracting the upper plate with a specific speed.

In case of a force-controlled approach, it can occur that the defined normal force is reached before the grease is spread to the whole plate. Besides that, the grease can be pressed out of the gap before retraction. Both would influence the measurement results by a changed area of the wetted plate or by providing additional grease from outside the gap to form strings. Additionally, the chosen speed-controlled procedure corresponds more to the behavior of the real sealing system. Including the trimming, the upper plate remains for 60 s at the gap height, before it is retracted upwards with a specific speed v_{up}.

First, a preliminary study was conducted to evaluate test parameters, which on the one hand show differences between the greases and on the other hand provide reproducible measurement results (for a detailed description and results see [14]). After that, the tests were conducted with two different gap heights of h_0 = 0.25 mm and 1.00 mm and a speed of v_{up} = 3 mm s^{-1} at room temperature. As results, the maximum retracting force F_{max}, the gap height at which the strings tear h_{tear} (as the height from which on the normal force remains constant on the level of the gravitational force of the grease sticking to the upper plate), and the separation work W_{sep} (as the integral of the normal force and the gap height) were evaluated; see Fig. 2.

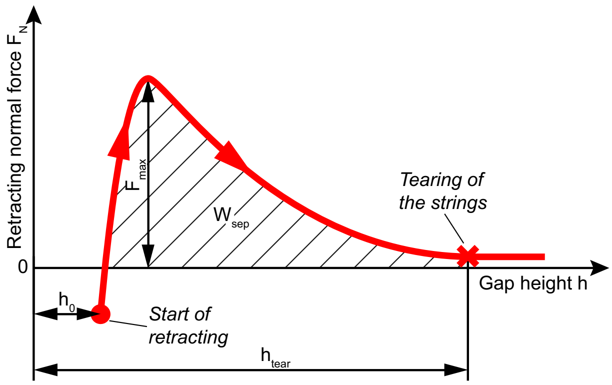

Figure 2. Pull-off tackiness measurements, schematic illustration.

3.3 Tear-In Tests

In different prior works, a tear-in of the grease film was observed in tests on a tribometer [11, 17] or in non-contact seals [23]. For this work, it was investigated, whether tear-in is reproducible with a rheometer and if the results correlate with the lubricity of a grease. On a rheometer, a tear-in is a separation of a grease sample in a moved and a static part at the according plates. This reduces the effectively sheared volume and thus the shear stress. The tear-in tests were carried out on the rheometer using the parallel-plate setup with a diameter of 25 mm. They were evaluated regarding the measured shear stress over the time or rotational speed. Additionally, a high-speed camera was used to optically estimate the flow behavior of the grease in the gap between the two plates.

After a pre-study to evaluate the principle flow behavior in the gap and to investigate whether and under which conditions tear-in occurs in a rheometer test, the tear-in tests were conducted with a gap height of h = 2 mm and a test time of t = 60 s. The rotational speed was linearly accelerated from 0 to 3000 rpm. A qualitative evaluation of the recordings of the high-speed camera combined with the measured shear stress (see Fig. 3) led to the definition of the so-called jump count A.

The jump count A describes the number of values at which the measured shear stress deviates by more than a defined threshold from the averaged shear stress, in a specified time before and after the value. Therefore, the measured values are first averaged (= averaged values). The averaged values are then subtracted from the measured values (= fluctuating values). The jump count A indicates the count of fluctuating values that are above a defined threshold. Fig. S2 shows an exemplary measurement with the measured, averaged, and fluctuating values and a threshold. A more detailed description of the tear-in tests and their evaluation is planned to be published near-time.

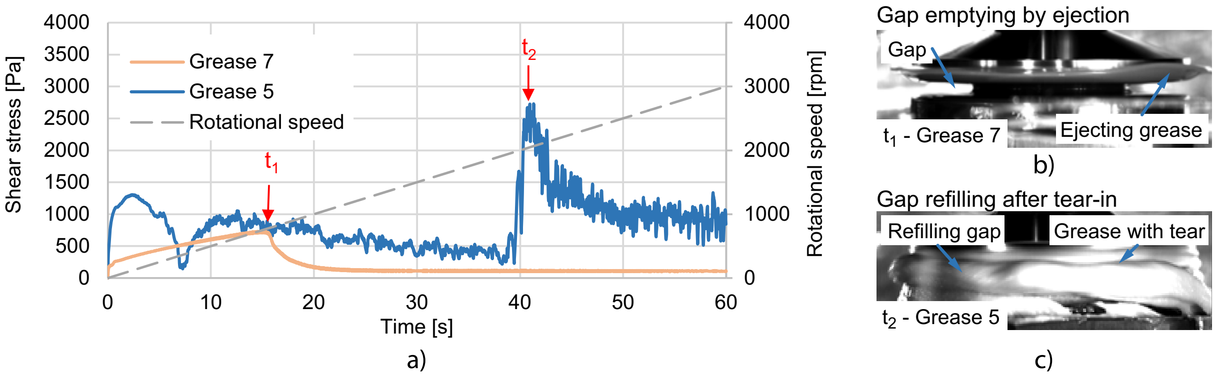

Figure 3. Tear-in tests, exemplary measurement record of grease 7 and grease 5 (a); images from the high-speed camera records (b, c).

4 Test and Evaluation Method

A test and evaluation method was developed to assess the lubricity of all 23 greases in a robust and repeatable way, described in detail in [14, 24]. To provide a data basis for a quantitative correlation with the grease properties, the test and evaluation method results in a single key indicator, the so-called overall grease score.

The test method consists of a 24-h test run on a frictional torque test rig which is shown in Fig. S3a. The test rig allows to measure the frictional torque via a load cell on which the seal holder rests. The temperature on the air side of the sealing edge is measured by a pyrometer. The test rig provides two modules whose shafts rotate in opposite direction. The sealing system is lubricated with an initial lubrication in form of a circumferential 1-ml grease ring which is applied on the counterface with a syringe. The rotary shaft seal is then pushed over the grease ring, so that the grease is located directly on the fluid side of the sealing edge, see Fig. S4. The test run contains an 8-h test cycle which is repeated three times, resulting in a total duration of 24 h for each test run, see Fig. S3b.

Starved lubrication presents itself in many different aspects [25, 26]. To assess the lubricity of the greases, eight criteria were evaluated for each test run, which proved themselves to correlate with starved lubrication [14, 24]:

- temperature fluctuation (qualitative)
- friction torque fluctuation (qualitative)
- abrasion particles (qualitative)
- grease: visual impression (qualitative)
- sealing edge: wear width (quantitative)
- wear track: visual impression (qualitative)
- wear track: depth (quantitative)
- wear track: width (quantitative)

To compare quantitatively and qualitatively evaluated criteria with each other and to summarize them to a single indicator, each criterion was rated with a grade between 1 (very good) and 5 (very bad). The evaluation of the criteria is described in detail in [14] and [24]. After the evaluation, the grades of the eight criteria were averaged arithmetically to a single test run score. For each grease at least two test runs, one in each rotational direction, were conducted and evaluated. To finally compare the greases with each other, all test run scores with the same grease were averaged arithmetically to an overall grease score N again. In Sect. 5, the grease properties are correlated with N.

Fig. 4 shows the overall grease score of each grease together with the maximum and minimum test run score in form of error bars. It is clearly visible that the test run scores of most greases only vary slightly between the tests, the differences lay mostly under 0.19. As exceptions, the greases 10, 4, and 17 show differences between their test run scores of up to 2.9, which is why they cannot be declared as starved lubricating greases but only as greases that have a higher risk of starved lubrication. Nonetheless, the test and evaluation method displays major differences between the greases and allows to identify well lubricating greases with an overall grease score below and starved lubricating greases with an overall grease score above 3. As a result, the evaluation divides the 23 greases into two groups of 17 well lubricating greases and 6 starved lubricating greases.

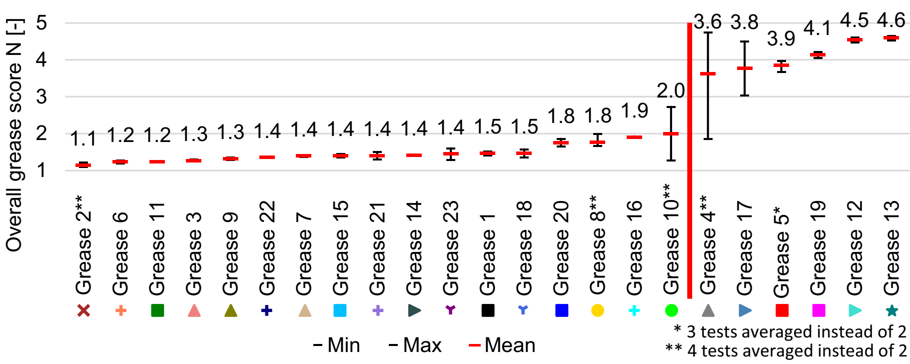

Figure 4. Assessment of the tested greases [24].

5 Correlation

For this work, the correlation of the overall grease score N or mathematical variations of N (N^2, $1/N$,…) with more than 200 measured values or other grease properties was analyzed. Of these over 2000 correlations, the most important correlations are presented in the following section. They do not only base on the described rheological measurements but also on commonly used grease properties. The detailed procedure and many additionally examined parameters or combinations of parameters are available in [14].

The following diagrams illustrate the correlation between the overall grease score N of the greases, as an indicator for its lubricity, and specific grease properties. The Pearson correlation coefficient R is used to give a number for the correlation quality. Nevertheless, it should not be overestimated because the correlation of the data is usually not linear and must be assessed qualitatively depending on the form of the individual chart.

5.1 Thickener

Fig. 5 shows the overall grease score for all thickeners, the thickeners are sorted according to their thickener group. While the 2 AlX greases and all 7 Li greases show no starved lubrication, the greases containing Ca and PU show both, starved and good lubrication. In the following section, the correlation between starved lubrication and grease properties is analyzed regarding both, all 23 greasets together and each thickener group separated. This correlation within a thickener group only makes sense, if both, good and starved lubrication, occurred within the group. According to this, grease 23 is grouped to the Ca soaps. This simplification enlarges the Ca soap group from 7 to 8 greases and enhances the correlation in this thickener group. The group-individual correlation can subsequently be analyzed for the Ca and the PU group.

5.2 Shear Viscosity Measurements

As shown in Fig. 6, greases with a low shear viscosity at the beginning of the shear time η_A lubricate well, whereas badly lubricating greases possess a high shear viscosity. However, a high shear viscosity does not mean that the lubricity is poor, as there are also some well lubricating greases with a rather high shear viscosity. While the Pearson correlation coefficient R slightly varies, the principle behavior is quite similar for the different temperatures/shear rates. The same applies to the viscosity at the end of the shear time [14]. Another reason for the different correlation coefficient is the varying amount of greases X per temperature/shear rate combination due to ejection, see Sect. 3.1.

When correlating the shear viscosity with the overall grease score within a thickener group, the correlation improves. It reaches, e.g., for a shear rate of 500 s^{-1} and a temperature of 60 °C, a Pearson correlation coefficient of up to 0.844 for the greases containing calcium as thickener (Fig. 7). Since the analysis of individual thickener groups is accompanied by a reduction in the number of greases evaluated, the statistical significance should be treated with caution.

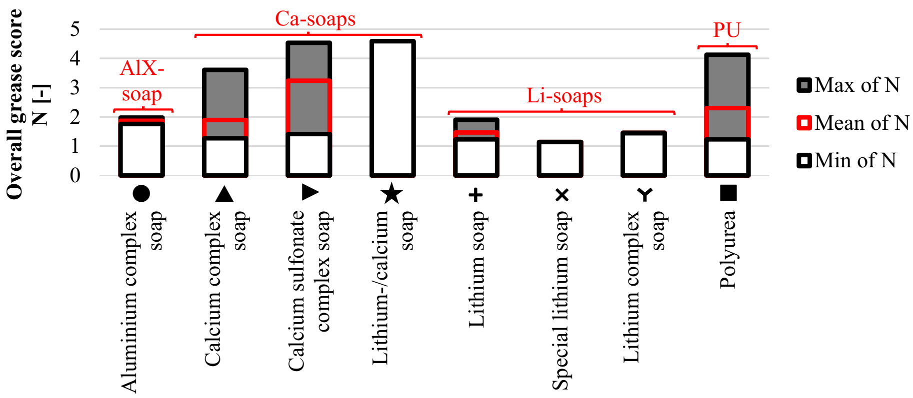

Figure 5. Maximum, averaged, and minimum overall grease score for different thickener groups.

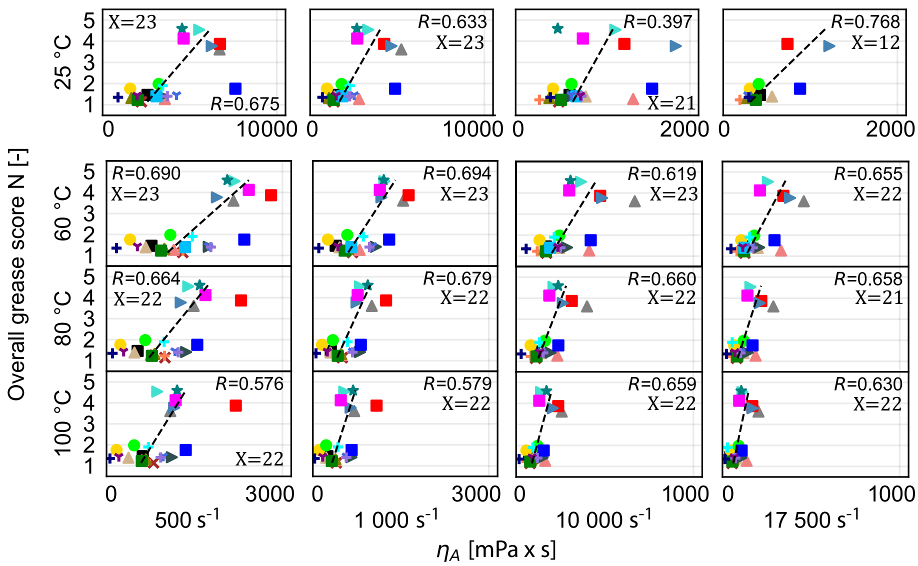

Figure 6. Correlation between the shear viscosity at the beginning of the shear time η_A (linear scale of x-axis) and the overall grease score N for the temperatures 25, 60, 80, 100 °C and the shear rates 500, 1000, 10 000, 17 500 s^{-1}.

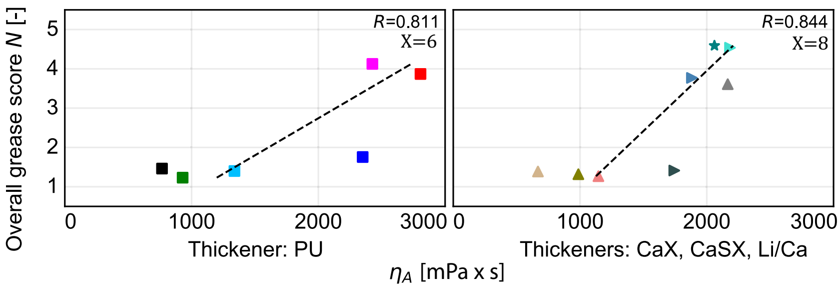

Figure 7. Correlation between the shear viscosity at the beginning of the shear time η_A and the overall grease score N for 60 °C and 500 s^{-1} for the PU and Ca thickener groups.

5.4 Tear-In Tests

The correlation of the tear-in tests was analyzed using the quantitatively evaluated jump count A. Greases with a low jump count lubricate well, whereas all starved lubricating greases possess a high jump count. But there are also some greases with a rather high jump count that lubricate well; see Fig. 10.

When looking at the correlation for the single thickener groups in Fig. 10, two things in particular strike the eye: First, the already good correlation (R = 0.803) between A and N for all greases gets even better for the Ca greases. Second, the correlation for the PU greases is again weaker than the correlation for the other greases.

5.3 Tackiness Measurements

The maximum force F_{max} and the separation work W_{sep} seem to correlate only slightly with the overall grease score ($R \leq 0.401$). However, a qualitative assessment of the correlation between F_{max} and W_{sep} for h_0 = 0.25 mm shows that greases with a low F_{max} or a low W_{sep} lubricate well, whereas greases with higher values show both sufficient and starved lubrication; see Fig. 8.

As for the shear viscosity, the correlation of the tackiness measurements is analyzed for the single thickener groups, see Fig. 9. It is remarkable that the correlation significantly gets better for the Ca thickener group. While well lubricating calcium greases show low maximum forces F_{max}, starved lubricating calcium greases have high maximum forces with a Pearson correlation coefficient of R = 0.863. However, the PU greases show no correlation between F_{max} and N.

5.5 Worked Penetration and Oil Separation

Greases with a high worked penetration P (or a low NLGI grade, [27]) show a good lubrication, whereas the starved lubricating greases have a low worked penetration (or a high NLGI grade). However, greases with a low worked penetration can also lubricate well. Greases with a high oil separation M according to ASTM D 6184 [28] likewise exhibit good lubrication, whereas the starved lubricating greases have a low oil separation. Here, it must be noted that the data basis with only two starved lubricating greases is very low. The correlation is pictured in Fig. S5.

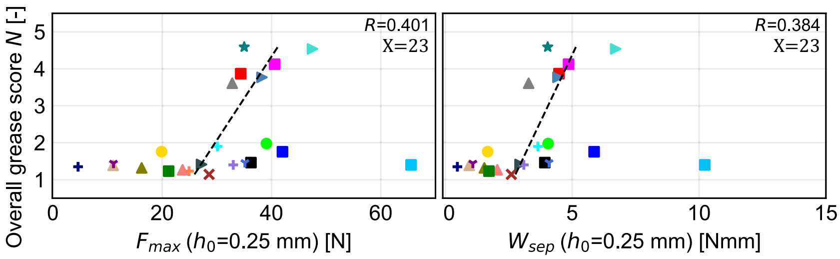

Figure 8. Correlation between the maximum force F_{max} respectively the separation energy W_{sep} for a gap height of h_0 = 0.25 mm and the overall grease score N.

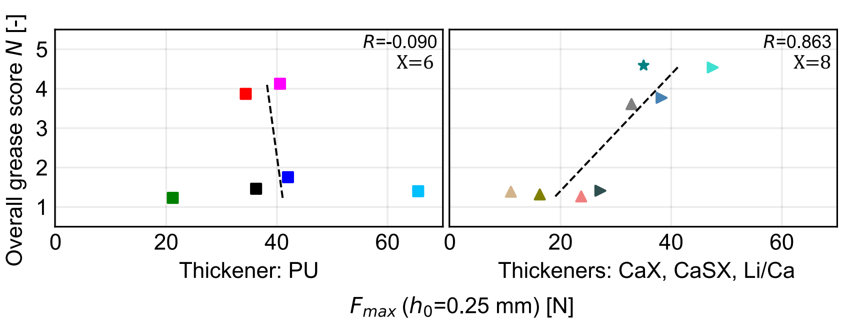

Figure 9. Correlation between the maximum force F_{max} for a gap height of h_0 = 0.25 mm and the overall grease score N for the PU and Ca thickener groups.

5.6 Viscosity of the Base Oil

The kinematic viscosity ν of the base oil at 40 °C does not correlate with the overall grease score as can be seen in Fig. S6.

6 Summary and Conclusion

Starved lubrication is a common problem with grease-lubricated rotary shaft seals. The grease is often optimized for other tribological contacts, e.g., bearings, and the lubrication of the seal is considered with lower or no priority. Knowledge about grease properties that usually correlate with a good lubrication of seals is rare. This makes it difficult to select a grease which is also suitable for the sealing contact.

For this work, the lubricity of 23 commercially available greases was evaluated in test runs with a real sealing system and compared with rheological or other tribology-related grease properties. It was not possible to find a single grease property that directly correlates with starved or good

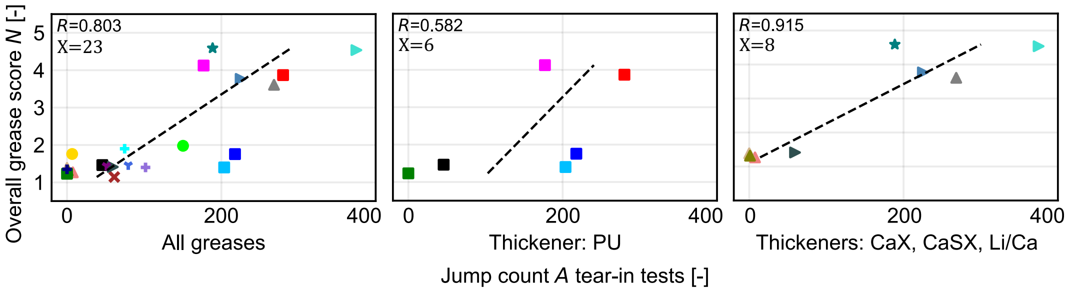

Figure 10. Correlation between the jump count *A* and the overall grease score *N*.

lubrication. The correlation often varies for the single thickener groups. Anyway, there are connections between different grease properties and the lubrication condition. Together, they can be used to estimate the lubrication condition of a grease. In particular, the following conclusions can be drawn:

- Of the 23 greases, the 2 AlX greases and the 7 Li greases (Li, LiS, LiX) showed no starved lubrication. The 8 greases containing Ca thickener (CaX, CaSX, Li/Ca) and the 6 PU greases displayed both starved and good lubrication.
- Greases with a low shear viscosity usually lubricate well, whereas starved lubricating greases normally possess a high shear viscosity. The correlation improves when the correlation is evaluated within one thickener group.
- Greases with a low maximum force or a low separation work in pull-off tackiness measurements usually lubricate well, whereas starved lubricating greases have a higher maximum force and a higher separation work. This correlation improves for the Ca-containing greases, whereas for PU greases there is no correlation visible.
- The best correlation was found for the self-developed tear-in tests: greases with a low jump count lubricate well, whereas most greases with a high jump count cause starved lubrication. This correlation improves for the Ca-thickened greases and slightly decreases for the PU greases.
- While greases with a high worked penetration or a high oil separation (low data basis) lubricate well, no correlation was found between the viscosity of the base oil and the lubricity of the grease.

This work provides new knowledge about which grease characteristics correlate with a good or starved lubrication of a rotary shaft sealing system. This knowledge allows to consider the sealing system earlier in the development process by preselecting greases with a high probability to lubricate the sealing contact well. This can reduce the development effort and also prevent sealing system failures due to starved lubrication.

Supporting Information

Supporting Information for this article can be found under DOI: https://doi.org/10.1002/ceat.202200382.

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Symbols used

A	[–]	jump count
F_{max}	[N]	maximum force
h	[mm]	gap height
h_0	[mm]	gap height at the beginning of retraction
M	[mass %]	oil separation
N	[–]	overall grease score
P	[mm]	worked penetration, 0.1
R	[–]	Pearson correlation coefficient
t	[min, s]	time
v_{up}	[mm s^{-1}]	retracting velocity
W_{sep}	[N mm]	separation work
X	[–]	amount of included values/greases

Greek letters

$\dot{\gamma}$	[s^{-1}]	shear rate
η_A	[mPa s]	shear viscosity at the beginning of a shear period
η_E	[mPa s]	shear viscosity at the end of a shear period
$\nu_{base\ oil}$	[mm^{2}s^{-1}]	kinematic viscosity of the base oil

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