

No.94 page 1

Upper Operating Temperature of Grease: Too Hot To Handle?

Introduction

The definition of high temperature performance for lubricating grease needs to be divided into two parts. The upper temperature limit which can be defined as the temperature at which the grease can function for a very short period of time. And the upper operating temperature which states the highest temperature for which lubrication can be maintained for a longer period of time.

In general there seems to be a very large difference in how manufacturers determine the upper operating temperature. Looking at product data sheets for similar mineral oil based lithium EP2 greases we find upper operating temperatures range from 120 to 250°C. In a fairly recent paper Coe discussed the inconsistency in how the industry reports the upper operating temperature and showed that different tests may lead to different conclusions concerning the upper operating temperature (1).

In Europe two standards are generally accepted to classify lubricating grease: DIN 51502 and ISO 6743-9 (2,3). ISO 6743 specifies that the operating temperature should be determined by either a grease-life test according to ASTM D3336 or a FAG FE-9 test. DIN 51502 describes that the upper operating temperature should be determined by either a SKF R2F-B or FAG FE-9 test.

Lugt describes the use of the SKF ROF- or SKF R2F-test for determining the High Temperature Performance Limit, which he defined in a similar manner as the above-mentioned definition of the upper operating temperature (4).

Not all grease types are fit to run in these tests. The softer greases (NLGI 0 and lower) will tend to leak out of the bearing during the test, causing the test to fail not because of grease degradation but merely as a direct consequence of the greaseleakage. Other greases might have excellent high temperature stability, but may not lubricate properly in these type of bearing tests.

In this article the author will describe a combination of high temperature tests under suitable conditions which may be used to develop an alternative to determining the upper operating temperature of lubricating greases.

Grease Degradation

In order to be able to define an alternative test or combination of tests that might be used to determine the upper operating temperature for those greases that cannot be run in either the R2F-B- or the FE-9 test, we need to understand the parameters that play a role in grease degradation and the effect it has on the properties of the grease.

Cann et al showed that the oxidation of grease in rolling bearings varies with the location of the grease in the bearing and that degradation of the grease does not necessarily have a negative influence on the lubricity, but that some degree of degradation of the grease will facilitate track replenishment (5,8). For example several authors identified oil loss through evaporation as a critical process leading to loss of lubricity and eventually bearing failure (6,7). Komatsuzaki et al found that lithium thickened greases lose their lubricity in roller bearings when the grease loses 50-60% of its base oil (7). Another study showed that besides base oil oxidation, oil evaporation and thickener degradation, the anti-wear/boundary properties also play an important role in bearing failure (8).

Experiments

A selection of 7 commercially available greases (see table 1) were compared in a number of tests.

Code	Thickener	Oil Type	NLGI Grade
Grease 1	Lithium	Mineral	2
Grease 2	Lithium complex	Synthetic	2
Grease 3	Calcium sulfonate complex (CaSX)	Mineral	2
Grease 4	Bentonite Clay	Synthetic	2
Grease 5	Anhydrous Calcium	Mineral	2
Grease 6	Lithium-Bismuth Complex	Mineral	1.5
Grease 7	Polypropylene	Synthetic	1.5

Table 1. Description of tested greases

The oil separation and oil evaporation of these greases were tested according to ASTM D6184 (30 hours at 100°C). The oxidation stability of these greases was compared in a PDSC,

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No.94 page 2

where the oxidation onset temperature (OOT) was determined according to ASTM E-2009, method B (under 3.5 MPa oxygen pressure).

Besides these standardized tests, the greases were tested on an Anton Paar rheometer (MCR 301) with a rolling bearing assembly (RBA-test) (see figure 1 (left)). SKF 6204-2Z/C3VM104 bearings were half-filled with the test-greases using a syringe. A rubber ring was placed between the rheometer and the bearing in order to keep the bearing in place during the test. During the measurement a Peltier hood was lowered over the bearing in order to guarantee a controlled temperature throughout the bearing.

After a controlled run-in procedure the bearings were tested in a speed ramp test from 0.01-1000 RPM with an axial force of 50 N at the test temperature. In this study two temperatures were tested: 120°C and 140°C. These are common temperatures for the SKF R2F-B test and are both limits in the DIN 51502- and ISO 6743-classification.

The bearing was then placed on top of an upside down glass funnel (see figure 1 (right)) in a pre-heated oven at the test temperature. After 1, 2 and 3 weeks of static ageing, the bearings were taken out of the oven and the amount of oil loss was determined by comparing the weight of the funnel and bearing with the weight at the start of the test. Next the bearings were re-tested on the rheometer in the same procedure as at the start of the test. The speed ramp curves before and after ageing were compared with each other. Failure of the bearing was defined as the moment when the rheometer no longer could rotate the bearing. The torque limit for the rheometer that was used in this study was 200 mNm.

After 3 weeks of ageing (or as soon as the bearing failed) the bearings were opened. The remaining grease was removed from the cage and tested with a RULER test according to ASTM D7527 in order to determine the remaining amount of antioxidants in the grease.

The result of the RULER test is presented as RUL%, which represents the percentage of the amount of anti-oxidants from the fresh grease that is still present in the grease after the test.





Figure 1. Rheometer with Rolling Bearing Assembly (RBA) (left) and Ageing Set-Up (right).

Results and Discussion

The results of the oil separation / oil evaporation test (ASTM D6184) and the oil loss calculations from the RBA-test are presented in table 2.

These results show that the standard test method of ASTM D6184 alone is not sufficient to get an insight on the amount of oil loss at elevated temperatures inside a bearing. The clearest example is grease 7 which shows the highest oil loss in the ASTM D6184 test, but shows a significantly lower oil loss compared to the other greases when heated to 120°C and 140°C inside a bearing.

Another example is grease 5, which show the lowest oil loss in the ASTM D6184 test, but has a very strongly increasing oil loss at 120°C and 140°C inside the bearing. This can be easily understood from the fact that the dropping point of this grease type is very close to 140°C.

Table 3 shows the results of the oxidation stability tests with the PDSC as well as the amount of anti-oxidants that are present after the RBA-test.

Code	ASTM D6184	Oil Loss from bearing in RBA-Test (presented as % from original oil content in formulation)					
	30h@100°C	168h@120°C	336h@120°C	504h@120°C	168h@140°C	336h@140°C	504h@140°C
Grease 1	3.0% / 0.7%	11.7%	31.7%	65.0%	25.0%	32.5%	1
Grease 2	1.8% /0.5%	7.4%	10.2%	17.6%	14.8%	21.3%	31.4%
Grease 3	3.1% / 0.9%	11.5%	16.8%	18.9%	25.2%	36.7%	43.0%
Grease 4	1.3% / 0.9%	8.8%	13.2%	15.8%	22.8%	29.9%	36.0%
Grease 5	0.6% / 0.6%	11.8%	16.3%	20.0%	75.3%	1	1
Grease 6	4.4% / 0.2%	9.7%	12.4%	15.1%	26.6%	37.2%	1
Grease 7	4.3% / 0.7%	3.5%	4.3%	6.9%	11.2%	19.8%	22.4%

Table 2. Oil Separation / Evaporation (ASTM D6184) and Oil Loss from the RBA-test.

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No.94 page 3

From both the PDSC- and the RULER- measurement the CaSXthickened grease (grease 3) clearly shows the best performance. Even though the amount of anti-oxidant in the formulation of grease 3 is comparable to the other greases, as much as 49% of the original amount of anti-oxidants is left after 3 weeks at 140°C.

Code	ООТ	RUL%,120°C	RUL%,140°C	
Grease 1	207°C	1	1	
Grease 2	222°C	32%	8%	
Grease 3	236°C	73%	49%	
Grease 4	208°C	80%	24%	
Grease 5	220°C	²	2	
Grease 6	232°C	54%	1	
Grease 7	214°C	20%	3%	

Table 3. Oxidation Onset Temperature (OOT) and remaining anti-oxidants after the RBA-tests (RUL%, 120°C and RUL%, 140°C).

The lithium thickened grease (grease 1) and the clay-thickened grease (grease 4) show the poorest oxidation stability in the PDSC test. However, these greases behave completely different in the RBA-test. Where grease 1 fails after respectively 3 weeks at 120°C and 2 weeks at 140°C, the bearings filled with grease 4 passes both the 120°C and 140°C RBA-test after 3 weeks.

The results from the RBA-test can be found in figures 2 and 3. The general behaviour for all greases in the RBA-test can be divided into two stages. In the first part of the ageing process the torque is equal or lower to the torque for the fresh grease. The lower torque could be caused by the oil that has bled out of the grease during this time at elevated temperature, which might improve the flow towards the raceway.

In the second stage the torque starts to increase again. eventually leading to failure of the bearing. The increasing torque in the second stage may have been caused by oxidation and polymerization of the base oil and/or oil loss from the bearing leading to starvation.

The torque for grease 2 after 2 weeks of ageing at 120°C is higher than expected based on the behaviour of the same grease at 140°C. It is believed that this measurement is an artefact and should not be taken into account.

Grease 3 shows very consistent behaviour both at 120°C and at 140°C. The speed ramp is basically unaffected by the ageing process. Together with the oxidation stability results from table 3, this is an indication of the excellent high temperature performance of this grease.

Grease 4 also shows very consistent behaviour during the ageing process. Only after 3 weeks at 140°C the torque starts to increase significantly, indicating that the bearing is on the verge of failure.

Grease 5 shows a big difference between the test at 120°C and the test at 140°C. Where the grease shows excellent behaviour after 3 weeks of ageing at 120°C, the bearing failed after just 1 weeks ageing at 140°C. The main cause of this failure has been the extremely high oil separation at this temperature caused by the fact that the temperature approaches the dropping point of this grease.

Grease 7 was the only grease that did not show a typical Stribeck-like decency on the rotation speed. For this grease a peak at around 10 RPM is seen which decreases during the ageing process. Grease 7 contains an elastomer which is present in the formulation in order to improve the mechanical stability of the greases. Grease 7* in figure 3 is the exact same formulation but excluding this elastomer. As Grease 7* does show the typical Stribeck-like dependency on the rotation speed, the deviation at 10 RPM for the measurements on Grease 7 may well be related to the elastomer.

^{1:} Not sufficient material could be retained after the RBA test to perform an accurate RULER-test.

^{2:} Formulation does not contain anti-oxidant.



No.94 page 4

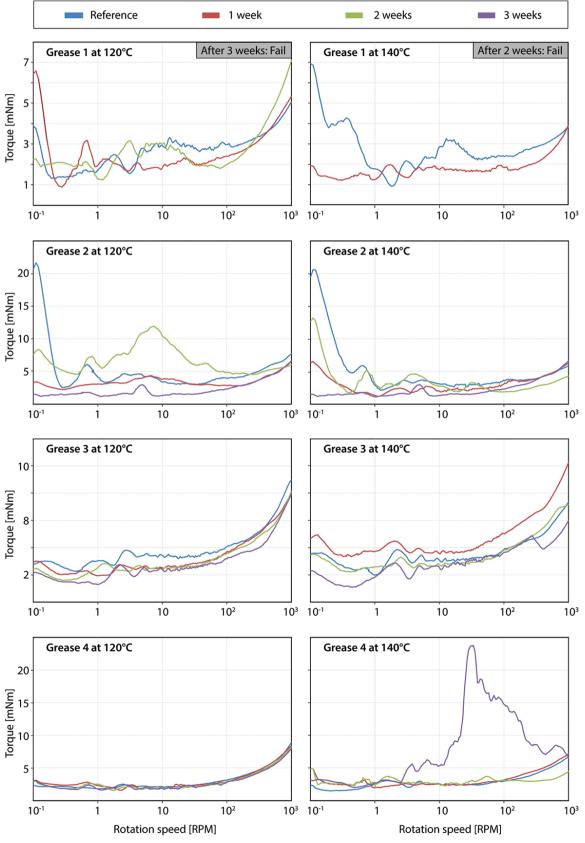
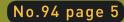
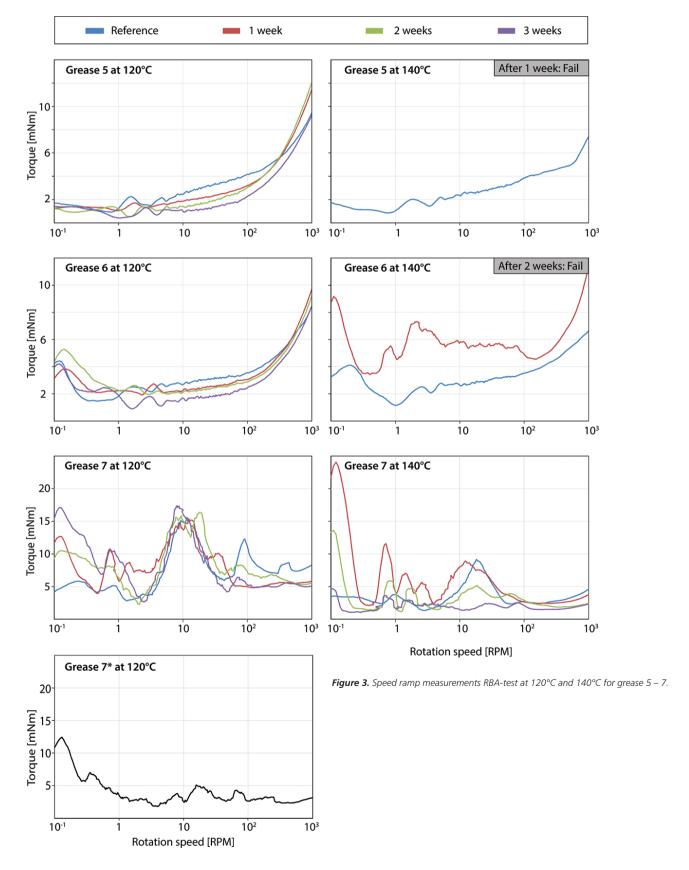


Figure 2. Speed ramp measurements RBA-test at 120°C and 140°C for grease 1 − 4.









No.94 page 6

Code	ООТ	Oil Loss 120°C RBA test	Oil Loss 140°C RBA test	RBA test at 120°C	RBA test at 140°C	SKF R2F-B Pass temperature
Grease 1	+/-	-/-	-	+/-	×	120°C
Grease 2	+	+	+/-	++	++	140°C
Grease 3	++	+	-	++	++	150°C
Grease 4	+/-	+	+/-	++	+	140°C
Grease 5	+	+/-	-/-	++	-/-	120°C
Grease 6	++	+	+/-	++	-	150°C
Grease 7	+/-	++	+	+	++	140°C

Table 5. Schematic overview of test results

Table 5 shows a schematic overview of the test results as well as the temperature at which the tested greases passed the SKF R2F-B test.

The results from the RBA tests at both 120°C and 140°C are well in line with the temperature at which the greases pass the SKF R2F-B test which is used to determine the upper operating limit in DIN 51502. The only grease that did not confirm the SKF R2F-B test result was grease 6. This may be related to oil loss from the grease, but will have to be investigated further.

Conclusions

The behaviour of various grease types during high temperature ageing has been investigated by several laboratory tests.

Rheological tests with a rolling bearing assembly showed two general stages during static ageing of the grease inside a ROFbearing. During the first stage of the ageing process the oil bleed may cause a reduction of the torque. During the second stage of the ageing the torque increases, eventually leading to failure.

The oxidation stability of the grease has not been the determining factor in the high temperature performance of the various greases. Oil loss from the bearing has played a far more important role in the process that causes failure. The critical amount of oil loss varies for each thickener type.

This work has shown clearly that the standardized oil separation test at 100°C for 30 hours (ASTM D6184) does not always lead to a good understanding of the oil loss at the actual temperature the grease will be exposed to during application.

The oil loss determination from the statically aged bearings showed a more realistic picture for the temperatures that are of interest when the upper temperature limit is to be determined. In addition the rheological measurements with the rolling bearing assembly give a good understanding of how various grease technologies behave when exposed to elevated temperatures for a prolonged time.

Although this research is in an early phase, the first results indicate that it may be possible to develop an alternative method of determining the upper operating temperature using rheological measurements with a rolling bearing assembly. This alternative method might be used as a complement to the established SKF R2F-B- and FAG FE-9 tests.

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