

Yield Stress and Low-Temperature Start-Up Torque of Lubricating Greases

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Received: 8 February 2016 / Accepted: 9 May 2016 / Published online: 20 May 2016
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Abstract A comprehensive study of the yield stress and start-up torque for six commonly used rolling bearing greases is presented. Both parameters were found to depend exponentially on temperature where the exponent changes below a low-temperature “break point.” This break point was found to be related to the pourpoint of the base oil, although the start-up torques of the greases were an order higher in magnitude than that of their corresponding base oils. The start-up torque is mostly used to measure the low-temperature limit of a grease. It was found here that this temperature is much lower than the break point. The start-up torque criterion is measured using a particular bearing type and conditions. The low-temperature break point for the yield stress is a more universal grease parameter that gives useful information about the behavior of a grease at low temperatures and can be used as one of the guidelines for grease selection for low-temperature applications.

Keywords Rolling bearings · Low-temperature limit · Arrhenius behavior

List of symbols

b Arrhenius parameter
 D Diameter of the thickener particle (m)
 F_N Normal load (N)

ω Frequency (Hz)
 G' Elastic modulus (Pa)
 G'' Loss modulus (Pa)
 L Length of the thickener particle (m)
 R Radius of the parallel plate (m)
 R_a Roughness parameter (m)
 T Temperature (°C)
 T_b “Break point” temperature (°C)
 T_0 Reference temperature (°C)
 t Time (s)
 ΔT Change in temperature (°C)
 γ Strain
 γ_{cr} Critical strain
 τ_y Yield stress (Pa)
 τ_{y0} Yield stress at $T = T_0$ (Pa)

1 Introduction

Lubricating greases are structured materials consisting of a dispersion of thickener within a suitable base oil [1]. The performance of the lubricating grease in rolling bearings depends strongly on the grease rheology, internal bearing geometry, and transient operating conditions. At very low temperatures, the grease may become so stiff that it causes high resistance to the motion of the rolling elements [2]. This may lead to skidding, damaging the bearing. The stiffening will be different for different greases, which is translated into a so-called low-temperature limit (LTL) below which a bearing should not be operated. Measuring the onset of damage is very difficult. Therefore, in practice the start-up torque is used as a criterion for grease stiffening. The LTL is then defined as the temperature at which

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the start-up torque exceeds a predefined value of say 1000 mNm in a standardized test setup [3].

Lubricating greases are viscoelastic materials up to a yield point where plastic deformation occurs. Earlier work suggests that the start-up torque is related to the shear stress experienced by the grease during start-up and is related to the yield stress [4, 5]. Edmund [5] showed that there is a linear relation between start-up torque and yield pressure for lubricating greases. Kobzova et al. [6] also assert the importance of the yield stress on the start-up torque.

In an earlier paper [7], the authors studied the temperature dependence of the yield stress from oscillatory strain sweep measurements for six greases that represented the most commonly used thickener—oil systems. The same greases were employed in this study (Table 1). At a temperature below zero degrees Celsius, a deviation from “Arrhenius behavior” was observed for those greases based on mineral and semisynthetic oil [7]. A “break” in the Arrhenius plot characterized by a change in slope is known to be caused by a change in phase, as shown by Kumamoto et al. [8] for biological systems. This change in slope causes a rapid increase in yield stress below a critical temperature.

In this study, yield stress measurements were taken over a wider range of temperature than in [8], and these measurements will be compared to the start-up torque. It will be shown that the “break” in the Arrhenius plot can be

associated with the pourpoint of the bled oil. The low-temperature break is a grease parameter and, other than the LTL, not measured with a particular bearing. Therefore, it gives useful general information on the behavior of a grease at low temperatures which is generally applicable to any bearing.

2 Materials and Methods

2.1 Tested Greases and Bled Oils

Six greases formulated from the most common thickeners and base oils were tested (Table 1). The bled oils were extracted from the greases by using Heraeus Biofuge 17RS centrifuge [9].

2.2 Rheological Characterization

Rheological characterization was carried out using oscillatory strain sweep. These measurements were taken on a controlled strain rheometer (MCR 501, Anton Paar) using sandblasted parallel stainless steel plate geometry ($R_a = 2.87 \mu\text{m}$, $R = 12.5 \text{ mm}$ and 1 mm gap setting). The oscillatory strain sweep measurements were taken using a strain ranging from 0.001 to 1000 % at a frequency of 1 Hz. A relaxation time of 20 min was considered sufficient for the sample to relieve the residual

Table 1 Properties of lubricating greases studied

Grease	NLGI	Thickener	Thickener concentration	Shape and size of the thickener particle	Base oil	Viscosity of the base oil (mm^2/s) (40/100 °C)	Consistency (60 strokes, 0.1 mm)
CaS/MS	1–2	Calcium sulfonate complex	26	Spherical particles $D \approx 0.26 \mu\text{m}$	Synthetic (PAO)/Mineral	80/8.6	300
CaS/M	2	Calcium sulfonate complex	27	Spherical particles $D \approx 0.26 \mu\text{m}$	Mineral oil	420/27	275
Li/M	3	Lithium 12-hydroxy stearate	15	Twisted fibers $L \approx 2 \mu\text{m}$ $D \approx 0.1 \mu\text{m}$	Mineral oil	99.9/10	207
Li/SS	2	Lithium 12-hydroxy stearate	17	Twisted fibers $L \approx 2 \mu\text{m}$ $D \approx 0.1 \mu\text{m}$	Mineral oil (semisynthetic)	41.9/7.5	270
Li/PAO	2–3	Lithium complex	20	Fibers $L \approx 0.4 \mu\text{m}$ $D \approx 0.1 \mu\text{m}$	Synthetic PAO	191/42	255
PU/E	2–3	Polyurea	26	Platelets $L \approx 0.65 \mu\text{m}$	Synthetic ester	70/9.4	283

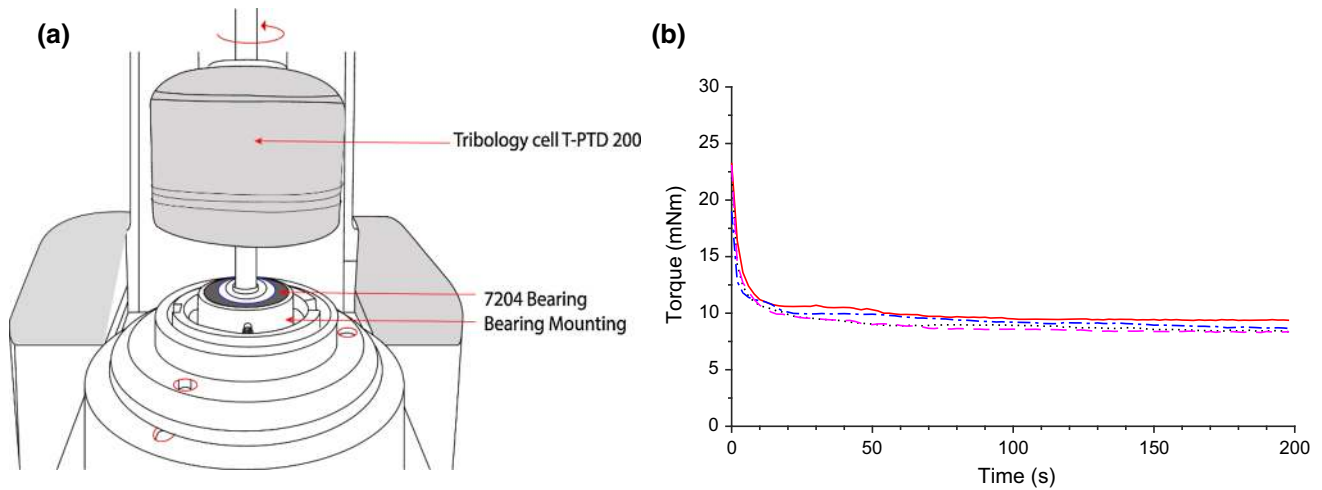


Fig. 1 **a** Bearing start-up torque measuring setup, **b** reproducibility of pre-shearing using oscillatory strain sweep for a representative grease at $T = 25\text{ }^{\circ}\text{C}$

Table 2 Pre-shear testing conditions

γ (%)	ω (Hz)	F_N (N)	T ($^{\circ}\text{C}$)	t (s)
0.5	0.5	44.1	25	200

stress, before taking the measurements. Experiments were carried out in the temperature range of -50 to $50\text{ }^{\circ}\text{C}$ and at a precision of $\pm 0.5\text{ }^{\circ}\text{C}$. The measurements were found to be reproducible with a maximum error of $\pm 5\%$.

2.3 Start-Up Torque Measurements

The start-up torque of the lubricating greases and corresponding bleed oils were measured using a controlled strain

rheometer (MCR 501, Anton Paar) fitted with tribology cell T-PTD 200 (Fig. 1a). The measurements were taken using the IP 186 standard testing procedure in the temperature range of -30 to $50\text{ }^{\circ}\text{C}$. An axially loaded angular contact ball bearing with detachable outer ring (SKF 7204 BEP), rotating at 1 rpm, was used.

After loading the weighted quantity of grease as per the IP 186 standard, pre-shearing was performed to achieve a uniform distribution of grease inside the bearing. The pre-shearing was carried out using oscillatory strain sweep, and the test conditions are summarized in Table 2. This was analogous to manual pre-shearing in the IP 186 standard. The measurements were found to be reproducible with a maximum error of $\pm 10\%$ (Fig. 1b). After pre-shearing, a relaxation time of 20 min was allowed before performing the torque measurements.

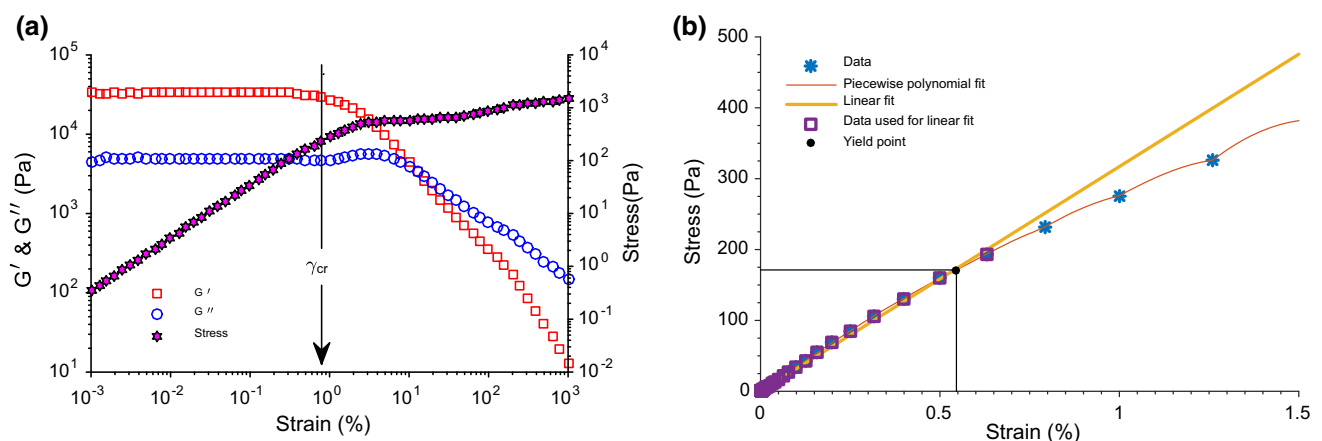


Fig. 2 **a** Storage and loss modulus versus strain and **b** yield stress obtained from oscillatory strain measurement for a representative grease

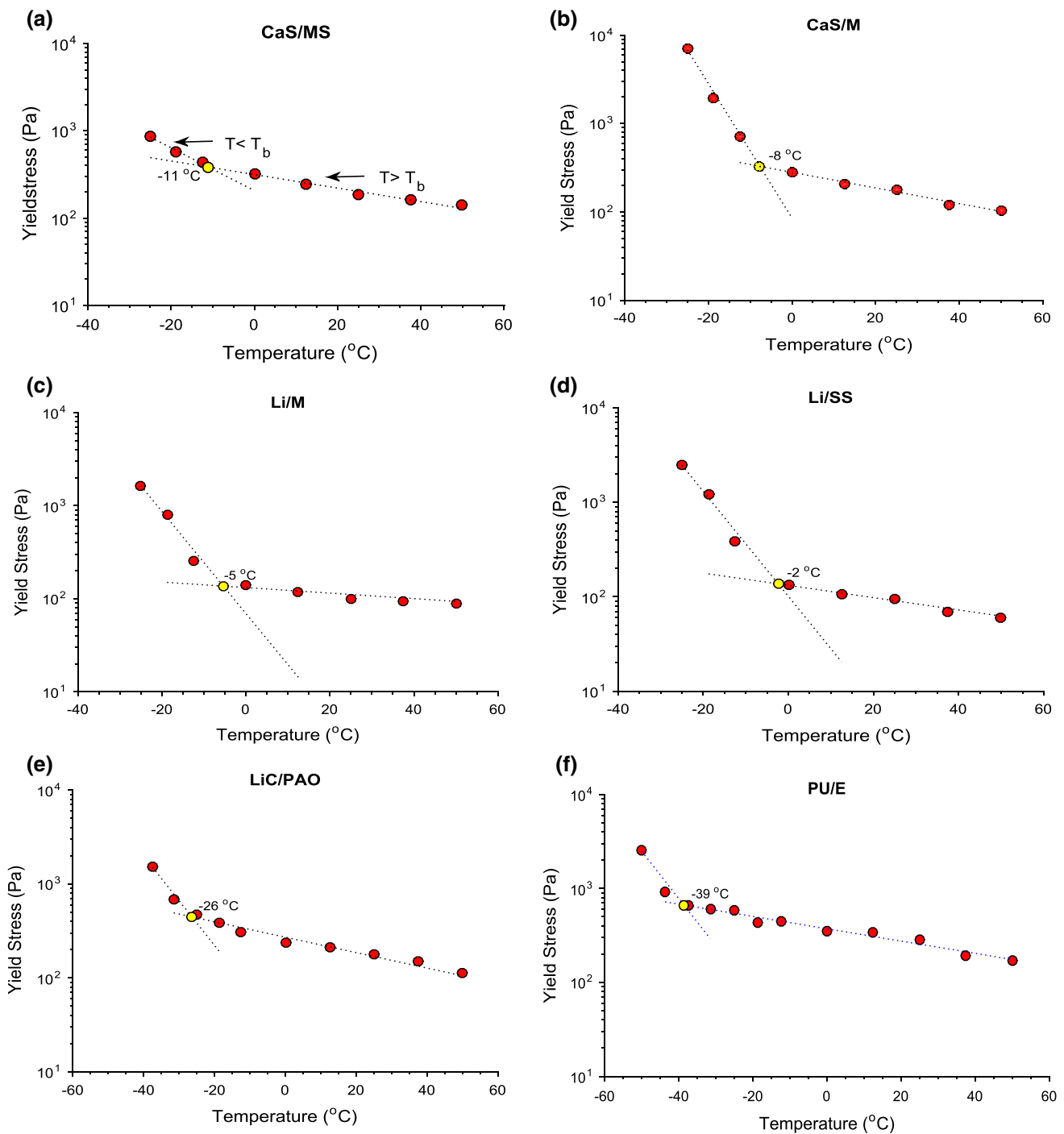


Fig. 3 Yield stress obtained from oscillatory strain sweep versus temperature. The yellow dot denotes the “break point,” which is the intersection point of two straight lines (Color figure online)

The start-up torque measurements were taken at an axial load of 44.1 N for a duration of 60 s and at a sampling frequency of 50 Hz. Temperature control was achieved

using a Peltier device offering a precision of $\pm 0.1^{\circ}\text{C}$. The maximum error associated with the measurements was found to be $\pm 12.5\%$.

Table 3 Parameter “ b ” obtained using Eq. (1) for the yield stress. The temperature corresponding to “break” in the Arrhenius plot, “ T_b ” is also shown

Grease	T_b (°C)	b ($T < T_b$)	b ($T > T_b$)
CaS/MS	−11	10.83	35.2
CaS/M	−8	3.57	34.1
Li/M	−5	5.81	78.4
Li/SS	−2	5.55	46.2
LiC/PAO	−26	5.26	36.6
PU/E	−39	5.10	39.9

2.4 Pourpoint Measurements

The pourpoint of the bled oils was obtained using the ASTM D 97 standard test procedure. In this test, the specimen in a jar is cooled and periodically examined for any movement of oil for every 3 °C decrease in temperature. The pourpoint is obtained by adding 3 °C to a temperature corresponding to “no flow” when the jar is held horizontally for 5 s.

3 Results

3.1 Rheological Characterization

In an earlier paper, the authors studied the yield stress of the same greases as used in this study, using various techniques [7]. They preferred oscillatory measurements over continuous shear for estimating the yield stress as the preceding method was found to be less sensitive to wall slip. The viscoelastic properties of the lubricating greases can be quantified using storage (G' , elastic component) and loss modulus (G'' , viscous component). Below a critical strain amplitude (γ_{cr}), the structural integrity of the network is maintained, and the material parameters (G' and G'') are found to be independent of the imposed strain (Fig. 2a). However, above γ_{cr} , higher strain energy may cause structural damage. As a result, both G' and G'' decrease and the material behaves as if it is a liquid. However, for lubricating greases, there is no sharp transition from solid-to liquid-like behavior. As a result, the yield stress is defined here as the stress corresponding to a predefined (0.5 %) deviation from the linear relation between stress and strain (Fig. 2b). In rolling bearings, the timescale of the flow problem varies. So, the prediction of yield stress

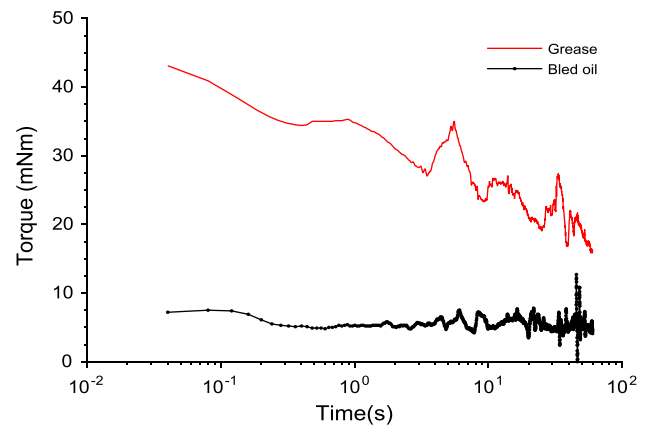


Fig. 4 Torque versus time for Li/SS grease and corresponding bled oil at $T = -18.75$ °C

should ideally take place over a wide frequency range. It was shown earlier that the yield stress varies linearly with the imposed frequency. In this study, an arbitrary frequency of 1 Hz was chosen.

3.2 Temperature Dependency of the Yield Stress

The yield stress versus temperature on a semilog scale is shown in Fig. 3. The yield stress was found to exhibit Arrhenius behavior, i.e., it varies exponentially with temperature. The yield stress versus temperature is conveniently written as:

$$\tau_y = \tau_{y0} \left(\frac{1}{2} \right)^{\left[\frac{T - T_0}{b} \right]}, \quad (1)$$

where τ_{y0} is the yield stress at temperature $T = T_0$.

In this equation, the yield stress halves for a temperature increase of $\Delta T = b$. This is more practical than using an activation energy and gas constant. The parameter, “ b ,” obtained using Eq. (1) is listed in Table 3.

A significant deviation from Arrhenius dependency was exhibited by all the grease samples when cooled below a certain temperature, characterized by a “break” in the curve. The temperature corresponding to the “break” is here termed T_b . Below this temperature, a second exponential constant b was found to apply where the sensitivity to a change in temperature is much larger for all greases (Table 3). Another important observation is the much lower T_b for greases based on PAO and ester oils in comparison with those based on mineral oil.

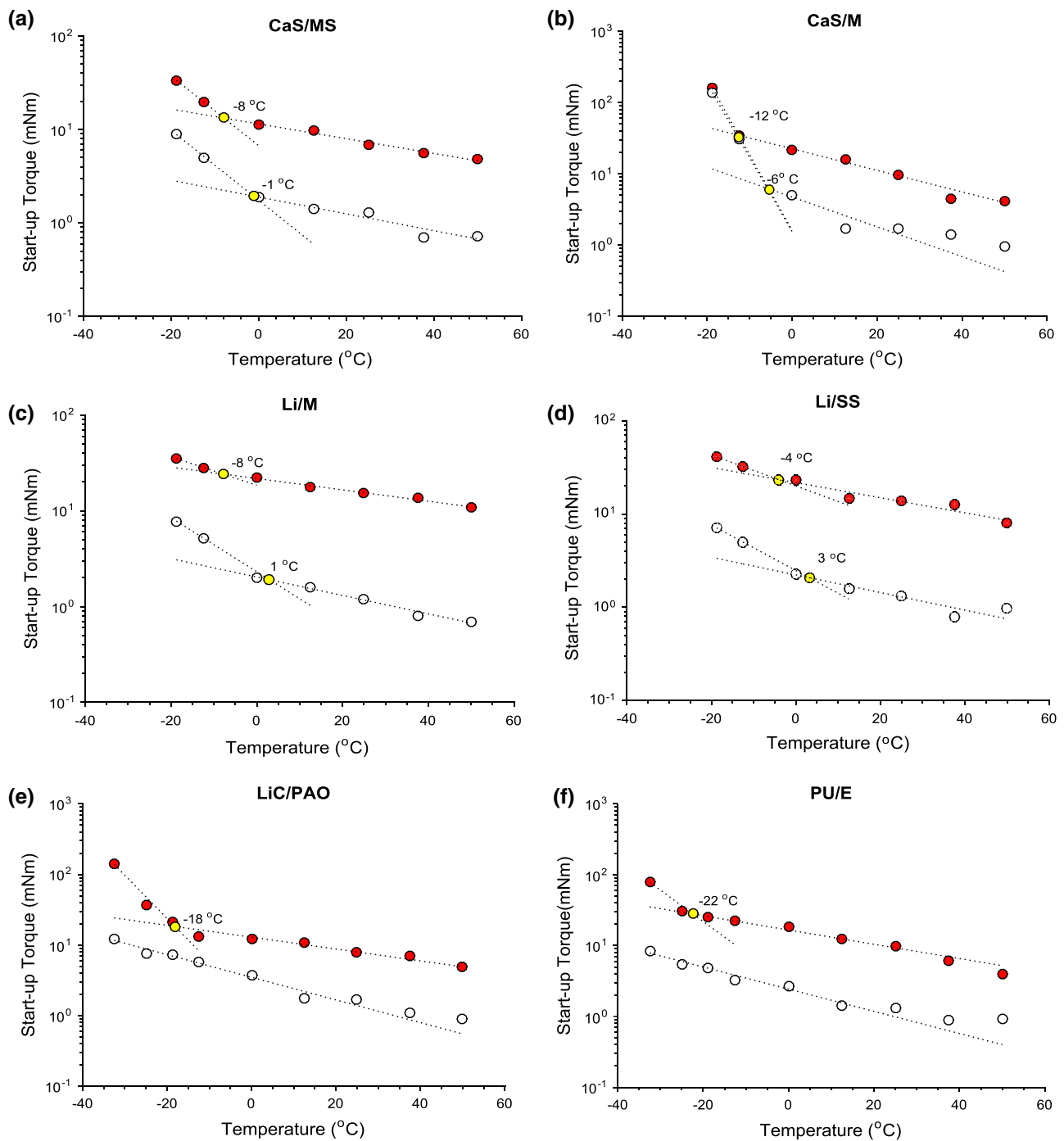


Fig. 5 Start-up torque versus temperature for lubricating greases (*filled symbols*) and corresponding bleed oils (*empty symbols*). The yellow dot denotes the break point (Color figure online)

3.3 Start-Up Torque Measurements

Figure 4 shows a typical plot for torque versus time for both grease and corresponding bleed oil. It illustrates the problem that occurs with a grease lubricated bearing at

very low temperature: The torque is much higher than that of an oil lubricated bearing. This is ascribed to the higher churning resistance caused by the grease. In the following section, only the start-up torque will be addressed.

Table 4 Parameter “*b*” obtained using Eq. (1) for start-up torque versus temperature for the greases and corresponding bled oils

Designation	Grease			Bled oil		
	T_b (°C)	b ($T < T_b$)	b ($T > T_b$)	T_b (°C)	b ($T < T_b$)	b ($T > T_b$)
CaS/MS	−8	8.14	38.1	−1	8.14	33.5
CaS/M	−12	7.11	19.1	−6	6.73	32.3
Li/M	−8	20.2	50.8	1	10.8	31.8
Li/SS	−4	18.1	37.2	3	12.3	31.8
LiC/PAO	−18	4.93	35.8	−	−	18.7
PU/E	−22	6.21	29.1	−	−	19.2

The temperature corresponding to the “break” in the Arrhenius plot, “ T_b ” is also shown

3.4 Temperature Dependency of the Start-Up Torque

The start-up torque for all greases was found to be an order higher in magnitude than for the corresponding bled oils (Fig. 5). However, an exception can be seen for CaS/M, for very low temperature. Similar to the yield stress, the start-up torque was also found to exhibit Arrhenius temperature behavior with a “break point.” The variation in start-up torque versus temperature can again be fitted using Eq. (1), and the parameter “*b*” obtained is shown in Table 4. The transition to high torque behavior (the break) is observed at a much lower temperature for PAO and ester-based greases in comparison with mineral oil-based greases. The “break” for PAO and ester oils probably occurs at temperatures lower than those that could be measured on the present system.

The variation in start-up torque with temperature was found to be different for greases and their bled oils, shown by different values of “*b*” (Table 4). The greases exhibited a lower variation in start-up torque with temperature in comparison with their bled oils. A larger variation in bearing friction torque to changes in temperature is exhibited by PAO and ester oil, given by lower values of “*b*” in comparison with the mineral oils studied (Table 4).

3.5 Start-Up Torque Versus Yield Stress

The start-up torque versus yield stress plots for the studied greases are shown in Fig. 6. The figure shows that there is a linear relation between start-up torque and yield stress.

Similar to yield stress—temperature and start-up torque—temperature, also the start-up torque—yield stress relation shows a break. This break was found to occur at temperatures close to the pourpoint of the bled oil, with the exception of polyurea grease (Fig. 6).

4 Discussion

There is a linear relation between start-up torque and yield stress. However, this relation is not unique. It is different for different greases. This means that the temperature dependence of yield stress and start-up torque is similar, which is reflected in quite similar values for “*b*” (see Tables 3, 4). The relationship changes at a characteristic temperature giving two temperature domains with different temperature dependence. This characteristic temperature, the “break point” in the Arrhenius plots (T_b), is correlated to the pourpoint of the bled oil (Fig. 7a; Table 5). So the change in temperature behavior is mainly ascribed to the temperature where the base oil loses its flow characteristics. The bled oil pourpoint and the “break” in the start-up torque with grease (Fig. 7b) and bled oil are less good correlated, which may be attributed to the fact that the start-up torque is not only governed by the yield stress.

The torque corresponding to the “break” (T_b) was not found to be comparable to the LTL, as for none of the lubricants, the start-up torque was found to exceed the often used limit of 1000 mNm. The LTL according to this definition is much lower than the break point, and the yield

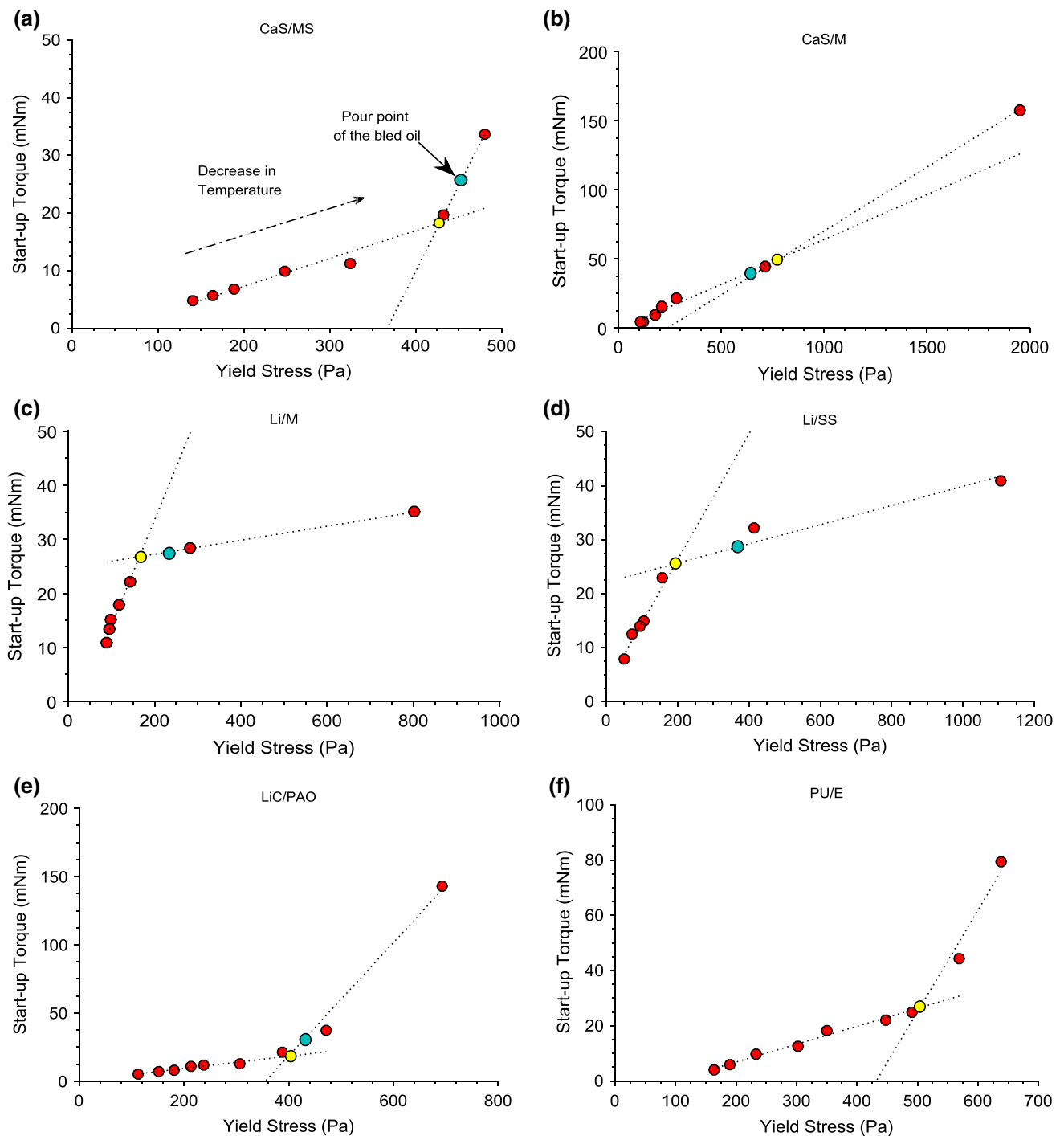


Fig. 6 Start-up torque versus yield stress from oscillatory strain sweep for the studied greases. The *yellow dot* denotes the break in the yield stress–start-up torque plot. The approximate position of the pourpoint of the bled oil is also shown (Color figure online)

stress will be extremely high at this point. The break point is a valuable grease parameter though. Greases with low values for the break point are more suitable for operation at very low temperatures.

The much lower T_b for greases based on PAO and ester oils in comparison with mineral oil-based greases confirms that PAO and ester-based greases can be used at lower temperatures than mineral oil-based greases.

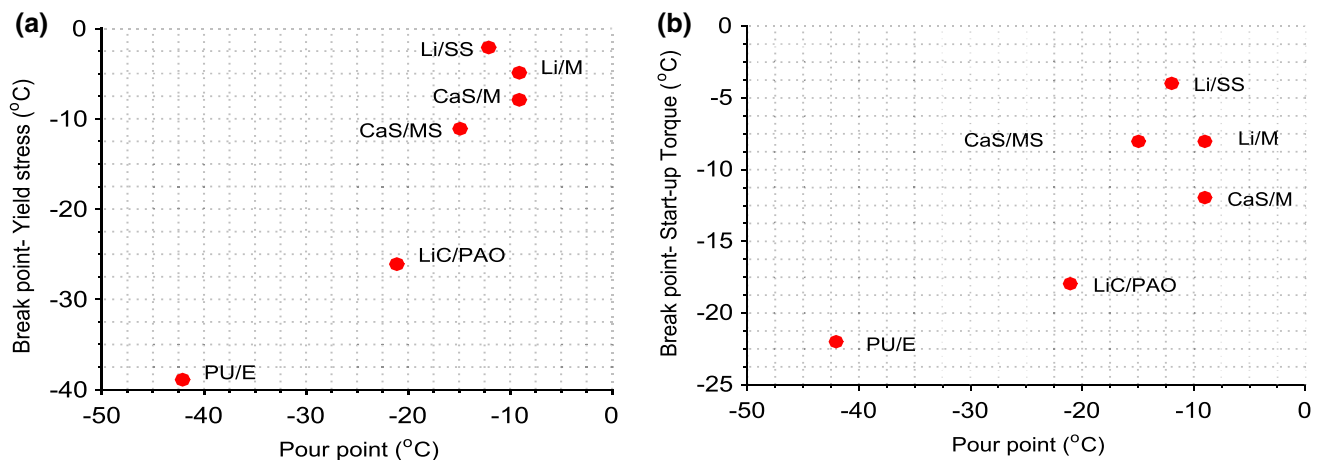


Fig. 7 Pourpoint versus **a** yield stress break point and **b** start-up torque break point

Table 5 Temperature corresponding to the “break” for yield stress and start-up torque and pourpoint of bled oils

Designation	Grease		Bled oil	
	Yield stress (°C)	Start-up torque (°C)	Start-up torque (°C)	Pourpoint (°C)
CaS/MS	-11	-8	-1	-15
CaS/M	-8	-12	-6	-9
Li/M	-5	-8	1	-9
Li/SS	-2	-4	3	-12
LiC/PAO	-26	-18	<-32	-21
PU/E	-39	-22	<-32	-42

5 Conclusions

The yield stress and start-up torque characteristics of six greases formulated with the most common thickeners and base oils have been studied in this paper. The start-up torque of the greases was found to be an order higher in magnitude than that for their corresponding bled oils. For rolling bearings operating at medium temperatures and medium speeds, the running torque is mainly given by the base oil viscosity of the grease. This clearly does not apply to the start-up torque, which is generated by the resistance of the balls to travel through the grease rather than the shear in the EHL contact. A deviation from Arrhenius behavior was observed for yield stress and start-up torque at lower temperatures, characterized by a “break.” The “break” was found to occur at a temperature close to the pourpoint of the bled oils. There is a linear relation between the start-up torque and the yield stress of the grease which indicates that the temperature dependence of the yield stress and start-up torque is similar. This linear relation changes at a temperature close to the pourpoint of the bled oil. At even lower temperatures, there is again a linear relation between start-up

torque and yield stress, but now with a different slope. The change in slope is ascribed mainly to the phase change in the base oil (the oil loses its flow properties) occurring at this temperature.

There are indications that the LTL, i.e., the lowest temperature at which a bearing can be started up without causing damage to a bearing, is much lower than the break point. This break point or pourpoint of the base oil can therefore not be used as a measure of the LTL. This does not mean that the break point or pourpoint of the base oil is not relevant. Greases with a low break point will also have a low LTL.

The fact that the break point is related to the pourpoint of the base oil is illustrated by the very low pourpoints of PAO and ester oils which can be used at lower temperatures than those greases based on mineral oils.

Acknowledgments This research was carried out under Project Number M21.1.12457 in the framework of the Research Program of the Materials innovation institute M2i (www.m2i.nl). The authors would like to thank SKF Engineering & Research Centre, Nieuwegein, The Netherlands, for providing the grease samples, technical and financial support and to A.J.C. de Vries, Director SKF Group Product Development for his permission to publish this paper.

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References

1. NLGI.: Lubricating grease guide. Kansas City (1996)
2. Lindenkamp, H., Kleinlein, E.: Grease Lubrication of Rolling Bearings at Low Temperatures. FAG Ball and Roller Bearing Engineering, pp. 40–43 (1985)
3. Lugt, P.M.: Grease Lubrication in Rolling Bearings. Wiley, Chichester (2013)
4. Wikström, V., Höglund, E.: Starting and steady-state friction torque of grease-lubricated rolling element bearings at low temperatures—Part I: a parameter study. Tribol. Trans. **39**(3), 517–526 (1996)
5. Thelen, E., Devine, M.J., Stalling, L.J.: Starting torques and rheology of lubricating greases at low temperature. Lubr. Eng. **27**(9), 305 (1971)
6. Kobzova, R.I., Klimov, K.I., Shul'zhenko, I.V.: Influence of temperature on static frictional torque of grease-lubricated bearings. Chem. Technol. Fuels Oils **10**(3), 227–229 (1974)
7. Cyriac, F., Lugt, P.M., Bosman, R.: On a new method to determine the yield stress in lubricating grease. Tribol. Trans. **58**(6), 1021–1030 (2015)
8. Kumamoto, J., Raison, J.K., Lyons, J.M.: Temperature “breaks” in Arrhenius plots: a thermodynamic consequence of a phase change. J. Theor. Biol. **31**(1), 47–51 (1971)
9. Cyriac, F., Lugt, P. M., Bosman, R., Padberg, C.J., Venner, C.H.: Effect of thickener particle geometry and concentration on grease EHL film thickness at medium speeds. Tribol. Lett. **61**(2), 1–13 (2016)