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Lubricating greases: Where rheology and tribology meet together

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Introduction

Grease lubrication is one of the most widely used types of industrial lubrication. Lubricating greases are also used in distributed or automatic lubricating systems in heavy-duty trucks, off-road vehicles, and other specialised machinery. A grease lubrication system usually includes a pump, flow divider valves and manifolds that provide precise amounts of grease to various lubrication points such as kingpins, bearings, joints, etc. Grease flow properties are therefore important for reliable operation of such systems.

However, what is grease and why is it used instead of oil? ASTM D288 defines grease as "a solid to semi-fluid product of dispersion of a thickening agent in liquid lubricant." Unlike lubricating oils, grease will not flow under the action of gravity and stays where it was applied. Lubricating greases have complex rheology and respond differently to changing the pump pressure as compared to lubricating oils. Rheology-wise, lubricating greases are regarded as viscoelastic materials. This means that under certain conditions they behave more as elastic solids and under other conditions they behave more as viscous fluids.

Even though the flow rate of grease increases as the pump pressure increases, excessively high pressure may cause oil bleeding and induce soap flocculation in high pressure zones, eventually leading to complete pipe blockage and lubricating system malfunction or damage.

Perhaps the easiest way to explain the concept of viscoelasticity is by using mechanical models that include an elastic spring and a dashpot with a viscous fluid. These two elements can be connected in a sequential (Maxwell) or parallel (Kelvin-Voigt) fashion, see Figure 1:

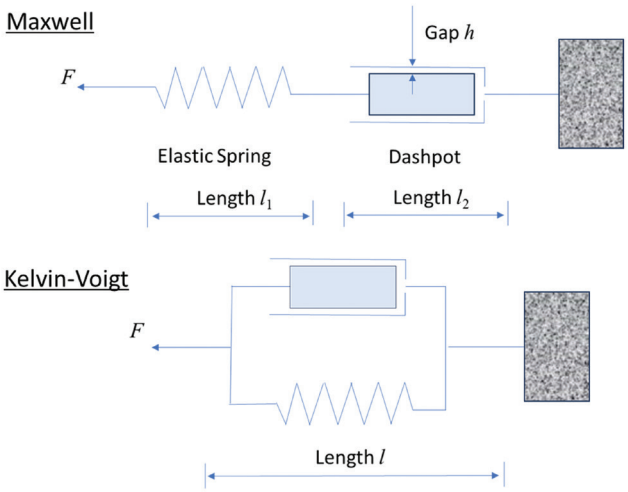


***Figure 1:** Mechanical models of viscoelasticity.*

The complete assembly is attached to a rigid wall on the right-hand side. Let us now analyse how each of these models responds to a sinusoidal force applied to the left side of the assembly,

$$F = F_0 \sin(\omega t)$$

The motion of piston in the dashpot is limited by viscous drag, which is independent of the piston displacement, Δl_2, but linearly increases with the rate of displacement, $\Delta l_2/\Delta t$. Hence, the displacement of piston will be very small if the oscillation frequency, ω, is sufficiently high. This means that, at high frequencies, the Maxwell model behaves like an elastic solid where the total deformation, $\Delta l = \Delta l_1 + \Delta l_2$, is determined by the deformation of the spring, the latter being inversely proportional to the spring stiffness. In the opposite limit of small ω, the total deformation is dominated by the piston displacement as in this case the piston can travel a long distance over the oscillation period. The distance travelled by the piston is inversely proportional to fluid viscosity. Hence, at low frequencies, the same model behaves like a viscous fluid.

The Kelvin-Voigt model behaves differently. The amplitude of deformation is always limited here - at high frequencies, by viscous forces, and at low frequencies, by elastic forces.

The Maxwell model is particularly useful to describe stress relaxation, and the Kelvin-Voigt model is useful to describe creep in viscoelastic materials.

In the literature, greases are often described as a "sponge impregnated by oil." This association is somewhat misleading since a sponge impregnated by oil would reveal the Kelvin-Voigt behaviour. Lubricating greases behave differently. In general, neither Maxwell nor Kelvin-Voigt model can adequately describe grease rheology. To understand why, let us look at how the grease microstructure changes under shear. Figure 2 shows the lithium soap thickener network imaged in situ using cryo TEM [1].

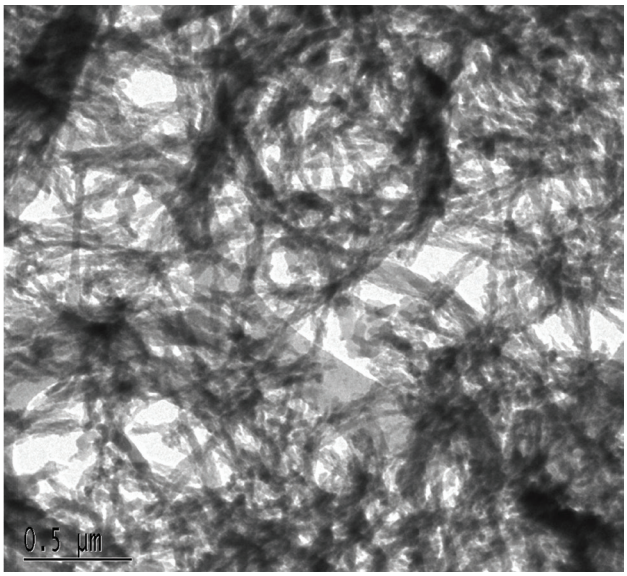


Figure 2: *Lithium soap fibre network in an NLGI 2 lubricating grease.*

This network is relatively fragile and starts to break down when grease is sheared (Figure 3).

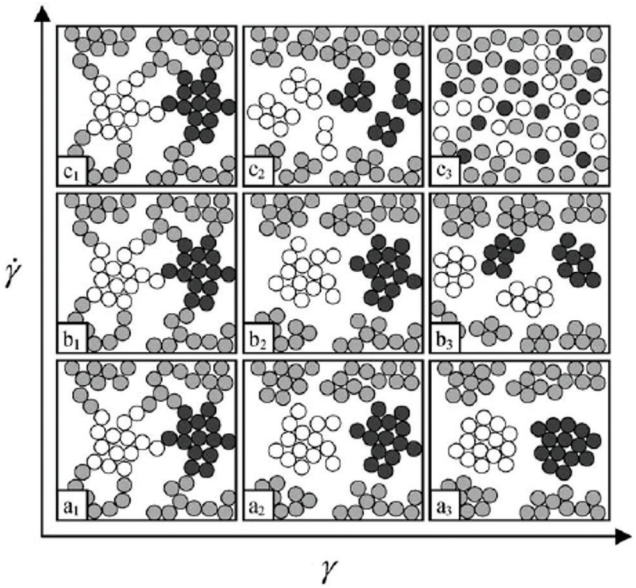


Figure 3: *Progressive disintegration of thickener network under shear.*

Loose aggregates break at low shear, whereas sturdier aggregates can withstand higher shear. In other

words, the grease microstructure is altered during the measurement. As a result, greases reveal complex non-linear viscoelastic behaviour.

To characterise the resistance of grease to deformation when it is subjected to force, the notion of grease consistency is used. Consistency is determined using the ASTM D217 cone penetration test. A standardised cone is allowed to sink into a sample of grease under its own weight for 5 min at 25°C. Nine National Lubricating Grease Institute (NLGI) grades are defined based on the penetration depth measured (Figure 4).

The NLGI grades

GRADE NUMBER	CONSISTENCY	PENETRATION (mm/10)
NLGI 000	Very fluid, almost liquid-like	445-475
NLGI 00	Soft, semi-fluid	400-430
NLGI 0	Soft, butter-like	355-385
NLGI 1	Semi-soft	310-340
NLGI 2	Normal consistency	265-295
NLGI 3	Firm	220-250
NLGI 4	Very firm	175-205
NLGI 5	Hard	130-160
NLGI 6	Very hard	85-115

Figure 4: *Definition of NLGI grease consistency grades based on cone penetration depth.*

Starting in the 1980s, considerable criticism was directed toward the cone penetration test, which was considered too empirical and insufficiently reproducible. Simultaneously, rheological methods began to make their way into industrial laboratories. In the 1990s, Lennart Hamnelid from Axel Christiernsson proposed to use the so-called grease rheology intersection stress (GRIS) as an alternative to cone penetration. In his 1998 article [2], Hamnelid states *"Stress rheometer provides data on the elasticity, shear stability, shear recovery and many more properties, and also, not least important - it can be used to derive a value of the consistency of the lubricating grease with a better accuracy than cone penetration."*

To determine GRIS, a controlled stress rheometer with a cone-plate geometry is used. The GRIS is defined as the shear stress where the storage modulus, G', and the loss modulus, G", are the same (Figure 5).

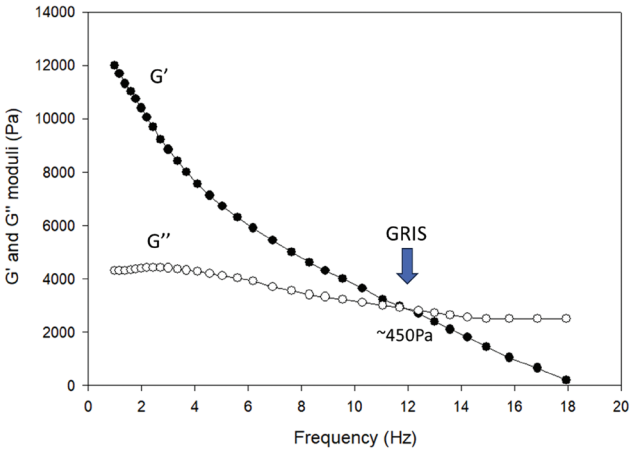
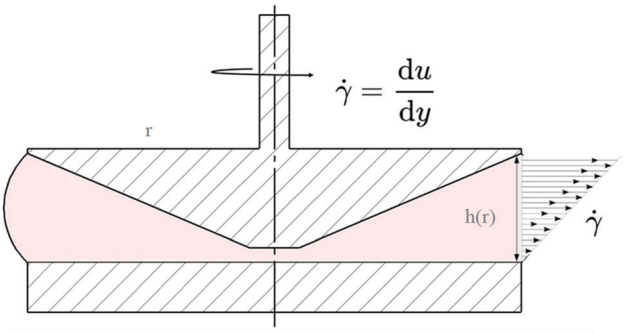


Figure 5: *Definition of GRIS using controlled stress rheometry for an NLGI 2 lithium grease.*

In general, the higher the NLGI consistency, the higher the GRIS. Unfortunately, this is where the simplicity ends.

To understand limitations of the above method, it is instructive to recall what a rheometer actually measures. The primary measurables include torque, deflection angle, angular velocity, and phase shift in the torque-deflection coupling. Everything else is calculated using certain model assumptions, which may or may not be valid. For example, when a cone-plate geometry is used, a constant shear rate throughout the sample volume is assumed (Figure 6).



Measured: Shear stress (torque), strain (deflection angle), shear rate (angular velocity) and stress-strain phase shift (δ)
Calculated: Apparent viscosity (η*), Storage modulus (G'). Loss modulus (G'')

Figure 6: *The principle of controlled stress rheometry: Primary measurables and derived properties.*

What can go wrong on the rheology track? Quite a few things, actually – definitely more than with cone penetration measurements. To fully illustrate the scale of the challenge, let us quote one of the pioneers in the field of rheology, Prof. Joachim Meissner from ETH Zurich, who in 1989 admitted that *"More than 40 years after Weissenberg's pioneering proposal for the measurement of shear and normal stresses during shear flow of elastic liquids, the determination of the rheological material functions is still a nightmare for the experimenter."* To be honest, his assessment is still valid today.

There are several sources of errors for a cone-plate rheometer that remain hidden from an unexperienced experimenter: gap opening, inertia effects, wall slip and secondary flow effects, torque correction due to edge effects and cone tip/plate gap, shear heating, etc. If you measure the kinematic viscosity of an oil, you would expect to obtain the same value regardless of which viscometer you use. To achieve a comparable level of reproducibility with different rheometers in multilab trials is practically impossible – at least without introducing very exacting standards for the hardware and measurement routines to be used, as different setups tend to probe different modes of rheological behaviour.

A brief review of published data conducted by one of the authors show a high incidence of measurement artefacts due to improper design of experiments and poor understanding of the test limitations. This seriously undermines credibility of the method. To give an illustrative example, Figure 7 shows the apparent viscosity data for an NLGI 2 lithium grease at three different temperatures. Taking a closer look, one can immediately notice that the data are controversial. It is well known that the apparent viscosity of greases always decreases with increasing temperature. How is it possible that the temperature-viscosity curves intersect and that low-shear-rate viscosity at 30°C is lower than at 70°C? The answer lies on the surface: what we see here is a rather common measurement artefact due to wall slip. At 30°C, grease is too stiff to be uniformly sheared and it slips against the wall as a solid block. Unfortunately, there is no way to compensate for such artefacts in rheological measurements, and hence the reported apparent viscosity values are meaningless – and so are all other derived characteristics, including moduli and GRIS.

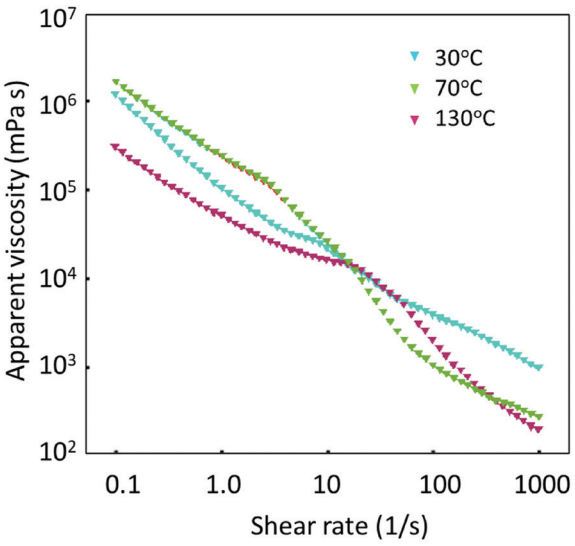


Figure 7: *Example of artefacts plaguing measurements of grease rheological functions.*

Finally, since both penetration and GRIS are just a 'one number says it all' characteristics of grease consistency, there is no obvious advantage in

replacing a simple-to-measure number by a more difficult-to-measure one. Neither of them can provide any additional information about grease flow properties at different temperatures, channel geometries, shear rates, and so on. Nor do they have to, as dedicated tests exist for those purposes. For example, to evaluate the ability of grease to flow through lines in a distributed lubrication system, one would use the ASTM D1092 test, which measures the pressure required to force the grease through an orifice. This is the primary test to characterise grease pumpability at different temperatures. To measure the maximum operating temperature at which a grease will "melt down," there exists the ASTM D2265 dropping point test. Theoretically, if the relevant test conditions are reproduced in a rheological experiment, one can gain a deeper insight into the underlying physics. Thus, rheological measurements show that as we approach the dropping point temperature, the storage modulus G' falls below a certain threshold, and the grease sample can no longer resist the gravity force and starts to drip. Hence, it is indeed possible to replace the dropping point test by a rheological test, but would it be useful?

At the same time, the ability to accurately determine rheological properties is invaluable for the optimisation of computer-aided engineering (CAE) models that are extensively used in engineering. As an illustration, Figure 8 shows flow imaging experiments used to visualise grease flow in bearings.

Let's finally talk about the relationship between grease rheology and its tribological properties. This relationship is very complex. The function of grease is not only to serve as a source of lubricating oil in the tribological contacts. All ingredients used by grease manufacturers in their cooking recipes may cross-interact and significantly affect grease tribology. Greases of the same NLGI class may exhibit huge differences in various aspects of tribological performance.

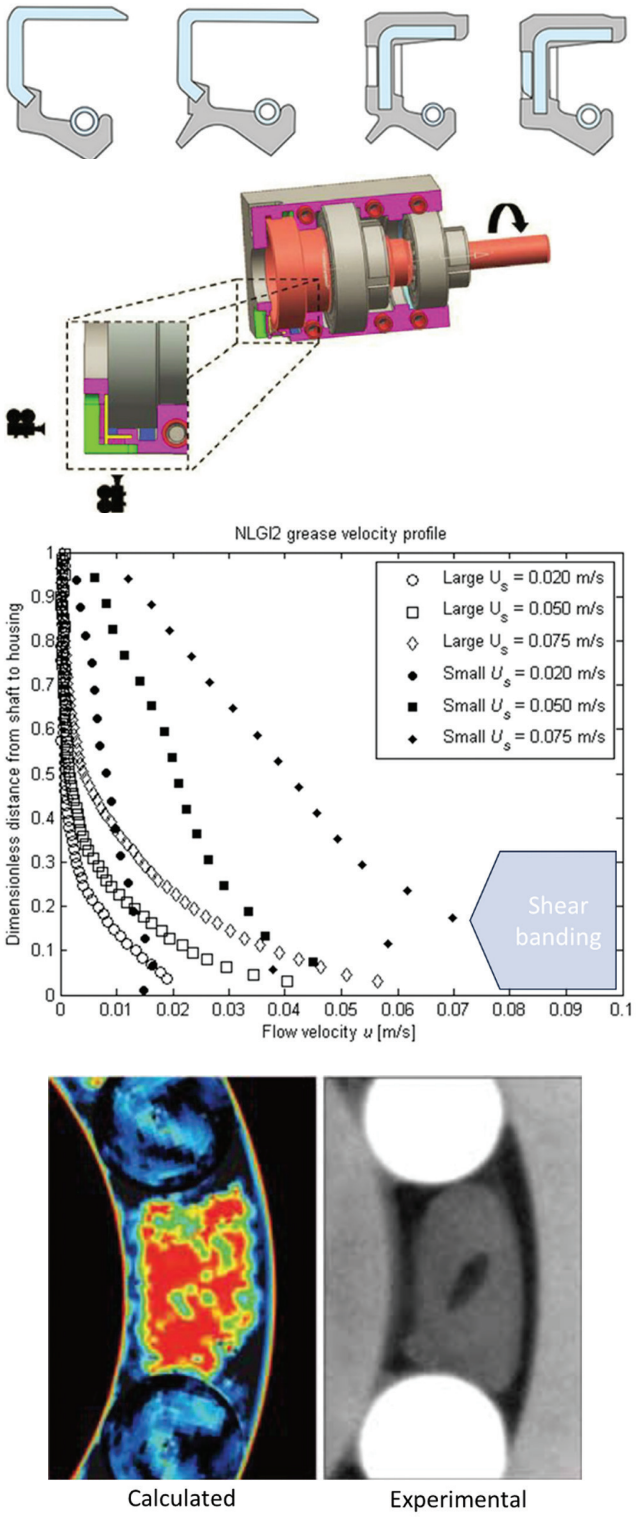


Figure 8: *Application of microparticle imaging velocimetry [3], meshless CFD simulations and in situ X-ray CT imaging [4] to visualise grease flow in bearings.*

Base oil is one major differentiator. The viscosity of the base oil used in grease will define the lubricant film thickness in a loaded contact. Besides that, different base oils have different temperature-viscosity and pressure-viscosity curves. For example, naphthenic base oils are a preferred choice when good low-temperature pumpability is important (see Figure 9), while paraffinic oils provide superior high temperature performance. Synthetic base oils such as polyalphaolefins (PAO) can combine the best of both worlds but come at a higher price.

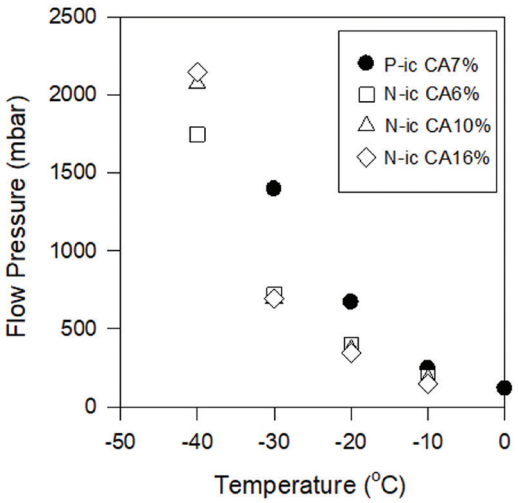


Figure 9: *Effect of base oil type on low temperature pumpability of grease. One paraffinic (P-ic) and three naphthenic (N-ic) oils of ISO VG 150 viscosity grade are compared.*

The solvency, or solvent power, of a base oil is also important for oil/thickener cohesion. For example, naphthenic oils have higher solvency than paraffinic oils of a similar viscosity grade. As a result, the former provide superior wetting of thickener fibres, contributing to better grease stability.

The tribological effects of EP/AW additives probably do not need to be explained. At the same time, it is worth noting that thickeners also have a significant impact on tribology. All soap thickeners are surface active compounds that form boundary layers at metal surfaces, and hence influence the boundary friction. For example, calcium sulfonate provides better lubricity than lithium 12-hydroxystearate. In addition, soap thickeners also act as corrosion inhibitors and dispersants. On the other hand, some inorganic thickeners, such as silica and clay, may contribute to increased wear.

One still largely unexplored area is the effect of grease tackiness on efficiency. Since tackifiers have a significant effect on the extensional rheology of greases, the use of overly tacky greases may lead to increased friction losses in high-speed bearings.

Conclusions:

Rheological measurements, in situ flow imaging, and meshless CFD simulation methods can provide a wealth of information about the flow properties of lubricating greases at different length and time scales. These studies show that there is an intimate interplay between rheological and tribological properties. This information is useful for the optimisation of CAE tools used in modern engineering. At the same time, with regard to grease manufacture, it does not appear practical to use rheological tests as a replacement for the cone penetration test solely for QA purposes. Rephrasing Douglas Adams, *"All you really need to know for the moment is that grease rheology is far more complicated than you might think, even if you start from the position that it is already quite complicated."*

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