APPLICATION**NOTE**

Rheological Properties of a Lubricating Grease in Accordance with DIN 51810-2

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Introduction

The rheological properties of materials help understand and anticipate their behavior during their processing. For example, they play a role in the lubrication ability, pumpability, and flow (yield/drop) point of lubricating greases.

The shear viscosity measurement of a lubricating grease according to DIN 51810-1 standard is described in our AN 222 [1]. In the following, the yield and the flow points of this material are determined with the Kinexus pro+ using the measurement conditions stipulated in DIN51810-2.

Measurement Conditions

Table 1 summarizes the test parameters specified in this standard [2]. Two different methods are described: The amplitude sweep can be strain or stress controlled, corresponding to methods A and B, respectively. In this work, both methods are employed.

Table 1. Measurement conditions

Measurement type	Oscillation
Geometry	PP25 (Parallel plate system, diameter: 25 mm)
Temperature	25°C (±0.1°C)
Trimming gap	1.025 mm
Measurement gap	1 mm
Frequency	1.59 Hz (corresponds to an angular frequency ω = 10 rad/s)
Method A: Amplitude strain sweep	0.01 to 100%
Method B: Amplitude stress sweep	0 to 1,000 Pa

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Measurement Results

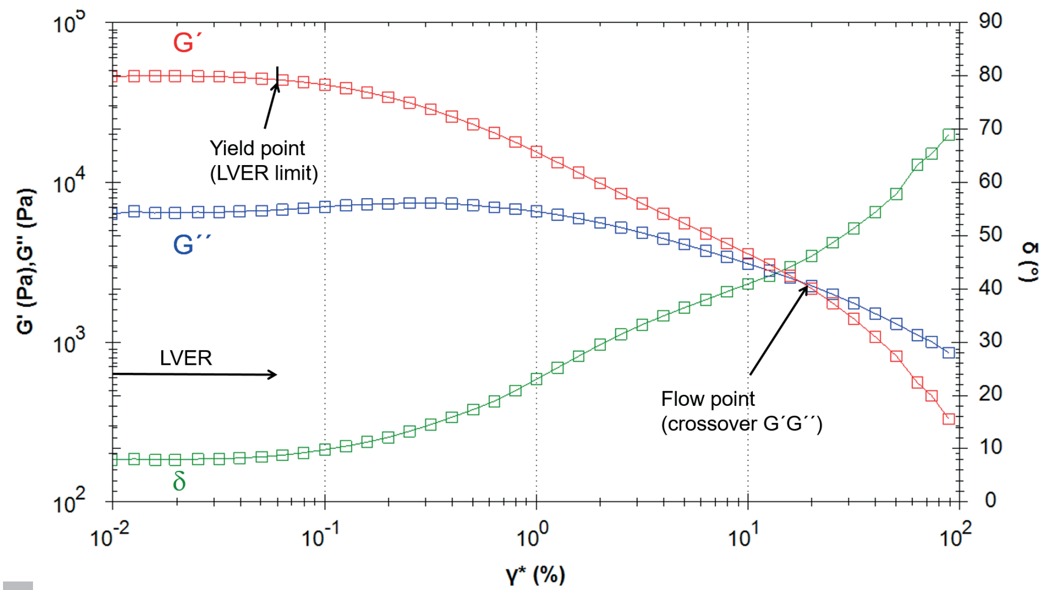
Figure 1 depicts the elastic and viscous shear modulus G´ and G" curves along with the phase angle curve during the amplitude strain sweep. At low deformations, the grease is in the linear viscoelastic range (LVER), as taken from the plateau of the shear modulus curves. Here, the values for G´ and G" are constant, as the shear stress and shear strain are proportional; the applied strains do not lead to a breakdown of the sample´s structure. In this range, the elastic component is higher than the viscous, so that the solid-like properties dominate over the liquid-like properties of the grease for the selected measurement conditions. This behavior can also be taken from the phase angle curve being lower than 45° (see description of the phase angle in the green box).

From a deformation of 0.1%, the elastic modulus curve (red) begins to decrease. This effect is related to the start of the breakdown of the sample's associated (internal) structure and indicates the end of the LVER (Linear Viscoelastic Region). The limit of this range is defined as yield point or also linearity limit at which the shear strain (γ_Y) and the shear stress (σ_Y) can be determined (see table 2).

Phase Angle
The phase angle is a realitve measure of the viscous and elastic properties of a material. It ranges from 0° for a fully elastic material to 90° for a fully viscous material.

A further increase in strain leads to a crossover of G´ and G". This point can be defined as the flow point of the grease. The related shear strain and shear stress are called γ_F and σ_F, respectively. If strains higher than the flow point are applied to the material, then it will begin to flow for the selected measurement conditions, i.e., for the used frequency.

The yield-flow index is defined as σ_F/σ_Y. This value gives information about the brittleness of the grease. In this case, it is much higher than 1, showing that the grease features an enduring behavior. Table 2 summarizes all values determined with the measurement on grease.



1 Amplitude strain sweep on lubricating grease

Table 2. Evaluation of the measurement

Flow point = cross-over point of the curve of G' and G"	Shear stress value	σ_F	597 Pa
	Shear strain value	γ_F	17.8%
Yield point = limit of the LVER range	Shear stress value	σ_Y	27.3 Pa
	Shear strain value	γ_Y	0.06%
Yield-flow transition index	σ_F/σ_Y		22
LVER	Elastic shear modulus	G'	$4.37 \cdot 10^4$ Pa
	Viscous shear modulus	G"	$6.73 \cdot 10^3$ Pa
	Phase angle	δ	8.76

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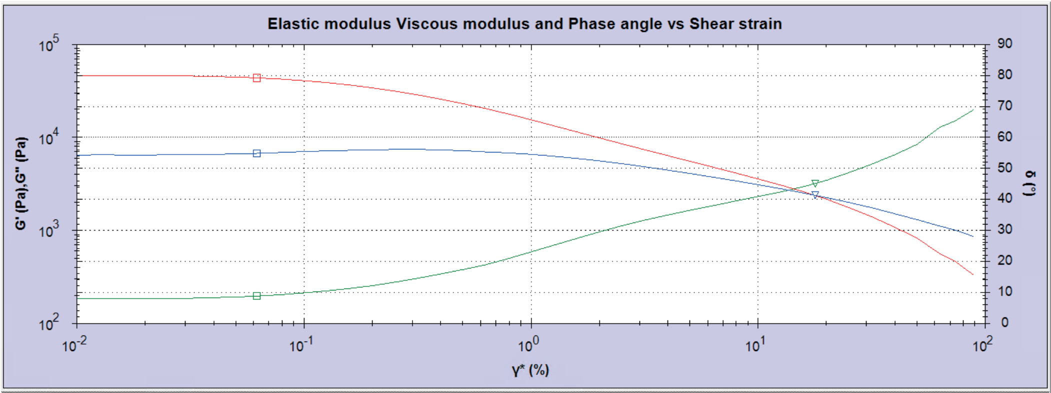
As displayed in figure 2, the rSpace software is capable of providing automatic evaluation of the required values as soon as the measurement is finished.

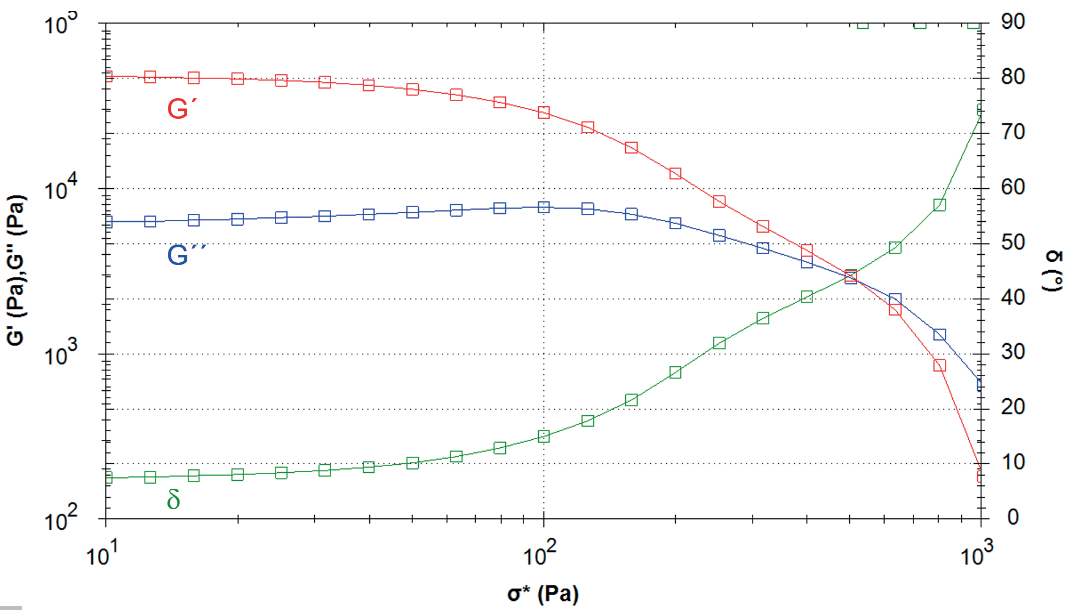
Figure 3 displays the curves resulting from the amplitude stress sweep measurement (method B described in DIN 51810-2).

The strain inducing by the applied shear stress can also be displayed in the x-axis for better comparison of the curves (figure 4). It shows the good repeatability of the measurements.



Action Name	Find value - Find stress and strain where G' drops by %5	Cross over
Experiment Name	LVER determination for DIN51810-2	Analyse_0001 Determination of the cross over point between G' and G''
Sample Description	071-3-20-04 Grease Din51810 Part2	071-3-20-04 Grease Din51810 Part2
Phase angle (°)	8.76	45.08
Point Index	1	1
Shear modulus (elastic component) (Pa)	4.368E+004	2.387E+003
Shear modulus (viscous component) (Pa)	6.729E+003	2.387E+003
Complex shear strain (%)	0.0618747	17.8519
Complex shear stress (Pa)	27.34	597.8

2 Amplitude strain sweep on lubricating grease with automatic evaluation of the LVER limit for the yield point determination and of the crossover for the flow point determination



3 Amplitude stress sweep on lubricating grease

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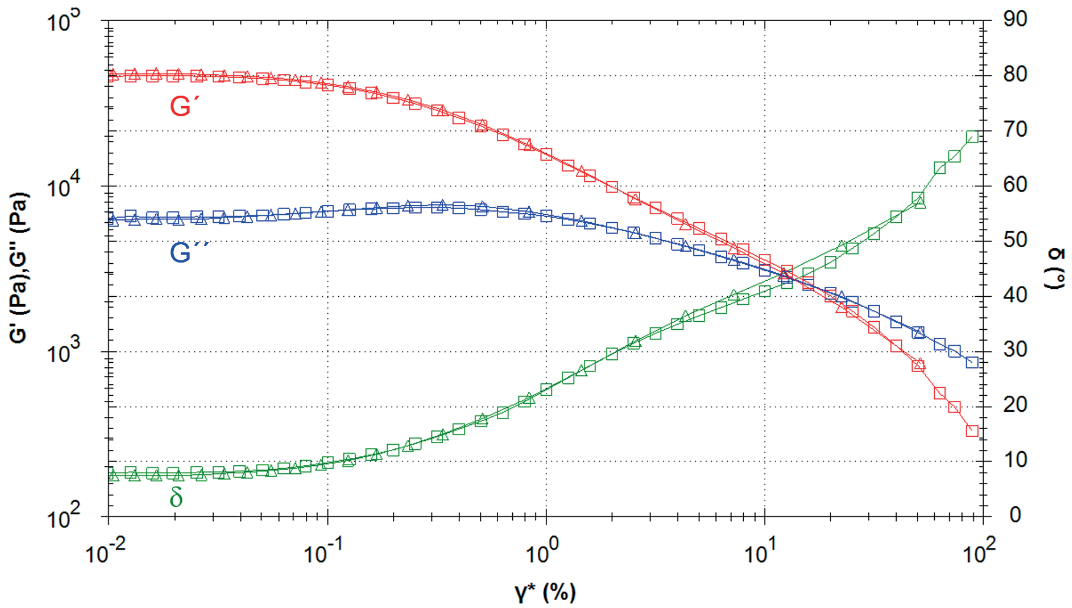

4 Comparison of the amplitude stress sweep, stress controlled (triangles) and strain controlled (squares)

Conclusion

Tests according to the second part of DIN51810 were performed on a lubricating grease. The subsequent evaluation for the determination of the yield and flow point was automatically carried out by the rSpace software.

Literature

[1] NETZSCH Application Note AN 222 – Rheological Properties of Lubricating Grease in Accordance with DIN 51810-1

[2] Testing of lubricants – Testing rheological properties of lubricating greases – Part 2: Determination of flow point using an oscillatory rheometer with a parallel-plate measuring system

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