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Direct Strain Oscillation: A new oscillation method enabling measurements at very small deflection angles and torques

Jörg Läuger, Klaus Wollny, Siegfried Huck

Physica Messtechnik GmbH, Vor dem Lauch 6, D-70567 Stuttgart, Germany
phone: +49-711-72091-0, fax: +49-711-72091-30, e-mail: info@physica.anton-paar.de
http://www.physica.de

ABSTRACT

A new method has been developed to improve strain controlled oscillatory measurements by adjusting the strain directly on the sine wave with the use of a real-time position control. The actual movement of the measuring system follows directly the required strain during each individual oscillation cycle.

INTRODUCTION

It was shown previously that a rotational rheometer equipped with an electronically commutated synchronous motor (EC-motor) allows to conduct real stress and strain experiments with the same rheometer[1]. Generally, a strain controlled oscillation test in a stress controlled rheometer consists of the following steps: applying one full oscillation cycle with an arbitrary stress amplitude, measuring the strain amplitude, adjusting the stress in the next oscillation cycle, and repeating this routine until the desired strain amplitude is reached. The newly developed Direct Strain Oscillation (DSO) method uses a different approach. It does not require a full oscillation cycle but uses a real-time position control and adjusts to the desired strain directly on the sine wave. Therefore, the actual movement of the measuring system follows directly the required change in strain during each individual oscillation cycle.

This new oscillation mode has several major advantages including:

1. the possibility of conducting real strain controlled tests in oscillation,
2. the exact strain setting right from the first oscillation cycle, i.e. no or only very slight overshoot in strain,
3. faster data acquisition even within an oscillation cycle,
4. measurements at extremely low angular resolution and low torques,
5. the absence of a rotational drift-

Due to the absence of strain overshoots and the ability of testing at low strain and low stress levels, this new method is especially valuable for measurements on samples with low viscosity and weak structure such as gels, emulsions, suspensions, colloids, surfactant solutions, lubricating grease, and foams.

METHOD

The principle of an EC-motor has been described in detail previously[1]. Here only the parts relevant to this study are shortly recalled. This motor uses a electronic commutation where the motor is excited by special permanent magnets with a high flux density. Therefore, the rotor field is known and the EC control makes use of this knowledge. It is possible to adjust the electro-magnetic torque in such a way that it is linear to the total amount of the stator current, i.e. $M \sim I_s$. Under these conditions

a change in the stator current will be followed by a change in the torque almost instantaneously.

Traditional Strain Oscillation with Stress Rheometers:

The frequency is fixed and a certain strain amplitude γ_{0d} is desired. In this case the instrument is applying a full stress oscillation cycle $\tau(t) = \tau_0 \cdot \sin(\omega \cdot t)$ and the resulting deflection is measured. Since in a real experiment only a finite amount of discrete deflection angles can be measured by the rheometer electronics, a fast Fourier transformation (FFT) is conducted after each oscillation cycle. Normally only the resulting first harmonic is used giving a strain oscillation: $\gamma(t) = \gamma_0 \cdot \sin(\omega \cdot t + \delta)$, where ω is the preset frequency and δ the sample dependent phase shift. The resulting strain amplitude γ_0 is compared with the desired strain amplitude γ_{0d}. In the next oscillation cycle the stress amplitude τ_0 is adjusted to reach a γ_0 which is closer to the desired strain amplitude γ_{0d}. This process is repeated until γ_0 and γ_{0d} are equal within a few percent. Depending on the sample and the torque which is needed, the adjustment process can take many oscillation cycles. There is the additional risk that during the adjustment the strain can be much higher than γ_{0d}, i.e. a strain overshoot occurs, the actual strain is beyond the linear visco-elastic range of the sample, and a sensitive sample structure might be destroyed.

Direct Strain Oscillation (DSO):

The desired strain is $\gamma_d(t) = \gamma_{d0} \cdot \sin(\omega \cdot t)$. In the direct strain oscillation mode a stress $\tau(t)$ is applied at the time t and the resulting strain $\gamma(t)$ is measured. Then the stress is adjusted to minimize $|\gamma_d(t + \Delta t) - \gamma(t + \Delta t)|$, i.e. that the actual strain follows the desired sinusoidal strain wave. Depending on the frequency used and the sample properties the length of the adjustment period differs, but normally the adjustment is finished within half of the first oscillation cycle. The applied strain now follows directly the desired strain wave. The necessary stress values are used to calculate the stress amplitude τ_0 and the phase shift δ. This can be done not only after a full cycle, but also within an oscillation cycle since there is no need for a Fourier transformation as in the traditional strain oscillation for stress rheometer. In other words, the adjustment routine of the direct strain oscillation mode gives directly an online determination of the stress amplitude and the phase shift.

INSTRUMENT

The measurements were conducted using the Direct Strain Oscillation (DSO) option on a Physica MCR 300 rheometer equipped with an EC-motor.

RESULTS

1. Amplitude Sweeps

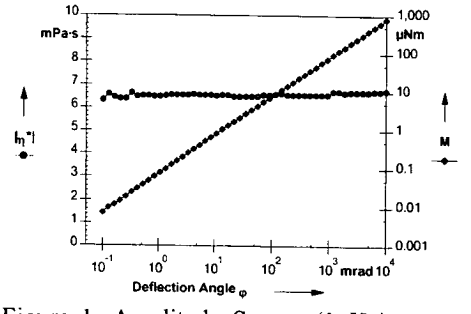

Figure 1. Amplitude Sweep (1 Hz) on a low viscous silicone oil at 20°C. Cone-plate system 50 mm 1°.

Fig. 1 shows a strain sweep on a low viscous (6.5 mPas at 20°C) silicone oil. As can be seen the viscosity can be determined over 5 decades of the deflection angle, i.e. strain, and down to torque values of 0.01 µNm. For the cone-plate geometry (50mm, 1°) used in this test the limits of the strain and stress are 0.5 % and 0.00025 Pa, respectively. The excellent agreement between the Direct Strain

Oscillation data and the shear viscosity from a rotational test can be seen in Fig. 2.

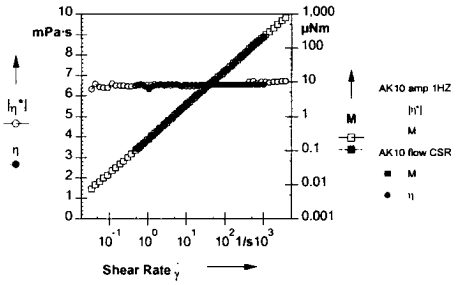

Figure 2. Comparison of DSO with flow curve. Shear rate in Oscillation: $\dot{\gamma} = \omega \cdot \gamma$.

Results of an oscillatory amplitude sweep at 1 Hz on a Polyisobutylene polymer solution are displayed in Fig. 3. The data show that it is possible to set angular displacement amplitudes as small as 0.1 micro radians and measure torques as low as 0.02 μNm. For the used cone-plate geometry with 50 mm diameter and a 1° cone this means strain and stress amplitudes as small as 0.0006 % and 0.001 Pa, respectively.

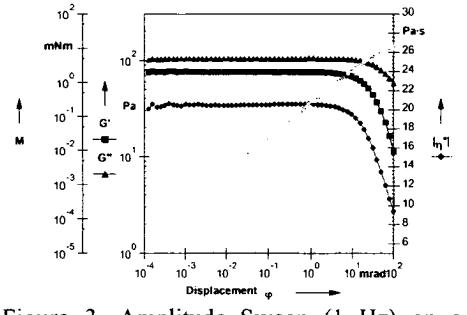

Figure 3. Amplitude Sweep (1 Hz) on a Polyisobutylene solution (NIST SRM1490) at 20°C. Cone-plate system 50 mm 1°.

Fig. 4 shows a similar experiment on a food sample. Again amplitudes starting from 0.1 microradians were set and torques smaller than 0.02 μNm were detected, resulting in strain and stress of 0.0004 % and 0.00023 Pa, respectively. The measuring range covers almost 8 decades in strain amplitude.

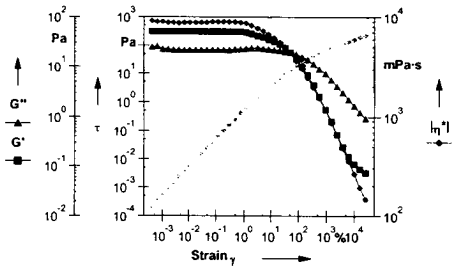

Figure 4. Amplitude Sweep (1 Hz) on a starch based desert. Plate-plate geometry (50 mm).

2. Fast oscillation measurements

a) Curing reaction:

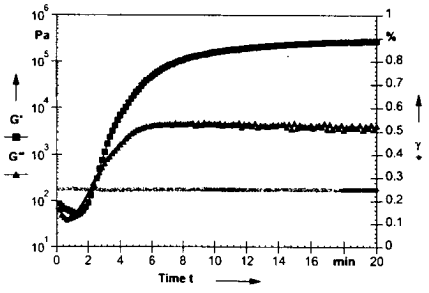

Figure 5. Curing reaction of a powder coating. Strain: 0.25%, angular frequency: 10 1/s, plate-plate geometry (25 mm).

Fig. 5 shows the measurement of a curing reaction of a powder coating at 180°C. At the start of this experiment the sample viscosity is very low and drops further before the viscosity increases due to the onset of the curing reaction. The resulting torque at the viscosity minimum is well below 0.1 μNm, making it difficult to set the desired strain and measure the rheological properties with a conventional oscillation method. As can be seen in Fig. 5 the DSO methods allows to keep the strain exactly at the desired value.

b) Fast data at low frequencies:

The ability of the Direct Strain Oscillation mode to produce valid data points even during a single oscillation

cycle is demonstrated in Fig. 6. An oscillation measurement with a constant frequency of 0.01 Hz and a strain of 10% was carried out and data points were taken every 20s, i.e. 5 data point per oscillation cycle. As can be seen the strain is reached already at the second data point, i.e. even before half of the first oscillation cycle has passed. Moreover, the resulting G' and G'' values have the final values already before first oscillation period is finished.

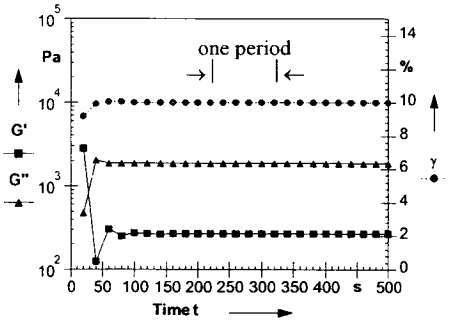

Figure 6. Constant oscillation measurement on PDMS, γ = 10%, f = 0.01 Hz, 20 s measurement point duration, Plate-plate geometry (25 mm).

3. Structure recovery on low viscous samples with weak structures

Another interesting application of the Direct Strain Oscillation mode is the measurement of structure recovery, i.e. thixotropy, of weak structures. It is well known that the amount and time dependence of structure recovery after a high shear load is crucial for a large variety of practical application. Fig. 7 shows an example of two chocolate milks. A three interval thixotropy test was performed. The first interval was an oscillation at rest. In the second interval a constant shear rate of 1000 1/s was applied for 50s. In the third interval the structure recovery was followed by a constant oscillation (ω = 3 1/s; γ = 1 %). As can be seen the two samples exhibit a large difference. The first sample shows a fast recovery with a crossover of G' and G'' after about 1.5 min, whereas in the second sample G'' stays below G' over the whole measuring time indicating that this chocolate milk will show sedimentation after shaking the bottle.

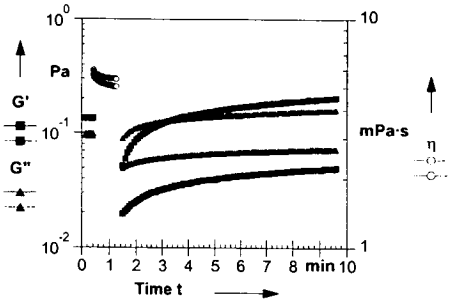

Figure 7. Structure recovery (thixotropy) of two chocolate milks. ω = 3 1/s, γ = 1%, double gap geometry (42 mm).

CONCLUSIONS

It has been demonstrated that by employing the Direct Strain Oscillation (DSO) mode an EC-motor equipped rheometer allows to perform real strain controlled tests in oscillation. This new oscillation method extends the range of the rheometer to new test modes and applications. DSO is especially valuable for measurements at low torques (<0.1μNm) and small deflection angles (0.1μrad) thus enabling the investigation of very weak structures. The absence of any rotational drift allows structure recovery, measurements of low viscous samples with weak structures without destroying the structure during its rebuild.

REFERENCES

1 J. Läuger, S. Huck, Proceedings of the XIIIth International Congress on Rheology, Cambridge, UK, 2000, p. 3-10.