

Measuring structure of low viscosity fluids in oscillation using rheometers with and without a separate torque transducer

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ABSTRACT

Rheometers with a separate detached torque transducer are not relying on the torque output of the motor to determine the stress. Instead the transducer measures the reacting sample torque - thus eliminating the inertia of the high mass motor - and provides accurate dynamic moduli on water-like materials up to and above frequencies of 100 rad/s.

INTRODUCTION

Measuring structure in low viscosity fluids like fruit juices, dilute water-based systems, low viscosity inks etc. can be challenging. The reason for this is the inertia of the instrument, masking the elastic contributions of the weak structure in these materials. If concentric cylinders or parallel plates with a gap of 1mm and larger are used while performing oscillatory measurements, effects of the inertia of the fluid have to be considered also.

During transient experiments, the inertia has a retarding effect on the materials response - or - for a Newtonian fluid the system response is "viscoelastic" with a characteristic time constant¹. In oscillation the inertia introduces an additional phase shift, which increases the elastic modulus G' .

The best metric for evaluating, to which extent the inertia of the motor in CMT (combined motor transducer) instruments influences the time or frequency dependant response of a material is the ratio of torque

used to overcome inertia and the total torque of the motor applying the deformation.

For SMT (separate motor transducer) rheometers with a separate transducer, the motor inertia has no influence on the torque measurement. The separate transducer due to the low mass and the fact, that it is hardly moving, has inertia contributions which are orders of magnitude lower than those of the motor.

In addition to the inertia effect of the measuring device, the inertia of the fluid becomes significant for low viscosity fluids in oscillation. If the wavelength of the shear wave has the same magnitude or is smaller than the sample thickness (gap in concentric cylinders or parallel plates) the fluids inertia must be taken into account.

TWO TYPES OF RHEOMETER DESIGNS

Two types of rheometers are currently in use (Figure 1): - the combined motor transducer (CMT) rheometer, which measures the energy input to the motor and - the separate motor transducer (SMT) rheometer, with an actuator to deform the sample and a torque transducer to measure the torque. The difference between the two designs is the detached torque transducer. In the SMT rheometer the sample torque is not determined from the motor torque, like in the CMT rheometer², but measured separately. Therefore no corrections for motor inertia or bearing friction need to be applied. The limitations for SMT rheometer are the inertia

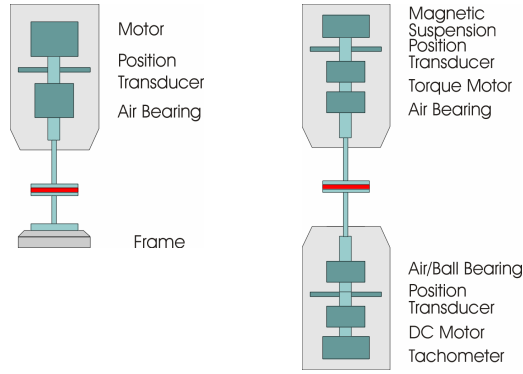


Figure 1. SMT and CMT rheometer designs

contributions of the transducer or the sample itself.

INERTIA EFFECT ON TORQUE AND PHASE IN OSCILLATION

In order to evaluate the effect of inertia on the measured torque and phase in oscillatory measurements, the ratio of inertia torque and sample torque is analysed as a function of the material and test parameter for the CMT and SMT rheometers.

Torque balance and torque ratio for CMT rheometers.

The motor torque T_m in a CMT rheometer is the sum of all torque contributions to overcome inertia, friction and the torque needed to deform the sample (Eq. 1).

$$T_m = T_I + T_S \quad (1)$$

$$T_I = I\omega^2\theta(i\omega) \quad (2)$$

$$T_S = g^*(i\omega)\theta(i\omega) \quad (3)$$

The inertia torque T_I is governed by the inertia of the rotating parts of the motor, the angle of rotation and the frequency squared (Eq. 2). The sample torque T_S is dependent on the material and the test geometry. For a Newtonian fluid ($\eta^* = \eta$), the material function represented by the complex modulus reduces to:

$$G^*(i\omega) = k_g g^*(i\omega) = \eta i\omega \quad (4)$$

k_g is a constant defining the test geometry. Eq. 1 can be arranged to show the system response for a Newtonian fluid:

$$\frac{\theta(i\omega)}{T(i\omega)} = \frac{(k_g/\eta)}{i\omega(\tau i\omega + 1)} \quad \text{with} \quad \tau = \frac{Ik_g}{\eta} \quad (5)$$

τ is a system response time, which increases with inertia and decreases with the sample viscosity. Part of the torque input is stored in the system (energy storage due to inertia). The stored energy interacting with the viscous contribution of the Newtonian fluid causes the system to behave like a viscoelastic fluid.

The best metric to evaluate the effect of inertia on the system's response is the ratio of inertia torque and sample torque. This ratio for a Newtonian fluid sample increases with the frequency and decreases with the viscosity (Eq. 6).

$$\frac{T_I}{T_S} = \frac{\theta_{SH}(i\omega)I\omega^2}{(\eta/k_g)\omega\theta_{SH}(i\omega)} = \frac{Ik_g\omega}{\eta} \quad (6)$$

For a Hookean solid ($G^* = G$), the ratio of inertia and sample torque increases with the frequency squared, the inertia and the geometry constant, but decreases with the material's stiffness. This system has a natural frequency at $\omega = \omega_b$ (Eq. 7).

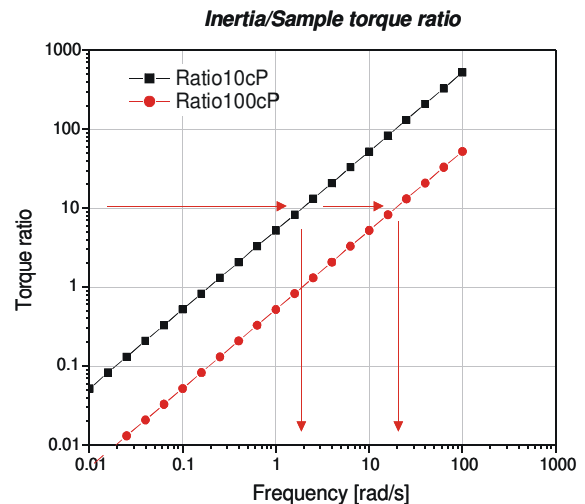


Figure 2 Ratio Inertia/sample torque for CMT rheometer. Inertia $I = 50 \mu\text{Nms}^2$

$$\frac{T_I}{T_S} = \frac{\theta_{SH}(i\omega)I\omega^2}{(G/k_g)\theta_{SH}(i\omega)} = \frac{Ik_g\omega^2}{G} \quad (7)$$

with $\omega_0 = \sqrt{\frac{G/k_g}{I}}$

Figure 2 shows the inertia/sample torque ratio for a 100cP and a 10cP silicone oil. A torque ratio of 10 (energy consumption of the inertia is tenfold the energy consumption of the sample) is obtained at 20 rad/s for the 100cP oil, at 2 rad/s for the 10cP oil.

The frequency dependence of the dynamic moduli G' and G'' as well as of the phase for the 100cP silicone oil is shown in figure 3. At low frequency the phase angle is -90° as expected for a Newtonian fluid. The storage modulus G' increases with a slope of 2 as a function of frequency. The scatter in the G' data results from the noise in the phase angle around 90° . Despite the inertia corrections, the effect of the motor inertia becomes dominant above 30 rad/s. At 100rad/s the inertia torque is about 50 times the sample torque for the specific CMT

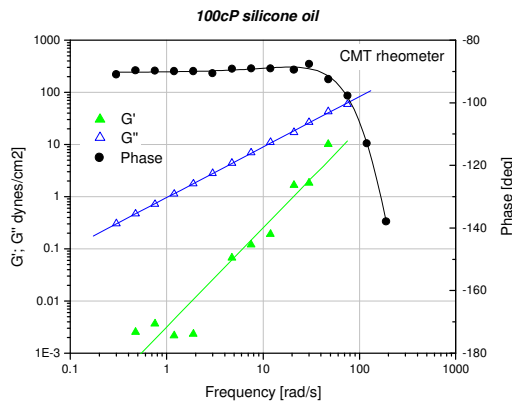


Figure 3. G' , G'' and phase for a 100 cP silicone oil measured in a CMT rheometer used in this study.

The measured G' contributions (slope 2) are related to the energy storage due to the system inertia. The measurement of structure in low viscosity fluids requires significant and accurate corrections of the inertia torque in order to eliminate the inertia contributions

in G' . Since the corrections are large, slight experimental errors or errors in the correction can easily falsify the measurement of the phase angle. G' in figure 3 is not a material property, but characterizes the system inertia.

Torque balance and torque ratio for SMT rheometers

Following the equations which govern the torque balance in an SMT rheometers:

$$T_m = T_I + T_S \quad (8)$$

$$T_I = I\omega^2\theta_{TR}(i\omega) \quad (9)$$

$$\theta_{TR}(i\omega) = T_m C_{TR} \quad (10)$$

$$T_I = T_m C_{TR} I \omega^2 \quad (11)$$

$$T_S = T_m - T_m C_{TR} I \omega^2 = g^*(i\omega)\theta_m(i\omega) - \theta_{TR}(i\omega) \quad (12)$$

The inertia torque depends on the inertia, the angular displacement of the torque transducer and the frequency squared. For transducers of the FRT type³, the angular displacement, i.e. the compliance C_{TR} is virtually zero at low frequency. The ratio of inertia/sample torque in this case (Eq. 13) is independent on the material's viscosity, the geometry constant and the strain amplitude. The remaining parameters in the torque ratio equation, transducer inertia and compliance are only a function of the instrument and not the material.

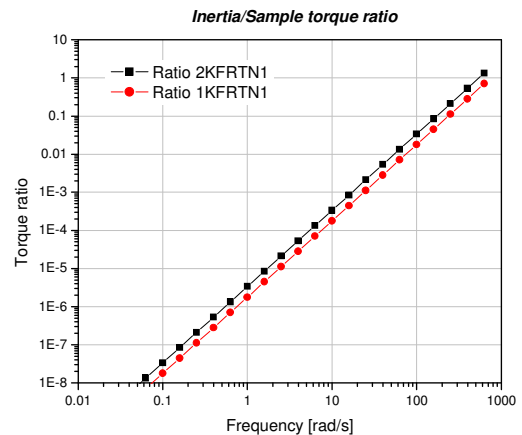


Figure 4 Ratio Inertia/sample torque for SMT rheometer.

$$\frac{T_I}{T_S} = \frac{C_{TR} I \omega^2}{(1 - C_{TR} I \omega^2)} \quad (13)$$

In figure 4 the torque ratio for the two standard FRT torque transducers (1KFRTN1 and 2KFRTN1)* are given. The torque ratios are much smaller than for the CMT

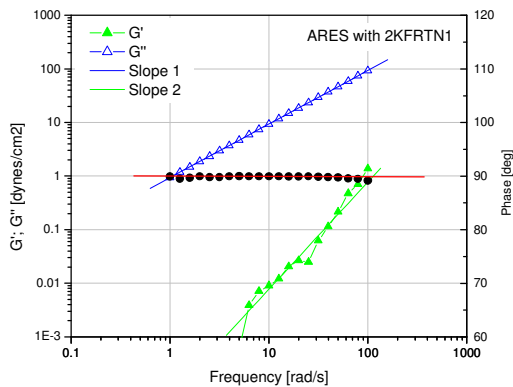


Figure 5. G' , G'' and phase for a 100cP silicone oil measured in an SMT rheometer

rheometers and do not exceed a few % at 100rad/s despite the relatively high transducer inertia of 60.6 or 134 μNms^2 respectively.

The dynamic moduli G' , G'' and the phase angle for the 100cP silicone oil, measured with an SMT rheometer are shown in figure 5. As expected, up to a frequency of 100rad/s the phase angle does not deviate from 90° at all. The measured values for G' are 25 times lower than the values obtained in the CMT rheometer. This means, that the energy storage contributions due to the inertia are 25 times lower in the SMT rheometer. Weak structures in low viscosity fluids can therefore be measured accurately whereas system inertia in CMT rheometers under the same testing conditions is dominant.

A typical example of a low viscosity structured fluid is coffee cream. (Figure 6) The viscosity is approximately 10cP. Coffee cream has some structure, which shows as

significant higher values of G' and a slope of 1.06 in G' vs. frequency. In a CMT rheometer the system inertia would dominate G' and mask the contributions of the structure of the material in G' .

Effect of sample inertia

Dynamic mechanical measurements are usually evaluated under the assumption, that inertia effects of the fluid can be neglected. This condition, also referred to as “gap loading”⁴ condition, is fulfilled when every fluid element between the driving and the stationary surface of the test geometry is oscillating in phase and experiences the same velocity at any point in time. A necessary requirement for this condition is, that the wavelength of the propagated shear wave is much larger than the sample gap. In this case the fluid sees only a small portion of the shear wave and no significant gradients in either the phase angle or velocity exist. If the shear wavelength has the same magnitude than the sample gap, the amplitude and phase of the velocity gradient are a function of the position within the gap. As a result the phase

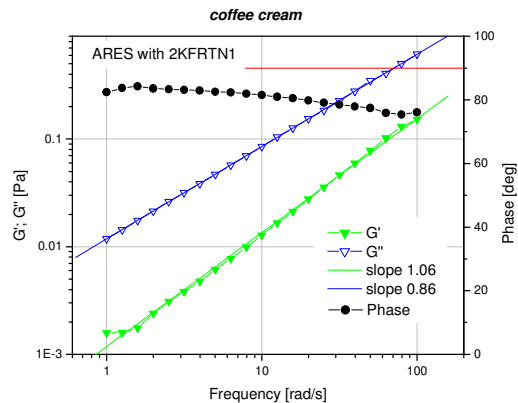


Figure 6. G' , G'' and phase angle for coffee cream measured in an SMT rheometer

angle of the strain rate at any point in the fluid lags the strain rate at the driven surface by some factor Φ .

In order to evaluate whether fluid inertia effects are significant or not, the complex Stokes number m (the ratio of inertial and viscous forces)⁵ is introduced. The gap

* 1KFRTN1 and 2KFRTN1 are transducers for the ARES rheometer from TA Instruments

loading condition is fulfilled for $m \rightarrow 0$. For a viscoelastic fluid the wavelength of a shear wave is given by:

$$\lambda = \frac{2\pi}{\cos(\frac{\delta}{2})} \left[\frac{\eta^*}{\rho\omega} \right]^{1/2} \quad (14)$$

with

$$m = \sqrt{\frac{\rho\omega}{\eta^*}} h \quad (15)$$

For $m=2\pi$, the shear wave has exactly the same length than the sample gap h and the sample inertia contributions have to be corrected for.

Schrag, Guess and Thurson⁶ have solved the wave propagation equation for the velocity gradient at any point in the sample gap and proved the validity of the solution. The effect of the fluid inertia is a phase shift of the velocity gradient by Φ , this term being a function of both an attenuation coefficient α and a phase factor β . At the stationary surface of the SMT rheometer (transducer side) the phase correction is:

$$\Phi = -\frac{\delta}{2} + \tan^{-1} \left[\frac{\tanh(\alpha h)}{\tanh(\beta h)} \right] \quad (16)$$

The measured complex viscosity has to be corrected as follows:

$$\eta^* = \frac{\eta_m^*}{f_c} \quad \text{with} \quad f_c = (h\dot{\gamma}/v) \exp(i\omega) \quad (17)$$

Figure 7 demonstrates the effect of the sample gap h on the fluid inertia for a 10cP silicone oil. For gap settings above 0.5, a significant decrease in phase angle and a slight roll-off in the complex viscosity are seen. This effect shifts to lower frequency with increasing sample gap. The solid line in figure 7 shows the phase angle and the complex viscosity after correction. For measurements in oscillation on very low viscosity fluids, the wave propagation

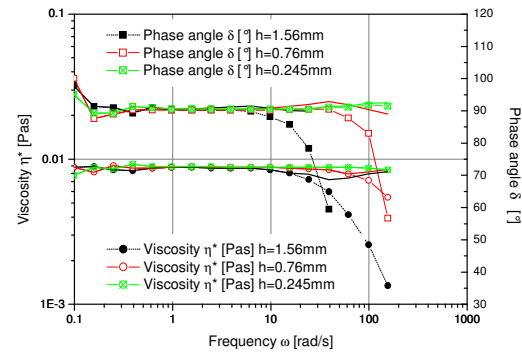


Figure 7. Effect of fluids inertia on phase and magnitude (viscosity)

correction is necessary, especially when concentric cylinders (typical sample gaps 0.5 and 1mm) are used.

CONCLUSION

SMT rheometers are the choice for performing oscillatory measurements on low viscosity fluids. The contribution of the system inertia is much lower than for CMT rheometers and inertia is not a function of the material's viscosity. In addition to the system inertia, the fluids inertia needs to be taken into account, when the geometry requires sample gap larger than 0.5mm, f.ex: concentric cylinders.

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REFERENCES

1. Krieger, I.M. (1990), *J.Rheology*, **34**, 4, 471-481.
2. Franck, A. (1992), *Proc. XIth Int'l Cong. Rheology, Brussels, August 1-21, 1992*, Vol2, 982
3. Force rebalance transducer, U.S.Patent No. 4601195
4. Schrag, J.L. (1977), *Transactions of the Society of Rheology*, **21**, 3, 399-413
5. Grehlinger, M. (1986), Rheometrics Internal document

6. Schrag, J.L., Guess, J.F., Thurston, G.B.
(1965), *Journal of applied Physics*, **36**, 6
1996-2000