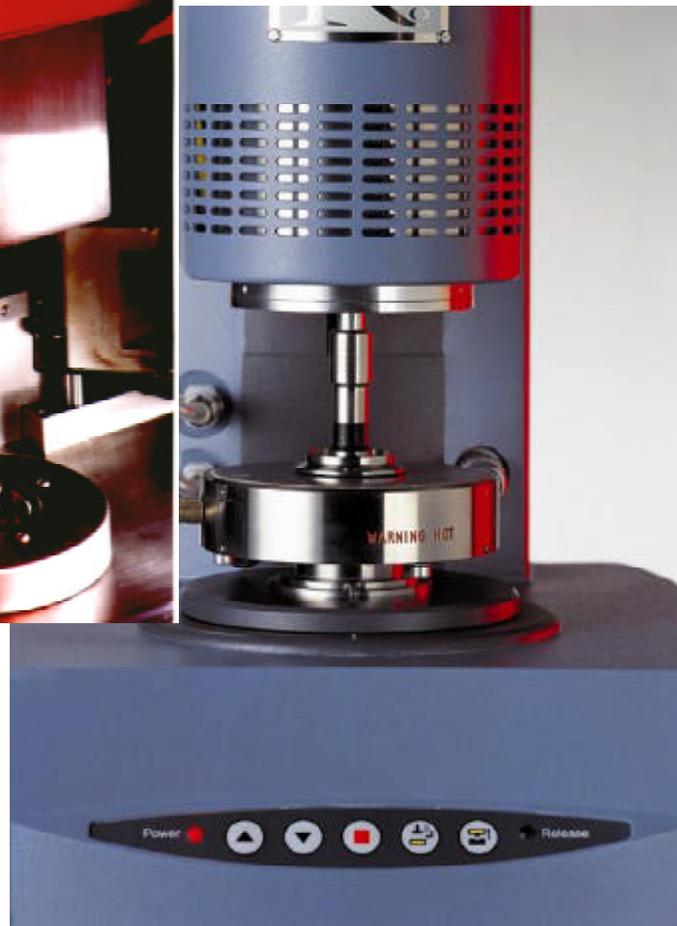




Understanding Instrument Inertia Corrections in Oscillation



Instrument Inertia Correction during Dynamic Mechanical Testing

Scope

Results from oscillation tests performed with rotational rheometer can be corrupted due to huge instrument inertia contributions, related to the periodic acceleration and deceleration of the motor shaft. In a rheometer the torque output of the motor is composed of the torque required to overcome the instrument inertia and the torque deforming the sample. The torque contributions for inertia increase with the applied frequency to the power two and usually dominate the instrument response for low viscosity test samples. Rotational rheometers with a separate transducer are much less susceptible to inertia effects, since the sample torque is determined independently from the motor torque.

Introduction

Two types of rotational rheometers are commercially available. These rheometers were traditionally referred to as controlled strain (CR) or controlled stress (CS) rheometers. The controlled

stress rheometer consists of a motor with a drive shaft and position sensor, supported by a “frictionless” bearing (Figure 1). In its native mode, the CS instrument applies a command torque in an open loop (no feed back) and measures the angular position. Since the expression “controlled stress” rheometer is used differently by different people, this type of rheometer will be referred to as single head (SH) rheometer in the following. The controlled strain or dual head (DH) rheometer consists of two separate elements, the motor providing the sample deformation and the torque transducer measuring the sample torque. In this design motor friction and motor inertia do not effect the torque measurement. How the instrument inertia does effect the test results for SH and DH rheometers and how inertia is corrected for, is the subject of the following sections.

Governing equations

Neglecting the bearing friction, the command or measured torque for a SH rheometer is the

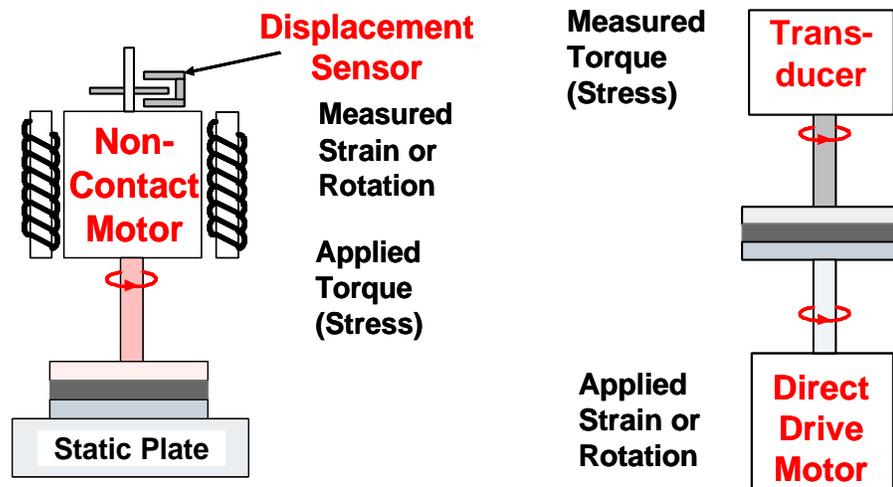
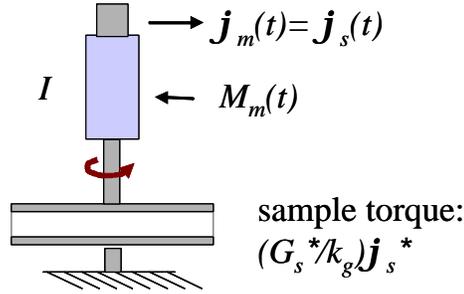


Figure 1: Schematic diagram of a CS (SH) and a CR (DH) rheometer

sum of the torque necessary to deform the sample and the torque to overcome the inertia as shown in figure 2^(*). The sample torque $M_s^* = M_s^o \exp\{i(\omega t + \mathbf{d})\}$ is



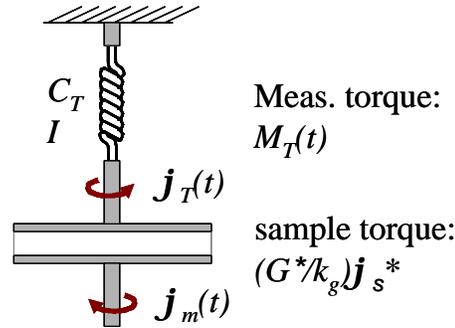
$$M_m^* = M_s^* + M_I^*$$

$$M_s^* = \frac{G_s^*}{k_g} \mathbf{j}_m^*$$

$$M_I^* = -I\omega^2 \mathbf{j}_m^*$$

$$M_m^* = \frac{G_s^*}{k_g} \mathbf{j}_m^* - I\omega^2 \mathbf{j}_m^* = \mathbf{j}_m^* \left(\frac{G_s^*}{k_g} - I\omega^2 \right)$$

Figure 2: Torque balance for a single head rheometer



$$\text{Sample deform. : } \mathbf{j}_m(t) - \mathbf{j}_T(t) = \mathbf{j}_s(t)$$

$$M_T^* = M_s^* + M_I^*$$

$$M_s^* = \frac{G_s^*}{k_g} [\mathbf{j}_m^* - \mathbf{j}_T^*]$$

$$M_I^* = -I\omega^2 \mathbf{j}_T^*$$

$$M_T^* = \mathbf{j}_T^* / C_T$$

$$M_T^* = \mathbf{j}_T^* / C_T = \frac{G_s^*}{k_g} [\mathbf{j}_m^* - \mathbf{j}_T^*] - I\omega^2 \mathbf{j}_T^*$$

Figure 3: Torque balance for a dual head rheometer with separate transducer

(*) Bearing friction is neglected in this analysis

(**) see Product note APN006 Cross Correlation

the product of the material function (here the complex modulus) and the sample deformation, divided by the geometry constant k_g . The inertia torque M_I^* is the product of the moment of inertia I and the angular acceleration. The solution of the torque balance in figure 2 for an imposed sinusoidal torque is as follows:

$$G_s' = G_m' + I\omega^2 k_g \quad (1)$$

$$G_s'' = G_m''$$

The sample storage modulus G_s' is the sum of the real part of the measured modulus and an inertia correction term, which increases with the test frequency squared. The loss modulus is not corrected. The real and the imaginary parts of the measured modulus G_m^* are obtained from **the measured position** $\mathbf{j}_m(t) = \mathbf{j}_m^o \cos(\omega t + \mathbf{d}_m)$ and stress $M_m(t) = M_m^o \cos(\omega t)$ signals, using a cross correlation technique^(**). \mathbf{d}_m is the measured phase or raw phase, \mathbf{d} is the sample phase.

The torque balance for the DH rheometer is illustrated in figure 3. Due to the non-zero transducer compliance C_T , the sample deformation is the difference of the angular displacement of the motor and the transducer shaft. With the transducer compliance constant, the complex transducer torque M_T^* is proportional to the complex transducer displacement \mathbf{j}_T^* . Transducer inertia contributions are small, because they are related to the transducer angular displacement $\mathbf{j}_T(t)$. The solution of the torque balance for an imposed sinusoidal deformation is more complex than the solution for the SH rheometer, and both measured moduli, G_m' and G_m'' have to be corrected for transducer compliance (C_T) and Inertia (I).

Force Rebalance Transducers (FRT) are quasi infinite stiff transducers below a test frequency of 100 rad/s and have a

low compliance value C_T (10^{-2} rad/Nm for the 1KFRTN1 at 10 rad/s). As a consequence the transducer displacement approaches zero and so does the inertia term. The motor displacement is now proportional to the sample deformation.

$$\begin{aligned} \mathbf{j}_T^* &\rightarrow 0 \\ I\omega^2 \mathbf{j}_T^* &\rightarrow 0 \end{aligned} \quad (2)$$

$$\mathbf{j}_s^* \rightarrow \mathbf{j}_m^*$$

In this case, the measured loss and storage modulus and the phase angle are true material quantities.

$$\begin{aligned} G_m' &= G_s' \\ G_m'' &= G_s'' \end{aligned} \quad (3)$$

$$\mathbf{d}_m = \mathbf{d} = \arctan\left(\frac{G_m''}{G_m'}\right) \quad (4)$$

Monitoring Instrument Inertia Effects

The best parameter to quantify the extend of inertia effects on the measured data is the ratio of inertia M_I to sample torque M_s , referred to as torque ratio (TR).

For DH rheometers, assuming the transducer scompliance is low and constant, the torque ratio depends only on the instrument parameters I and C_T as:

$$\frac{M_I}{M_s} = \frac{C_T I \omega^2}{1 - C_T I \omega^2} \quad (5)$$

For SH rheometers, the torque ratio depends also on the test sample itself. For a Newtonian fluid, the torque ratio is inverse proportional to the material's viscosity.

$$\frac{M_I}{M_s} = \frac{I\omega^2 \mathbf{j}_m^* k_g}{G_s^* \mathbf{j}_m^*} = \frac{I\omega k_g}{\mathbf{h}} \quad (6)$$

$$G_s^* = \mathbf{h}\omega \text{ Newtonian}$$

The torque ratio has been calculated for both types of rheometers as a function of frequency and is shown in figure 4. For SH rheometers the TR is a function of the material's viscosity and the instrument's moment of inertia. The torque ratio is equal to 1 (sample and inertia torque are the same) for the AR-G2 rheometer at approximately 1 rad/s for a 6 mPa·s silicone oil.

The torque ratio of DH rheometers depends only on rheometer parameters. Since the FRT transducer is quasi infinite stiff, the inertia torque is orders of magnitude lower, than the sample torque. Note, that above 8 rad/s, the inertia torque increases stronger. At this point the transducer compliance C_T increases. Nevertheless for the standard 1KFRTN1 transducer, the contribution of inertia torque is only around 2-3% at a frequency of 100 rad/s .

Since the TR is direct proportional to I , it is important for SH rheometers to have a system inertia as low as possible. The torque ratio for a high inertia system ($90 \mu\text{mNs}^2$) is shown for comparison in figure 4.

Another useful parameter to monitor the effect of inertia is the measured (raw) phase angle \mathbf{d}_m . If only inertia of the drive shaft is present $G_s^* = 0$ (inertia dominates),

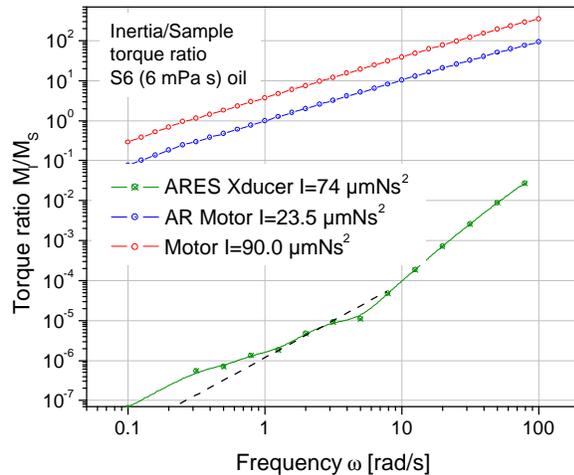


Figure 4: Torque ratio for SH and DH rheometers

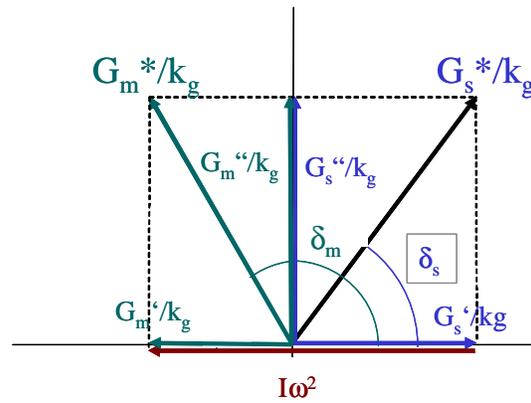


Figure 5: Vector representation of inertia, measured and corrected modulus

$$G_m^* = -I\omega^2 \quad (9)$$

$$M_m^* = -I\omega^2 \mathbf{j}_m^*$$

The solution of this equation for a sinusoidal torque input $M_m^* = M_m^o \sin \omega t$ is given as:

$$\begin{aligned} \mathbf{j}(t) &= -\omega^2 \frac{M_m^o}{I} \sin(\omega t) \\ &= \omega^2 \frac{M_m^o}{I} \sin(\omega t + \mathbf{p}) \end{aligned} \quad (10)$$

The measured phase shift between displacement and torque signal (raw phase) is π . Inertia shifts the phase angle to 180° , the phase angle for a Newtonian fluid is 90° .

Figure 5 shows the vector representation of the measured (G_m^*) and the sample (G_s^*) modulus for SH rheometers. If inertia dominates, G_s'/k_g and G_s''/k_g go to zero, the term $|I\omega^2|$ is equal to $|G_m^*/k_g|$ and δ_m is π or 180° . The raw phase δ_m is an-

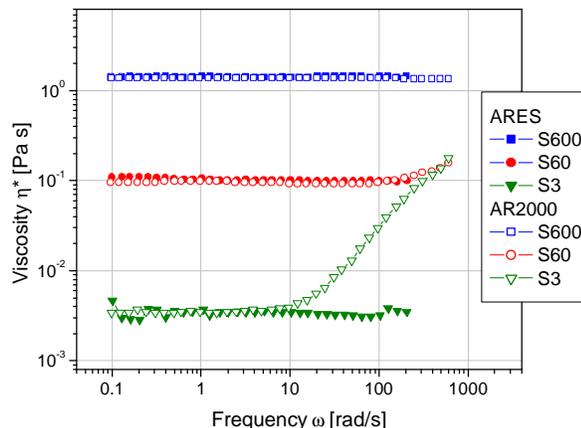


Figure 6: Complex viscosity of 3 silicone oils, measured on the ARES and the AR2000

other monitor of the inertia contributions. Measurements, exhibiting a raw phase value above 175° need to be evaluated very carefully, as experimental errors and system corrections are much larger than the final test results.

To demonstrate the effects of inertia, oscillation measurements on three silicone oils between 3 and 1000 mPa·s have been performed in the frequency range from 0.1 to 500 rad/s. (Figure 6) The complex viscosity $\mathbf{h}^* = G^*/\omega$ obtained with the DH rheometer (ARES) is constant over the complete frequency range for all three silicone oils. The SH rheometer provides good data for the 1000 cP oil, but shows a deviations for the 100 and 3 cP oil at 100 respectively 10 rad/s. The corresponding raw phase and corrected phase for the SH rheometer are shown in figure 7. For the 3 cP oil, the raw phase reaches a phase angle of 170° at 1 rad/s already. The corrected phase is wrong above 8 rad/s and the complex viscosity above 20 rad/s. What is the reason for the deviation of the corrected test results in SH rheometers?

Inertia Correction

From the equations (1) it is evident, that the instrument inertia contribution effects the storage modulus G' . The energy to overcome inertia is stored in the momentum of the rotation motor shaft, and is not dissipated. The increase of the complex viscosity in figure 6 with increasing frequency results from strong G' contributions under the given test conditions. For a more detailed investigation, the complex moduli G' and G'' for a 6 mPa·s silicone oil (S6), were determined in an ARES and AR2000 rheometer. For a Newtonian fluid, no G' contributions are expected; as such the G' data obtained with the ARES are less than 0.1% of the G'' values and

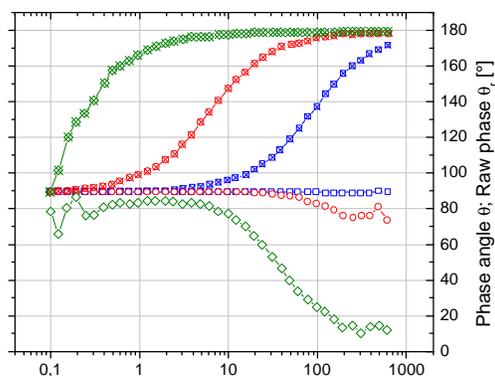


Figure 7: Raw and corrected phase angle for 3 silicone oils measured with the AR2000.

represent system noise. For the AR2000, the measured G' data are significant and show a plateau at low frequency. Above 10 rad/s the G' increases with the frequency to the power 2 and amounts to 20-30% of the complex modulus at 100 rad/s.

In SH rheometers, the correction of instrument inertia is orders of magnitude higher than for the DH rheometer. If inertia becomes dominant and the moment of inertia is only slightly under corrected, G' increases at a slope of two with frequency (Inertia torque depends on the product of the moment of inertia and the angular frequency squared). This is shown for a low viscosity silicone oil (S6) in figure 9. The instrument, the sample and inertia torque are plotted as a function of frequency. Both instrument and sample

torque turn into large numbers with increasing frequency, while the sample torque remains small and is two orders of magnitude lower at 100 rad/s. The corrected phase decreases above 10 rad/s. This means, that G'_s becomes more significant and increases with a slope of 2 (see also G'_s for the AR2000 in figure 8). In this case, inertia contributions are under-corrected. A slight increase of the moment of inertia I by 0.1% changes the sample torque by 10% at 100 rad/s and G' even more (*). If the inertia is over-corrected, the sample phase becomes larger than 90° and G' is negative.

In the DH rheometer, the situation is reversed. The inertia torque is in the order of 5 decades lower than the measured torque at low frequency and consequently, the sample torque is virtually identical to the measured torque. (figure 10). Above 8 rad/s the inertia torque increases faster with frequency (see also figure 4) - this is due to the decrease of the transducer's stiffness at higher frequency. At 100 rad/s, the inertia torque is only 2.7% of the sample torque and inertia corrections can be neglected. Raw phase and sample phase are the same and stay at 90° throughout the frequency range up to 100 rad/s.

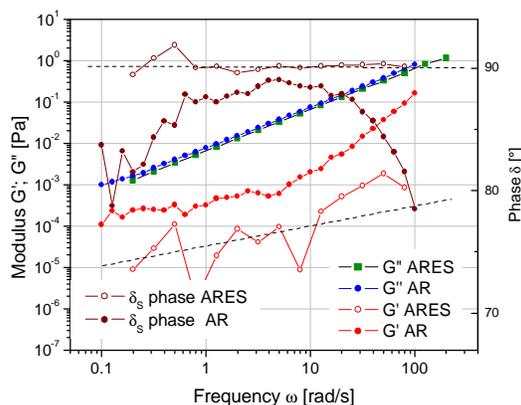


Figure 8: Dynamic moduli and phase angle for a 6 cP silicone oil measured with the ARES and AR2000 rheometer

The impact of inertia contributions is significant when analysing weak structures in low viscosity fluids. The test sample in figure 11, a printing ink, is expected to have a light structure. In this graph the ink results are compared to the low viscosity silicone oil S6, measured with the ARES rheometer. The test temperature for the silicone oil was increased to match the viscosity of the ink at 20 °C. Therefore the G'' data for the ink and the silicone oil measured on the AR2000 and the ARES rheometer superpose very well. The lowest curve is the G' of the sili-

cone oil and represents a kind of system “G’ resolution”. The upper G’ curves are obtained for the ink. Clearly the G’ data are higher than

for the silicone oil and the data obtained from the two instruments match quite well. This is surprising, considering the amount of correction necessary for the SH rheometer. The higher G’ response for both SH and DH rheometer supports the existence of a slight structure for the printing ink.

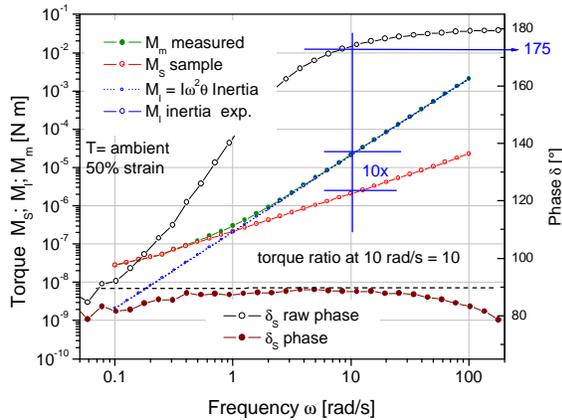


Figure 9: Measured, corrected modulus and inertia term for the S6 silicone oil measured with the AR2000

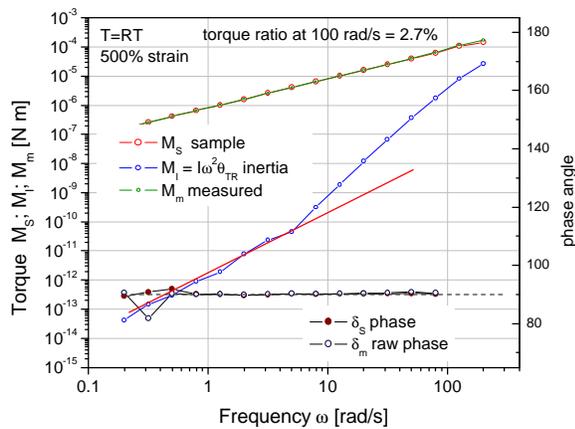


Figure 10: Measured, corrected modulus and inertia term for the S6 silicone oil measured with the ARES

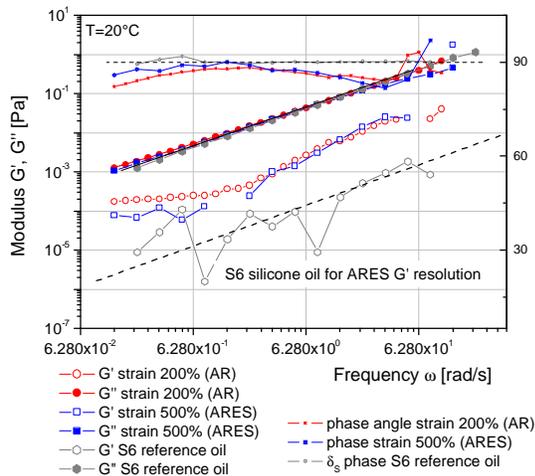


Figure 11: Dynamic moduli for a printing ink measured with ARES and AR2000

How do SH and DH rheometer differentiate for this application?

The SH rheometer has to be calibrated in term of inertia and torque very carefully to be able to measure small values of G’ of a low viscosity fluid. Slight deviations will cause significant variations of the final data - SH rheometers therefore are not reliable for this application. Careful calibration and verification with a Newtonian oil is necessary.

The ARES with FRT transducer doesn’t need any sophisticated calibration and inertia corrections up to 100 rad/s. Minor errors in the system setup parameters do not significantly effect the test results.

Conclusions

The ARES rheometer measures the dynamic properties from high to low viscosity up to 100 rad/s without major correction of the phase angle due to inertia effects. For the AR rheometer the sample response for low viscosity materials is only a small fraction of the measured G_m^* and a valid data correction can only be done after careful calibration of the system inertia and mapping of the instrument. As such, oscillation data obtained on non Newtonian low viscosity fluids can easily be corrupted. It is therefore an absolute must for SH rheometers to monitor the level of correction done to the measured data. Two parameters are available to be used, the ratio of inertia to sample torque or the measured (raw) phase.



Keywords: moment of inertia, inertia corrections, raw phase, controlled stress rheometer, strain controlled rheometer