COMMENTS ON THE APPLICATION OF STRESS CONTROLLED AND STRAIN CONTROLLED RHEOMETERS

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ABSTRACT

Examples will illustrate areas of application of stress controlled and strain controlled rheometers. Closed loop and open loop circuits in rheometers entail different performance characteristics. The significance and implications of inertia, inertia correction and compliance will be discussed for both types of rheometers.

INTRODUCTION

Rotational rheometers differ essentially in the type of commanded input function used, i.e. either stress controlled or strain controlled. Both types of rheometers can operate dynamic mechanically with an oscillating input signal as well as in steady rotation with a commanded stress or shear rate. Instrument inertia, instrument compliance as well as motor response times, however, assume different degrees of significance depending on the type of rheometer under consideration and must be taken into account when evaluating rheological material data. For example, Fig.1 shows a creep test of water carried out on a stress controlled rheometer - Rheometrics Dynamic Stress Rheometer, DSR. The viscosity is distorted by inertial effects up to 100s. Fig.2 shows the temperature dependence of the storage modulus G' of polymethyl-methacrylate (PMMA) and the effect of compliance in the transition zone. The PMMA data were measured on a strain controlled instrument - Rheometrics Dynamic Analyzer, RDAII.

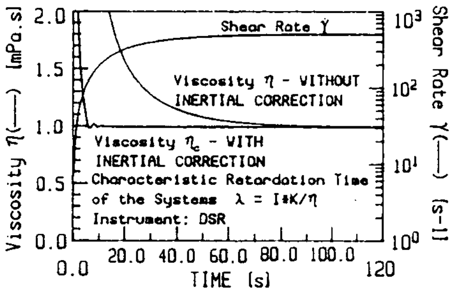

Figure 1: Water and inertial retardation.

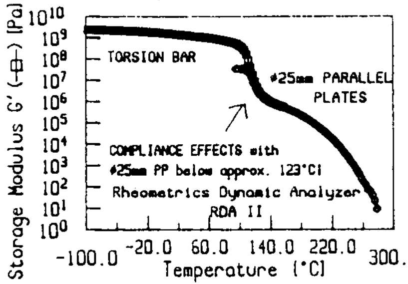

Figure 2: PMMA and compliance effects.

SYSTEM SETUP AND EXAMPLES OF APPLICATION

Strain Controlled Rheometers

In strain controlled rheometers the actuator and torque transducer are separate system components whereas in stress controlled rheometers they comprise one unit. A closed loop actuator enables a strain and shear rate input free of inertia. In general, inertial effects of torque transducers of the FRT-type (Force Rebalance Transducer) can be neglected as the electromagnetic circuit ensures a null-position even in the upper frequency range. This is exemplified by the frequency sweep of an approx. 10mPa.s silicon oil in Fig.3. The data were measured with a strain controlled rheometer - Rheometrics Fluids Spectrometer, RFSII. On the other hand spring-based torque transducers are prone to significant inertial effects at high frequencies.

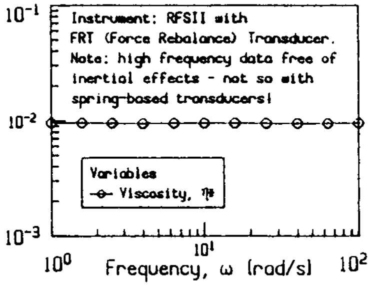

Figure 3: Silicon oil frequency sweep.

If test geometries with a high mass and inertia such as a large couette are attached to a torque transducer additional inertial contributions may occur in the upper frequency range.

Compliance effects occur in all strain controlled instruments most often under conditions of either high frequencies and high viscosities and/or low temperatures and high moduli. Their influence on the measured data is dependent on the relative magnitude of the sample stiffness to the instrument's transducer stiffness. The latter is known for every transducer and can be used in a software correction of the shear moduli as well as for calculating the largest measurable material modulus for a given test geometry.

In stress relaxation experiments all actuators require a finite time to apply the imposed strain. There will always be a characteristic actuator response time of the order of milliseconds.

Stress Controlled Rheometers

The fact that stress controlled rheometers have an open loop actuator implies inertial effects in all transient and oscillatory motions. These have to be taken into account and corrected accordingly. Inertial corrections are necessary to ensure meaningful rheological material data. Fig.4/Franck/ shows the system setup of a stress controlled rheometer. As shown inertial effects occur because the applied torque of the actuator has to overcome not only the viscous drag due to the sample but also the inertia of the actuator, the position sensor and the attached tool as well as any frictional losses in the air bearing of the actuator.

For low viscosity fluids generating low torques frictional losses in the air bearing cannot be neglected. On the other hand inertial effects for low viscosity materials such as 10mPa.s can be neglected if the gap between the test surfaces is small. This has been discussed in detail by /Grehlinger/ and by /Böhme and Stenger/. A software correction of fluid inertia is available for situations with significant fluid inertial effects.

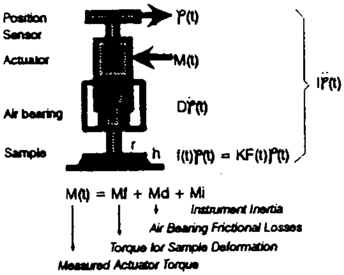

Figure 4: Stress controlled rheometer.

Solving the equation in Fig 4. leads to a second order differential equation with a characteristic retardation time ß=IxK/eta, where ß is a function of a geometrical constant K, system inertia I and sample viscosity eta.

In practice the effect of this retardation time can be clearly seen in any transient motion such as a creep test and stress ramp and can delay proper data collection by up to 100s as shown by the creep test of water in Fig.1. Only after 100s does the measured viscosity approach steady state, i.e. 1mPa.s. On the other hand the inertia corrected viscosity approaches 1mPa.s in less than 10s. A reduction in tool inertia and plate gap, for instance, reduces the retardation time of the system. A software inertial correction, however, is always required unless the viscosity is sufficiciently high to reduce ß to the order of milliseconds.

In both multistep creep and creep/recovery experiments as well as in stress ramps inertial corrections are virtually mandatory to determine meaningful rheological material data. /Krieger/ has shown that inertial effects rapidly gain in significance with increasing stress ramp gradients as well as decreasing material viscosities. Apart from making rapid thixotropic transformations inaccessible inertial effects can generate false thixotropic material behaviour as shown in Fig.5.

In dynamic mechanical analysis a sinusoidal torque is applied and inertial effects are reflected by a limiting upper frequency that is a function of material viscosity, system inertia and test geometry such as plate gap. The extent of inertia correction becomes apparent in Fig.6 where a PVC plastisol has been subjected to a frequency sweep with and without inertial correction. Notice the strong effect of inertia on the storage modulus G'. For high viscosity materials such as PDMS inertial effects at high frequencies tend to diminish as shown in Fig.7. This is due to the fact that for high viscosity materials significantly more energy is required to deform the sample than to overcome inertial and frictional losses. Thus inertial corrections can be neglected for high viscosity polymers, i.e. most thermoplastic melts.

CONCLUSIONS

Both strain controlled and stress controlled rheometers can operate dynamic mechanically with an oscillating input signal commanded stress or shear rate as well as in steady rotation with a commanded stress or shear rate. However, they differ substantially in terms of compliance and inertial effects as shown by a number of examples.

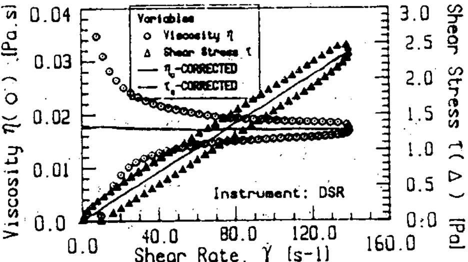

Figure 5: Stress ramp and inertia

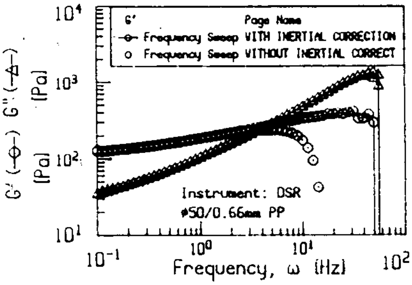

Figure 6: PVC plastisol and inertia.

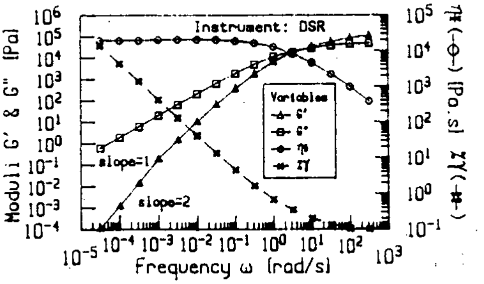

Figure 7: PDMS frequency sweep.

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