ARES-G2: A NEW GENERATION OF SEPARATE MOTOR AND TRANSDUCER RHEOMETERS

SCOPE

The new ARES-G2 is a rotational rheometer based on the unique concept of separation of motor and force/torque transducer. Designed from ground up, a key objective of the development project was to provide increased flexibility designing rheological experiments and to allow new and application specific test procedures. In order to achieve these goals all major instrument components such as the actuator, transducer, stage, data acquisition, environmental systems, etc. are developed as independent intelligent sub-assemblies, controlled by a central processor. The rigid firmware based on fixed test modes is replaced with a versatile user interface allowing a free combination of instrument instructions, which are downloaded to the instrument prior testing. Fast digital signal processing replaces the analog electronics providing faster, more accurate motor and transducer control and allowing the implementation of full stress control in oscillation and transient test modes. Significantly improved data acquisition with 5 fast data channels in all test modes enables SAOS and enhanced LAOS testing with complete support of higher harmonic analysis.

INSTRUMENT DESIGN

The ARES-G2 is built into a cast ductile iron frame for maximum stiffness & damping as well as thermal and mechanical stability. A brushless DC motor is mounted in the base of the instrument and the transducer on the bracket of the vertical slide. A high resolution linear optical encoder measures the stage position. An LCD touch screen in the front provides instrument and testing information and permits interactions with the instrument during sample loading and in between tests. Two rails on both sides support options such as the gas convection oven and/or accessories such as a draft shield, additional illumination, etc.

TRANSDUCER

The transducer used in the ARES-G2 rheometer is a patented torque and force "rebalance" transducer. Similar to a laboratory balance, the sample torque (force) is rebalanced by a torque (force) generated in the transducer. A rotary (linear) motor applies the reactive torque (force) and fast position feed back from a capacitive position sensor assures a quick response which results in quasi zero deflection of the transducer shaft. The motor current is a measurement of the reactive sample torque i.e. force.

The main benefit of the rebalance technique is the virtually, non compliant behavior of the transducer. However in reality an absolute zero position does not exist, which means that the transducer has a very high, nevertheless finite compliance in the order of magnitude of the compliance of the test geometry. The measured angular displacement is the difference between motor and transducer motion and therefore is independent of the transducer compliance.

At frequencies above 100 rad/s the time constant of the transducer control loop is too low to suppress the movement of the transducer shaft completely. In this case, the measured torque (motor current) is the sum of sample and inertia torque contributions (TA application note APN05: Understanding Inertia Corrections in Oscillatory Tests)

$$M_m = M_I + M_s$$

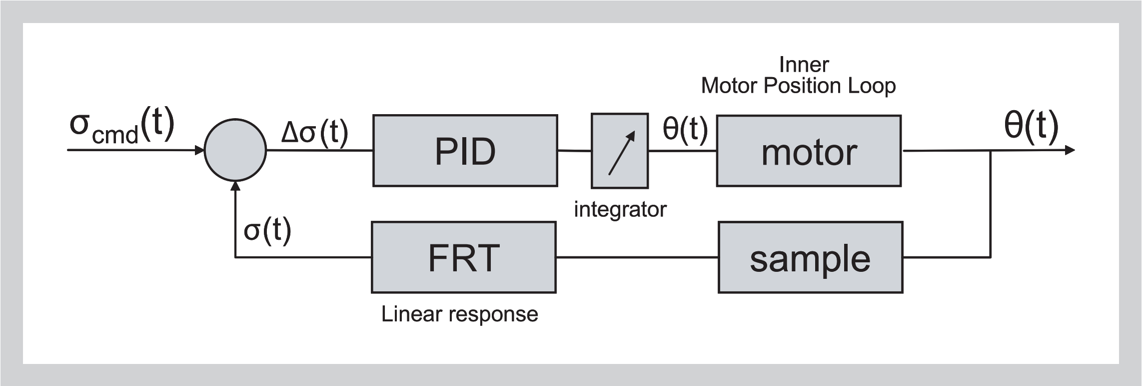

Figure 1: External control loop for stress controlled test with the ARES-G2.

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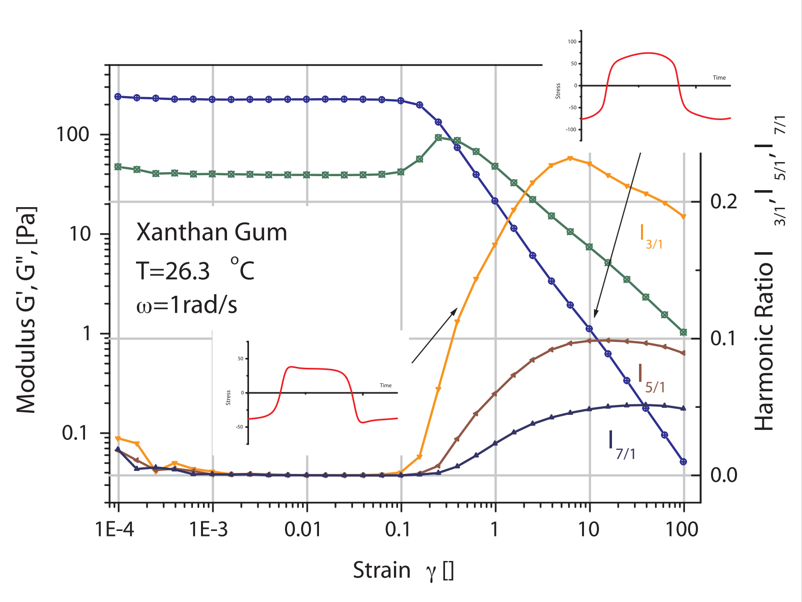

Figure 4 (above): Strain sweep showing G', G", 3rd, 5th, 7th harmonic ratio during the transition from the linear to the non-linear regime.

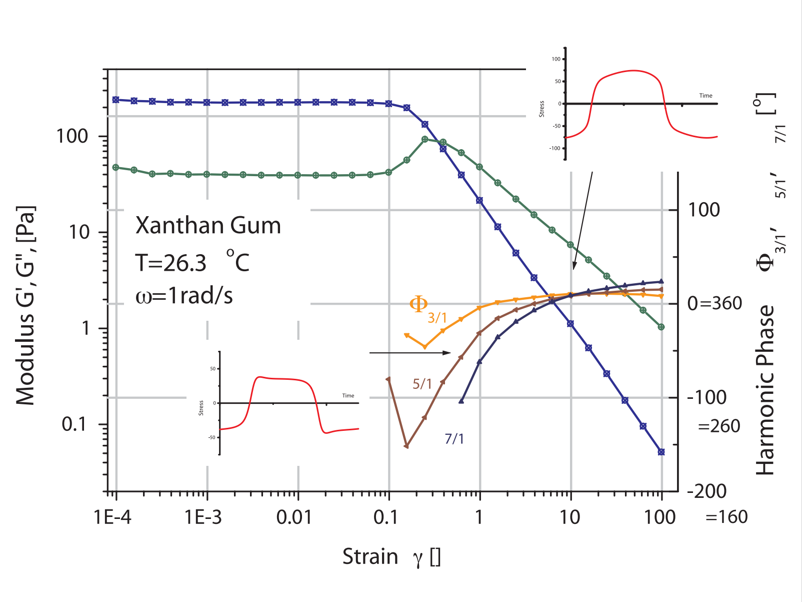

Figure 5: Strain sweep for Xanthan gum showing the transition from the linear to the non-linear regime. The harmonic phase increases from negative 90° to 0° at large strain.

ing stress controlled tests as well as during oscillation measurements.

Creep recovery experiments on the standard PIB 2490 have been performed at a creep stress so from 0.03 to 100 Pa (Figure 2). The creep compliance obtained from tests performed at a stress below 10 Pa is stress independent and as such is the viscosity calculated from the slope at steady state flow. The recoverable compliance is stress independent below 0.1 Pa, approximately 2 decades lower in stress. The highest equilibrium recoverable compliance measured is 0.011 1/Pa, and agrees with results obtained from the oscillation frequency sweep and the normal stress growth experiment.

The SMT control loop effectively eliminates inertia effects, which are responsible for creep ringing in an open loop experiment. In the ARES-G2 braking is accomplished through the inner motor position control loop. The recoverable compliance in figure 2 builds up slower than the creep compliance. It obviously takes longer for the feed back loop to stop the actuator (braking) before accelerating in the opposite direction than starting up from a rest position.

In order to prove that the same control strategy works with more challenging materials such as structured fluids exhibiting a yield stress, stress ramp experiments have been performed on a highly concentrated (4 %wt) aqueous Xanthan gum solution (Figure 3).

Applying a ramp rate of 1 Pa/s, the instantaneous viscosity and the strain are recorded versus time. The viscosity initially increases from zero to reach a maximum after 20 s and then drops by one decade within a few seconds. The slow increase of the viscosity is predominately a result of the viscoelastic nature of the Xanthan gum in the undisturbed state. Inertia effects are eliminated by the stress feed back loop. The viscosity maximum appears at a stress of 25 Pa. The stress at the viscosity maximum is used as a measure of the material's yield stress.

NON-LINEAR OSCILLATION MEASUREMENTS

Until recently, dynamic mechanical data were only analyzed when the conditions of linear viscoelasticity were fulfilled. In the linear region, the stress response to a sinusoidal strain excitation is sinusoidal and the magnitude ratio independent of the strain amplitude. The Fourier transformation of a sinusoidal signal shows only one single peak in the frequency spectrum. Manfred Wilhelm (Wilhelm M: Fourier Transform Rheology, Macromol. Mat. & Eng. 287 (2002) 83-105) was the first to systematically investigate experiments in the non-linear viscoelastic region. He measured the full stress response in the time domain during large strain oscillation experiments (LAOS) and evaluated the complete frequency response using Fourier transformation. In the frequency spectra odd higher order (3, 5, 7, ...) harmonics appear in addition to the fundamental stress beyond the on-set of non-linear behavior with growing strain.

The ARES-G2 offers two methods to analyze oscillation results outside of the linear range:

- Real time correlation to determine magnitude and phase of the fundamental and the harmonics while the experiment is running,

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- post processing of the raw strain and stress data using discrete Fourier transformation. In the linear viscoelastic region the storage and loss modulus G' and G" are independent of strain. Higher order harmonic contributions are zero. At the onset of non-linear behavior, along with changes of the storage and loss modulus, the harmonic stress contributions become significant. The higher harmonic contributions are typically presented as the ratio of the magnitude of the nth harmonic and the fundamental stress $I_{n/1} = I(\omega_n)/I(\omega_1)$.

For structured fluids such as the Xanthan gum, the strain sweep in the non-linear region shows some interesting features. At the on-set of non-linear behavior G' decreases. G" however increases first before deceasing along with G' at higher strain (Figure 4). The harmonic ratios $I_{3/1}$, $I_{5/1}$ and $I_{7/1}$ increase and show a maximum at a strain of 800 to 1000 %. It is worthwhile to compare the shape of the stress waveform at different strain amplitudes in the non-linear region. During the initial phase of the non-linear behavior, the stress wave is distorted and asymmetric. Near and beyond the maximum of the harmonic ratio, the shape of the waveform becomes more symmetric again, however with a flat peak, which is an indication of shear thinning. The analysis of the harmonic phase supports this trend (Figure 5).

At the on-set of the non-linear behavior, the harmonic phase is negative (- 90°) which explains the asymmetry of the stress wave and the tilt to the left. With increasing strain the harmonic phase approaches a strain independent value, here slightly above 0°, in the region the harmonic ratio has a maximum. The stress shape of the waveform is almost symmetric at this point.

CONCLUSION

The new ARES-G2 is a completely redesigned rotational rheometer based on the separate motor and transducer technology. Two key features have been presented:

- controlled stress testing and
- non linear oscillation analysis.

Stress can be controlled on an SMT rheometer, when the correct control strategy is applied. The advantage is that inertia effects during creep and recovery are eliminated by the inner motor position control loop. The high torque resolution of the FRT transducer allows recovery tests with the same accuracy than open loop measurements.

Higher harmonic analysis requires fast data acquisition in order to reduce random noise and provide the desired high frequency resolution. The ARES-G2 offers harmonic analysis up to the 9PthP order during standard oscillation tests as well as a fast transient sampling of the raw strain and stress signals for post analysis.

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