TA Instruments

Thermal Analysis & Rheology

RHEOLOGY APPLICATIONS NOTE

MULTIWAVE OSCILLATION

FEATURES/BENEFITS

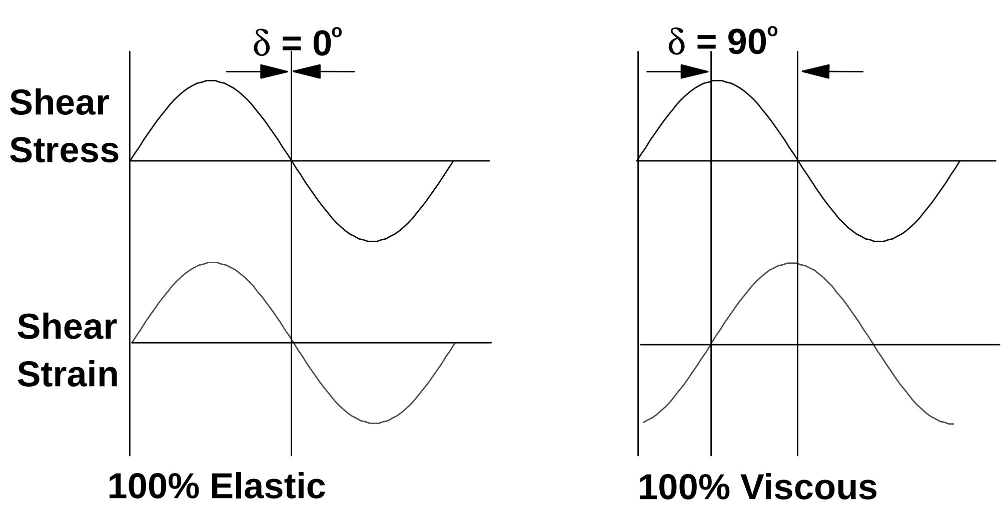
The oscillation mode in controlled stress rheology is widely used to nondestructively characterize the structure of materials. In this mode, a sinusoidal stress wave is applied to the material and the resultant strain wave is measured. Multiwave oscillation is an enhancement to this conventional (single frequency) oscillation mode which allows the sample material to be exposed simultaneously to multiple oscillation frequencies, thereby providing improved productivity (shorter analysis time) and increased insight into specific processes like high temperature curing. The TA Instruments CSL^2 Controlled Stress Rheometer with its wide torque range (0.1 µNm to 50 mNm) and responsive drag cup motor design is ideally suited for making multiwave oscillation measurements.

SPECIFICS

Figure 1 shows typical responses of materials to the application of a sinusoidal oscillating stress. If the material is purely elastic, the phase difference between the imposed stress and the resultant strain is zero degrees. If the material is purely viscous, the phase difference is 90 degrees. However, most materials are viscoelastic and, therefore, have phase differences between those values. This phase difference together with the amplitudes of the stress and strain waves are used to determine specific material properties such as G', G", η', δ, etc.

Multiwave oscillation is similar to this simple oscillation except that the material is exposed simultaneously to oscillations at two or more frequencies. As these multiple oscillations pass through the material, they act independently. However,

Figure 1. STRESS AND STRAIN WAVE RELATIONSHIP DURING OSCILLATION FOR PURE ELASTIC AND VISCOUS MATERIALS



the strain present in the material is the sum of the strains caused by all the individual oscillations. This is shown in Figure 2. (Note: The total resultant strain must be within the material's linear viscoelastic region.) Using suitable mathematical treatment of the complex wave forms produced (this is done automatically by the software), it is possible to obtain the same standard parameters obtained from conventional single freqeuncy oscillation. The result from multiwave oscillation, therefore, is the same as running multiple experiments each at a different frequency....but analysis time is significantly reduced! This reduced analysis time is particularly useful in QC or routine testing where a dynamic rheological fingerprint at a low, medium, and high frequency provides a rapid check of material behavior. Multiwave oscillation is also valuable in evaluating materials such as thermosets whose behavior is changing with time, and for which there is only a finite period of time to complete the evaluation before curing has stiffened the material to the point where it is outside the range of the rheometer. Figure 3, for example, shows the results of two frequency ramps on polyisobutylene. One performed as a series of 25 sequencial frequencies (standard) and one as a 5 part multiwave with 5 harmonic multipliers (total of 30 points). The two data sets show excellent agreement. The multiwave ramp was performed in 2/3 of the time of the standard test with 5 more data points.

Ideally, a controlled stress rheometer designed for multiwave oscillation should have:

(1) A broad torque range. As indicated previously for the resultant strains, the stresses (torques) in multiwave oscillation are additive, and since their sum cannot exceed the upper torque limit of the rheometer, the higher the rheometer's upper torque limit, the better. The CSL^2 Rheometer's drag cup motor has been specifically designed to address issues such as heat dissipation at higher torques. Hence, it is able to achieve the highest torque levels available in a commercial controlled stress rheometer (50 mNm).

(2) Adjustment of torque amplitude. Generally, G' (the material's elasticity) increases as oscillation frequency increases and this leads to decreasing strain amplitude for the same applied stress. Also inertial effects, both fluid and instrumental, reduce the strain amplitude as frequency increases. These decreases in strain amplitude need to be corrected in order to avoid poor results at higher frequencies. The CSL^2 allows the operator to choose an "enhancement factor" for each frequency being used ,which is then automatically applied by the rheometer during the experiment (by adjusting torque). This enhancement ensures that the strain amplitude and hence the quality of the data remains constant even at higher frequencies.

Figure 2. GENERATION OF A COMPLEX WAVEFORM

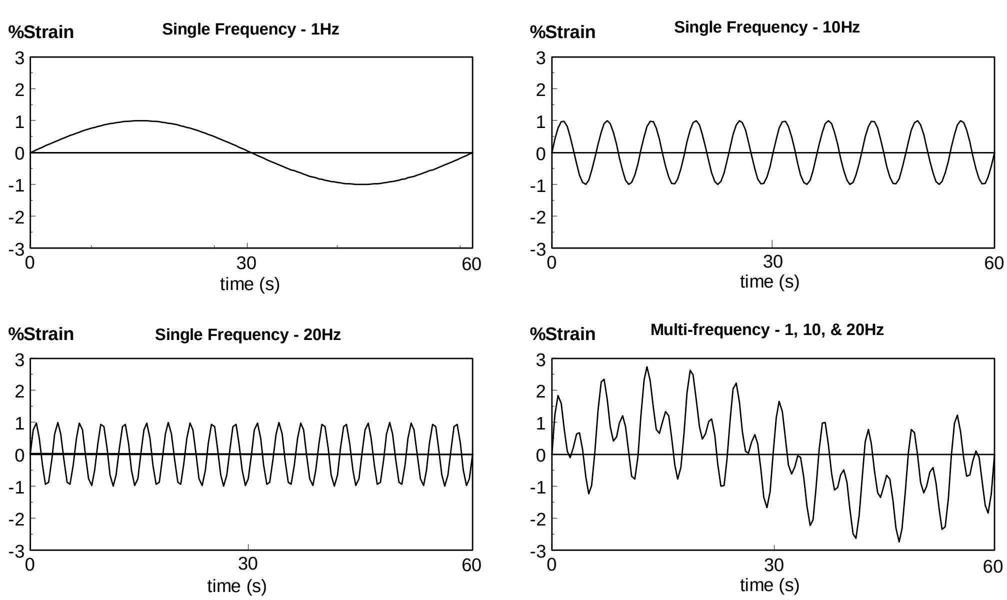
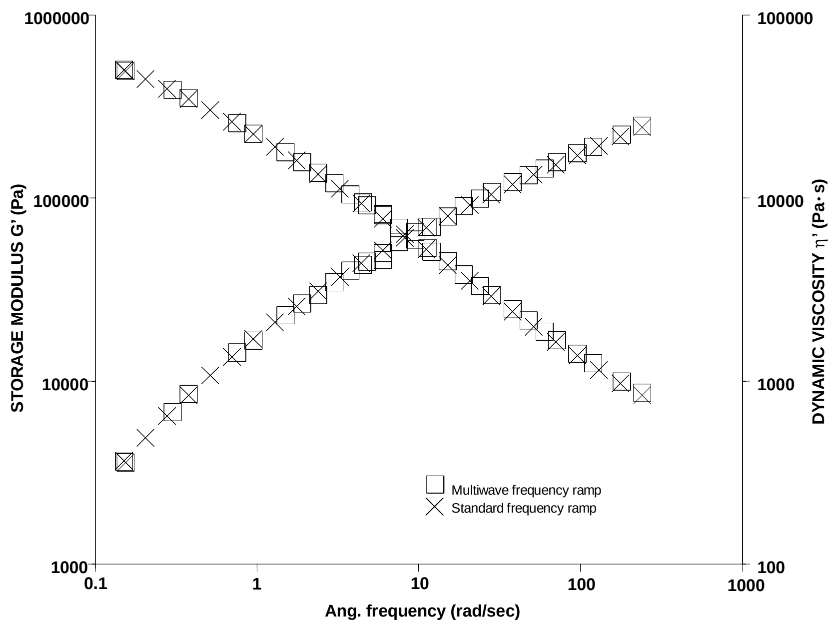


Figure 3. FREQUENCY RAMP COMPARISON



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