How to determine linear viscoelastic properties with optimal accuracy using higher harmonic analysis

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Abstract

In oscillatory shear, the upper limit of the Linear Viscoelastic Region (LVR) of a material is often defined as a critical shear stress and shear strain amplitude, at which a certain deviation of a viscoelastic property is measured compared to a reference (plateau) value. Furthermore, standard oscillatory shear experiments under variation of frequency or temperature require prediction of shear stress and shear strain amplitudes within the linear viscoelastic range of the sample, despite structural changes occurring during testing, such as curing. With the advantage of higher harmonics derived from the raw oscillatory signal, it is possible to determine the optimum shear stress and strain amplitudes that will provide the best signal-to-noise ratio at lowest structural decay of the sample.

Harmonic Distortion

Harmonic Distortion is an indication of the nonlinear response in the detected raw signal, which separates into contributions from instrument noise and sample structure.
Harmonic Distortion enables a generalized definition of the linear viscoelastic range of a sample, established by the viscoelastic plateau value.
By utilizing Harmonic Distortion analysis, the interpretation of test results in oscillatory shear on samples with time-dependent structural change can be improved.
The percentage of harmonic distortion (HD%) is automatically reported within the Live data during a measurement of the NETZSCH rSpace software.

Determination of Plateau-Modulus by Higher Harmonics

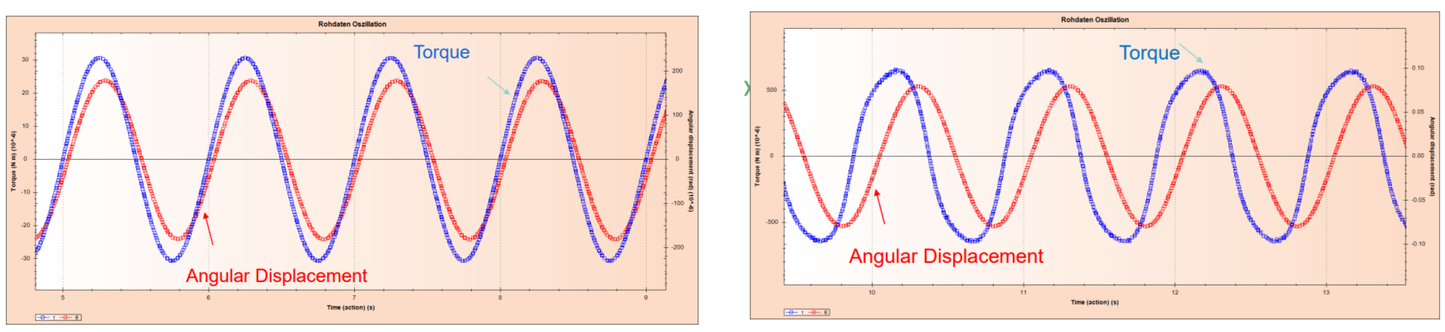

Figure 1: Comparison of raw data within (left) and outside (right) of the Linear Viscoelastic Region

Figure 1 shows excitation with sinusoidal shear wave leads to sinusoidal response within the LVR (left). The frequency of excitation is equal to the frequency of response signal. The right image shows a non-sinusoidal response outside the LVR. The response signal contains odd higher harmonic frequencies. For example, excitation with 1Hz, the response signal contains 3, 5, 7Hz…

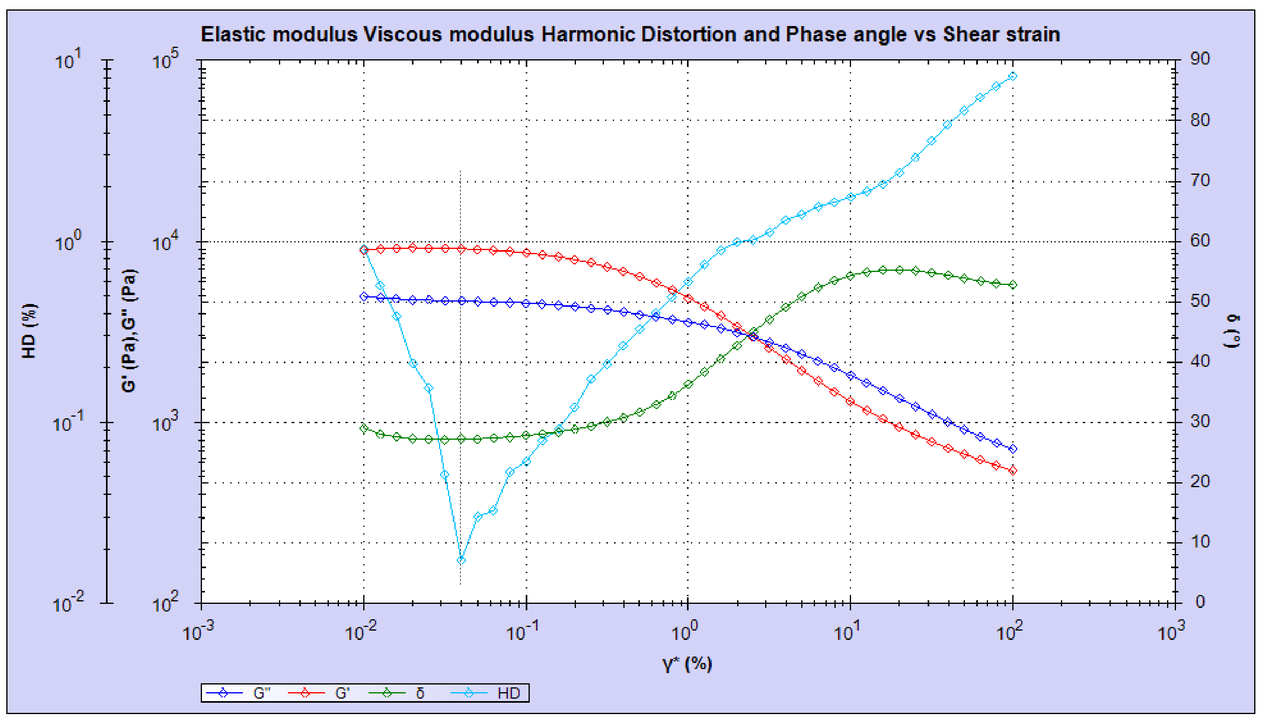

Figure 2: Elastic Shear Modulus G´, Viscous Shear Modulus G´´, Phase Angle δ and Harmonic Distortion HD as a function of Shear Strain Amplitude γ_0

Figure 2 shows the harmonic distortion, the percentage of all amplitudes of higher harmonics within the response signal compared to the amplitude of the base frequency (harmonic distortion of 0% would be perfect linearity). Determination of plateau modulus taken at minimum distortion: optimum signal-to-noise ratio at lowest structural decay.