FT-Rheology: a very sensitive experimental technique to characterize the non-linear regime in materials

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Rheology allows the characterization of different mechanical properties, of simple and complex topological structures, in melts or solution, both in the linear and non-linear regime. In general, processing and utilize conditions include high strain or high shear rates consequently lead to non-linear material response. This will effect dynamics, orientation and fatigue of the polymer materials and dispersions. Experiments in the non-linear regime are expected to be crucial with respect to the characterization and formation of interfaces, morphologies, different topologies and failure. Additionally quality control will also benefit from simple non-linear characterization techniques that allow a non-linear quantification..

In this lecture I would like to give first an overview with respect towards a very sensitive, but still simple, mechanical technique where large amplitude oscillatory shear (LAOS) using a frequency $\omega_1/2\pi$ is applied and the torque response is analyzed in frequency space. The non-linear response generates mechanical higher harmonics at $3\omega_1$, $5\omega_1$, $7\omega_1$ and so forth. A Fourier transformation can unravel the intensities and phases in a spectrum $I(\omega)$; consequently we use the term "FT-Rheology" for this technique. The experimental set-up is shown in Fig.1.

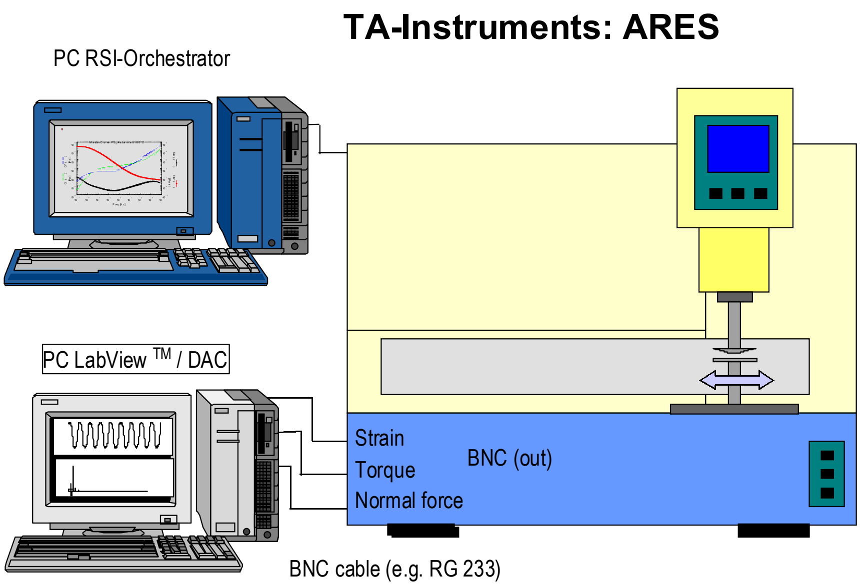

Figure 1 Experimental set up for FT-Rheology experiments. The strain, torque and normal forces are digitized via a ADC in a stand alone PC where the Fourier transform of e.g. the torque is conducted. The rheometer is controlled via the usual PC that allows to measure linear mechanical properties via the commercial software.

A typical FT-rheology spectrum is shown in Fig. 2, where a signal to noise ratio of 100,000 : 1 can be achieved.

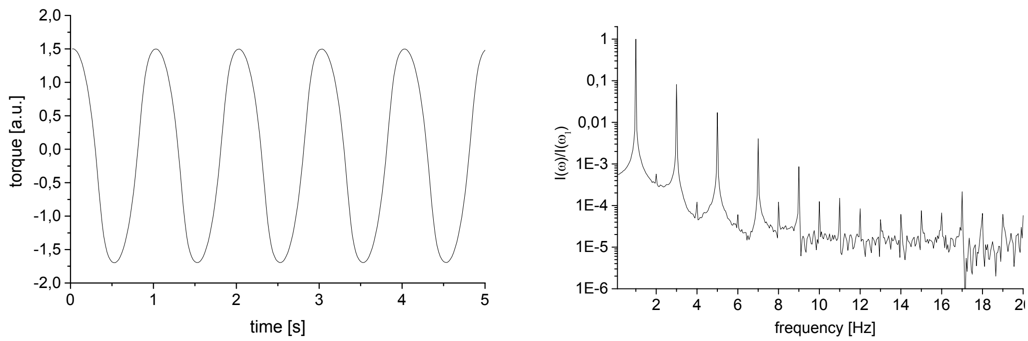

Figure 2 Time, respective frequency data set for polyvinylalcohol solution, at $T = 278°K$, $\gamma_0 = 20$, $\omega_1/2\pi = 1$ Hz for a single measurement. The S/N of the spectrum is in the range of 100,000 : 1.

Several application of FT-Rheology with respect to rubbers, dispersions and resins are presented. One emphasis in this talk is the correlation between topology and non-linear mechanical response in polymer systems. These systems are made of polystyrene: linear, 3 and 4 arm stars and serve as models for more complex systems like H, combs, and long chain branched structures. The result show a strong correlation between the phase of the third harmonic and the topology. Further more the correlation between the non-linear mechanical response and finite element simulations using the Polyflow package are presented. A second emphasis of this presentation is towards the influence of shear on the complex macroscopic morphology in a diblock copolymer system. The block copolymer was macroscopically first oriented and afterwards the shear conditions where changed so that the orientation tilted by 90 degrees. Using 2D-SAXS and FT-Rheology the kinetics of the reorientation was followed in detail.

Lit.:

[1] M. Wilhelm, P. Reinheimer, M. Ortseifer; High Sensitivity Fourier-Transform Rheology; *Rheol. Acta* **38** (1999) 349

[2] M. Langela, M. Wilhelm, U. Wiesner, H.W. Spiess; Microphase reorientation in block copolymer melts as detected via FT-Rheology and 2D-SAXS; *Macromolecules* **35** (2002) 3198

[3] M. Wilhelm; FT-Rheology (feature article); *Macromol. Mater. Eng.* **287** (2002) 83

[4] T. Neidhöfer, M. Wilhelm, B. Debbaut; Fourier-Transform rheology experiments and finite-element simulations on linear polystyrene solutions; *J. Rheol.* **47** (2003) 1351