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More Rheology – Less Effort

When Super Strength Meets Shear Flexibility

Process Optimization by Rheology

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Introduction

Nowadays, steadily growing energy prices for gas and electricity result in highly expensive polymeric raw materials and rising processing costs. Therefore, resource-, cost-, energy- and eco-efficiency is more important than ever. The aforementioned objectives cannot be achieved if the effectiveness of the manufacturing processes is not (re)considered, i.e., if the process is not optimized and tailored to the processed polymeric material or vice versa. Process optimization needs to start with understanding the flow behavior of the polymer employed. That's where rheology comes into play.

Rheology is the study of the deformation and flow of matter, unveiling the flow behavior of complex fluids such as polymers, food, slurries, suspensions and other compounds [1]. Rheometers such as rotational and/or high-pressure capillary rheometers contribute to better understanding the rheological properties of the materials being analyzed (Figure 1). However, in order to optimize the processing conditions, the materials must be investigated at comparably high shear rates and temperatures. Nevertheless, flow simulations may also substantially contribute to better manufacturing efficiency. Therefore, along with the rheological properties, also the parameters relevant to flow simulation shall be determined.

The Rosand high-pressure capillary rheometer line (Figure 2) allows for examination of the materials at process-relevant shear rate ranges up to extremely high shear rates (~10^8 s^{-1}). Besides the shear dependency of the viscosity (shear thinning, shear thickening, Newtonian behavior), the rheological behavior under extensional flow can also be

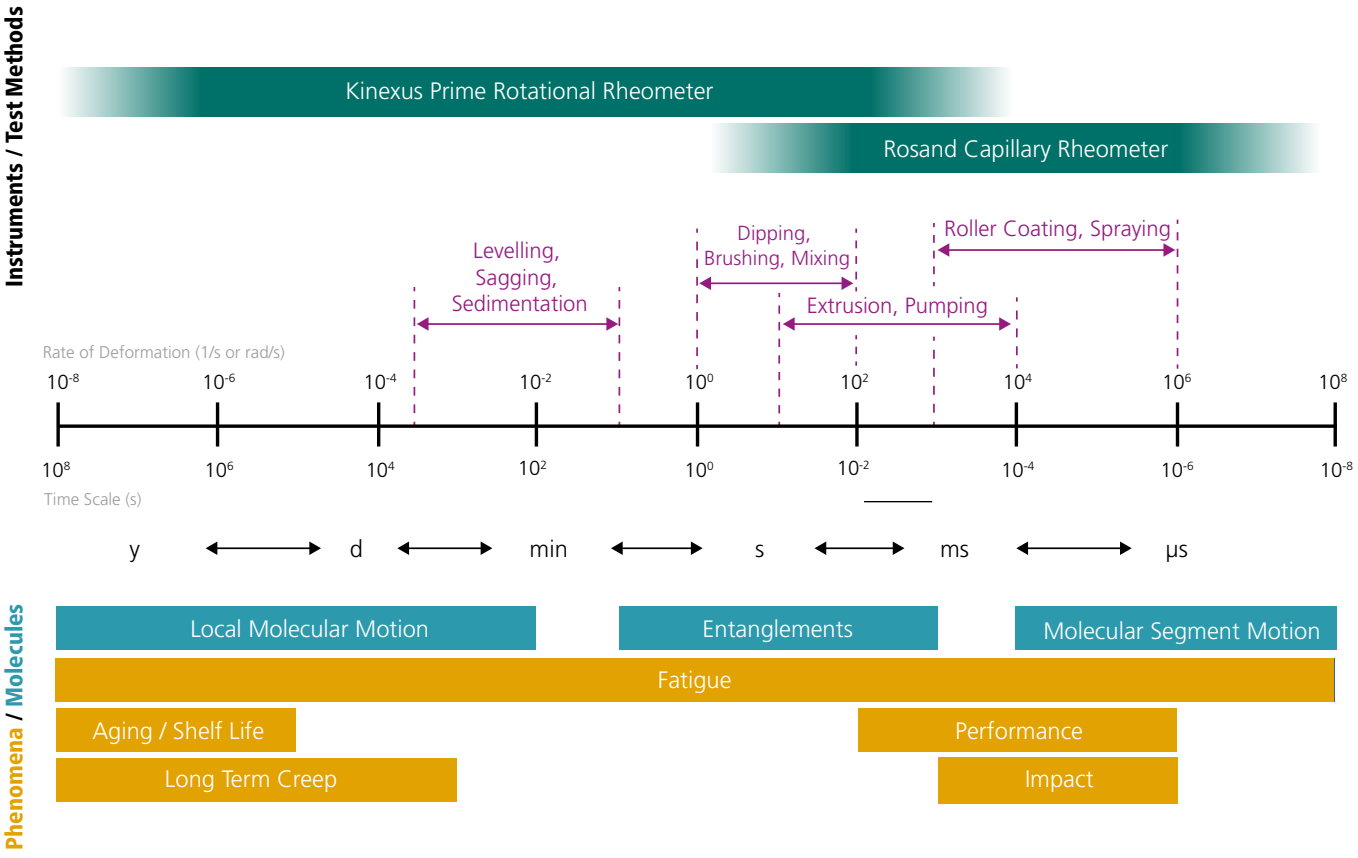

Fig. 1. Shear rate range covered by our rotational and capillary rheometers and its relevance in processing and understanding molecular dynamics

Rosand Capillary Rheometer

Shear thinning is a phenomenon occurring with some non-Newtonian fluids in which the fluid viscosity decreases with increasing shear stress.

Shear thickening is the opposite phenomenon.

In contrast with both of these, the viscosity of a **Newtonian fluid** is independent of the applied force (shear stress).

See for more definitions:
https://analyzing-testing.netzsch.com/en/training-know-how/glossary

analyzed. Moreover, by means of pVT measurements, simulation-relevant parameters such as pressure-dependent specific volume, crystallization or melting temperature are accessible as well.

In simple terms, the working principle of a high-pressure capillary rheometer for determining the shear viscosity of a material is that the material (molten polymer, liquid, paste, food, cream, etc.) be pressed with a well-controlled piston speed from a barrel through a capillary (die) with a well-defined diameter, length and entrance angle. The precise characteristics of the die, barrel and piston speed are necessary to later establish the shear stress (σ_{app}, see *Eq. 1*) and shear rate ($\dot{\gamma}_{app}$, see *Eq. 2*). Thus, the viscosity of the processed sample can be determined (η_{app}, see *Eq. 3*) [2].

$$\sigma_{app} = \frac{PR}{2L} \quad (1)$$

$$\dot{\gamma}_{app} = \frac{4Q}{\pi R^3} \quad (2)$$

$$\eta_{app} = \frac{\sigma_{app}}{\dot{\gamma}_{app}} \quad (3)$$

Where P is the pressure, R and L are the radius and length of the die, respectively, and Q is the volume flow rate.

Fig.2. Rosand RH7/10 Capillary Rheometer

pVT Measurement

In order to fully exploit the capabilities of a Rosand high-pressure capillary rheometer and contribute, by means of the data obtained, to process optimization, measurements must also be carried out along with shear and/or extensional measurements. In contrast with such shear and extensional measurements, the sample during a pVT measurement is enclosed in the barrel and the die is closed. This is necessary for investigating the change in sample volume as a function of pressure. Furthermore, first-order transitions can also be detected. The information obtained can be used in process simulation together with the shear viscosity measurement.

Rosand Capillary Rheometer

HDPE Under Pressure – Process Optimization

In the following, a Rosand RH7/10 high-pressure capillary rheometer with a maximum force of 100 kN was employed to unveil the pVT behavior of a high-density polyethylene (HDPE). The measurement was performed under isothermal conditions in the temperature range between 190°C and 40°C (cooled in steps of 10°C). The pressure range varied from 1 MPa (10 bar) up to 100 MPa (1000 bar). To increase the efficiency (less material and shorter measurement time), the Low-Level Script software option was used. Thus, multiple isotherm analysis sequences were created. For the duration of the experiment, in order to avoid any leakage, a PTFE seal was mounted on the piston end tip. Care must be taken to ensure that no air bubbles are formed during sample preparation (filling and compacting). Their presence and the resulting incorrectly determined weight lead to errors that strongly influence the quality of the measurement. Hence, not only can information regarding the pressure-dependent volume change be gathered, but it is also possible to measure the specific volume, density, and crystallization and melting temperature.

Precisely controlled piston movement (speed) compresses the HDPE at the desired pressure. A high-accuracy pressure melt-transducer measures the pressure (see Figure 3).

Knowing the applied pressure, the actual position of the piston, and the exact weight of the sample, the specific volume is calculated. Using third-party software, it is possible to plot a 3-D diagram (pressure, specific volume, temperature in Figure 4). This conveys further understanding of the material behavior (crystallization temperature, density) under

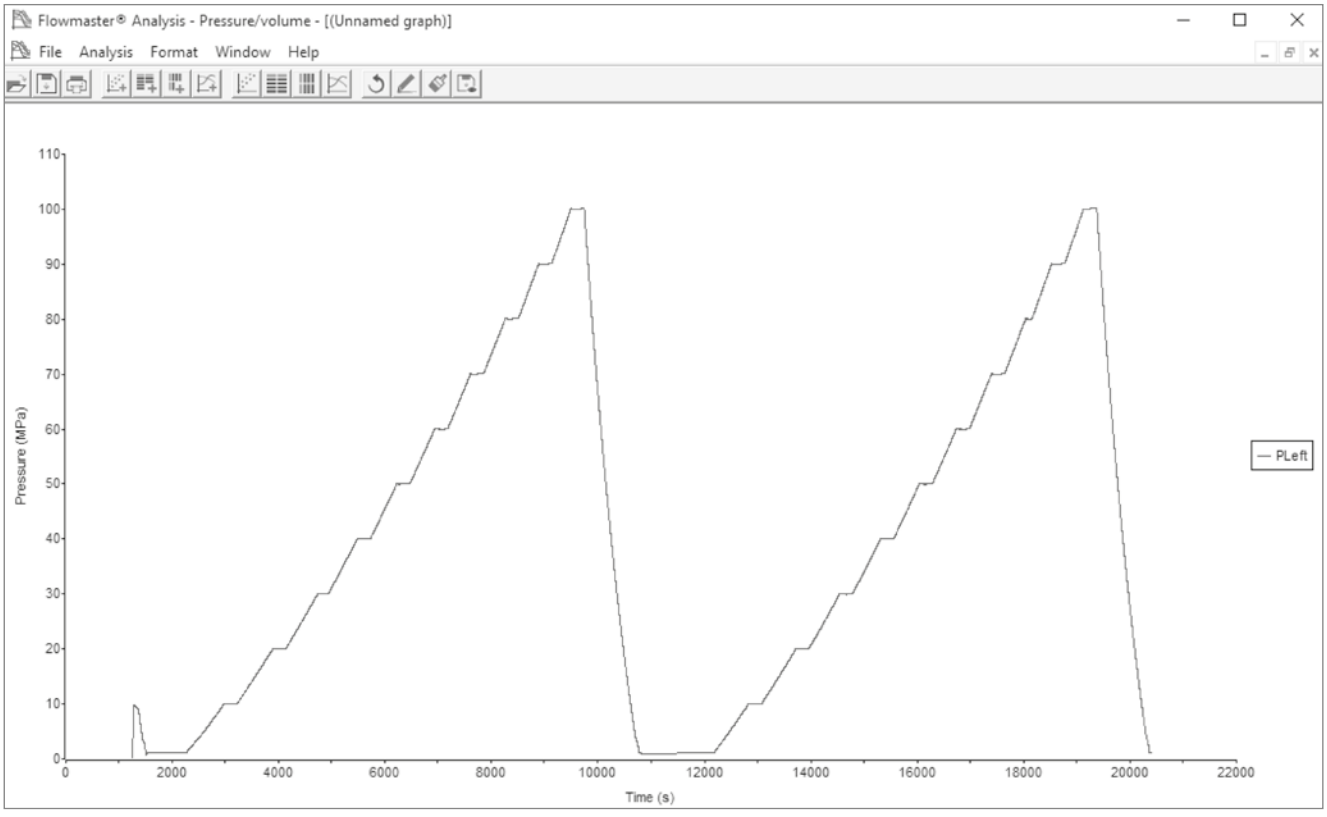

Fig. 3. Course of pressure as a function of time during a pVT measurement on HDPE

Rosand Capillary Rheometer

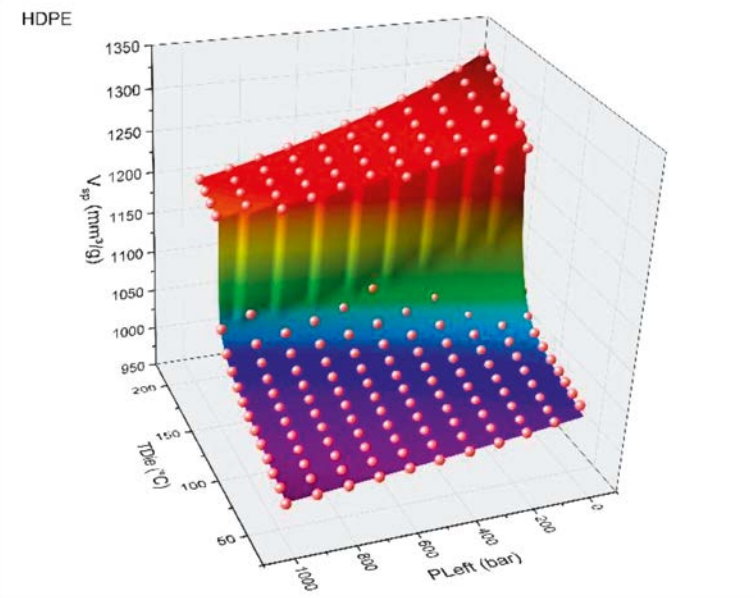

Fig. 4. Pressure-specific volume-temperature diagram of HDPE

high pressures and temperatures. Furthermore, processing-relevant parameters for flow simulation can be obtained by fitting the modified two-domain Tait [3] surface function to the data gathered from the pVT analysis. Later on, by combining the information originating from shear and pVT measurements, it is possible to significantly contribute to process optimization, thus achieving resource-, cost-, energy- and eco-efficiency objectives.

Summary

Investigation of the rheological properties of materials by means of a high-pressure capillary rheometer also allows, in addition to the determination of the shear rate dependence on the shear viscosity at extremely high shear rates (~10^8 s^{-1}), the determination of parameters relevant for processing and simulation. These parameters can be obtained, for example, by performing pVT measurements.

A Rosand high-pressure capillary rheometer offers insights toward better understanding of the material behavior. Such insights guide scientists in the creation of tailor-made, innovative materials and solutions. This later on significantly contributes toward optimizing industrial processes through increased efficiency and output, thus achieving multiple higher-tier objectives.

Literature

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[3] Wang, J. PVT Properties of Polymers for Injection Molding. In some critical issues for injection molding; Wang, J., Ed.; InTech: Rijeka, 2012.