New die design for a rapid rheological characterization of polymers

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Application Notes LR-67

Knowing the rheological properties of polymers in shear as well as in extensional flow are essential for their processing. However, their complete characterization in the laboratory requires the complementary use of sophisticated techniques which are time consuming and need great expertise. Therefore, quality control is mainly limited to the measurement of a melt flow index (MFI), a shear viscosity, and eventually a melt strength test to assess extensional properties. The on-line monitoring of material parameters would be very helpful but today is mostly restricted, whenever performed, to MFI or a capillary shear viscosity measurement.

The Thermo Scientific™ HAAKE™ X-die has been developed to cover the need for a quick characterization in shear and extension, with little or even no operator intervention. It can be mounted on a traditional torque rheometer or directly on-line with a melt pump feeding it. Data is obtained by this die with two batches of a standard PP (Polypropylen) with the same MFI but different performance in production by foaming the polymer. Oscillation tests with a rheometer correlate with this approach.

Industrial processes involving non-trivial polymer melt flows, such as injection moulding, extrusion, melt spinning and film blowing, a polymer melt is subjected to extensional deformation. This deformation makes predicting the polymer melt behavior difficult, potentially impacting the quality of the end product. The need for a fast and easy measurement of the extensional viscosity is therefore obvious. This die can be used off-line or on-line for process control determination of shear and extensional viscosity.

Experimental

Material

All measurements in this study were done with a done with two batches of commercial available PP. All experiments were conducted at 230 °C.

Methods

The measurements performed with the Thermo Scientific HAAKE X-die mounted on a Thermo Scientific™ HAAKE™ PolyLab™ OS torque rheometer with melt pump. For the oscillations tests a Thermo Scientific™ HAAKE™ RheoStress™ 6000 rheometer with 20 mm parallel plates was used.

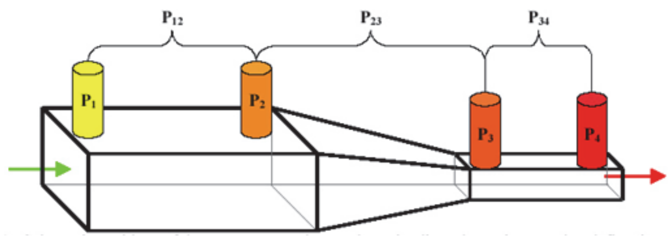

Fig. 1: Schematic drawing of the X-die

The HAAKE X-die consists of two slit dies linked by a defined contraction wedge, equipped with four pressure transducers located at the entrance and exit of each of the two slit dies sections (Fig. 1). The exact geometry of each part is defined so that the lubrication hypothesis can be applied (width at least ten times larger than the thickness). The contraction length and ratio have been chosen to enhance the extension and to minimize possible flow re-circulation. The two slit die geometries are such that the L/D ratio of both is the same. The change in cross-section results in a shear rate ratio of 1:64, and thus, in an expanded shear rate range.

The four pressure transducers (P1, P2, P3, P4) measure the pressure drop along the die. The volume flow is defined by the piston speed of the capillary rheometer or the r.p.m. of the melt pump when the die is mounted onto an extruder or by-pass rheometer. In the two slit sections of the die, the shear viscosity and the respective flow rates can be calculated using the well-known standard equations. With these two points of a flow curve, the power law coefficient **n** can be estimated for each flow rate. The calculation of the extensional viscosity is then performed in three steps:

1. The pressure drop along the wedge is determined from measured pressures P2 and P3 extrapolated to the exact entrance and exit of the wedge.
2. The calculation of the viscous pressure drop in the wedge due to shear flow is based on the standard equations using known geometrical parameters and the estimated power law coefficient **n**.

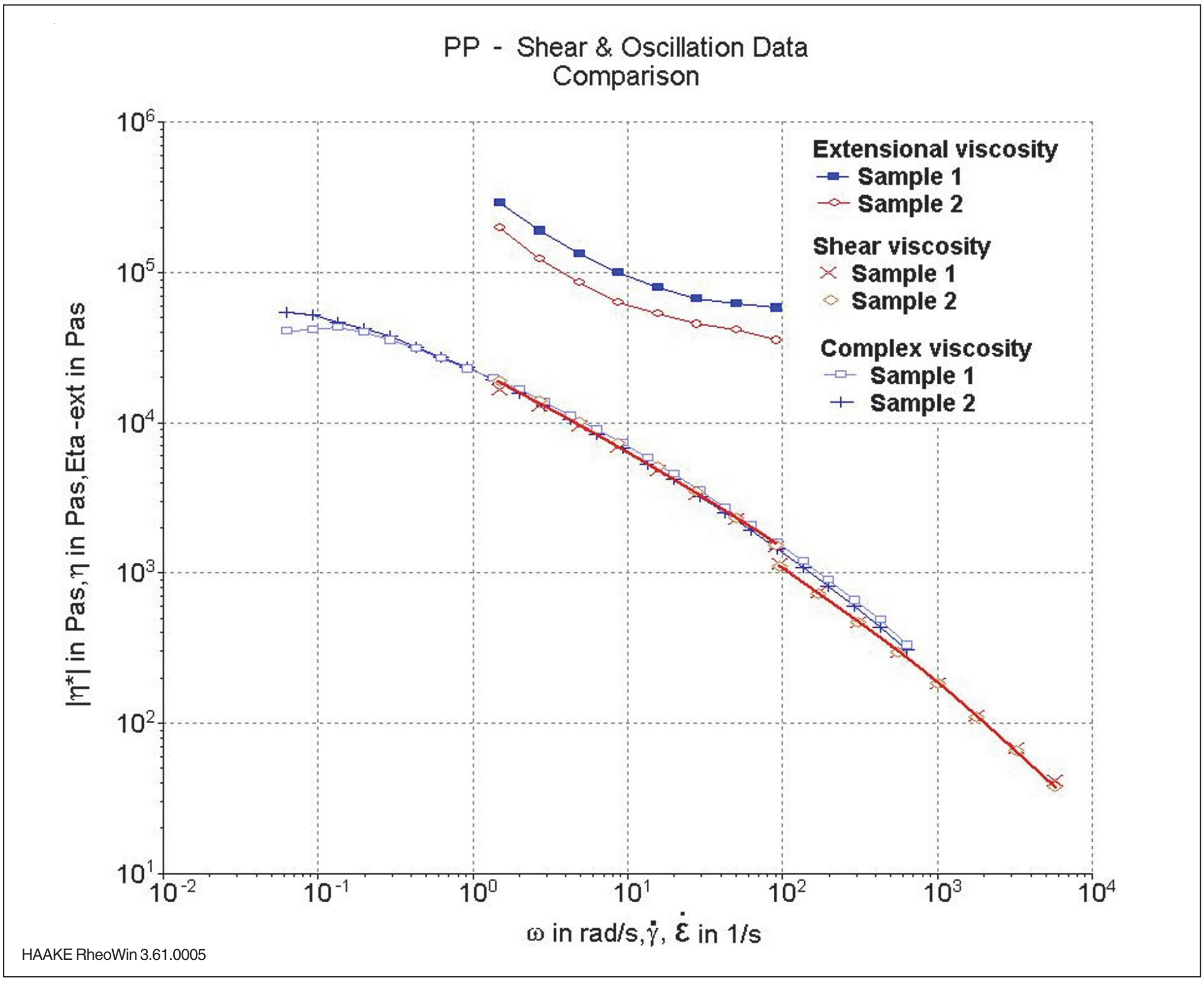

Fig. 2: Viscosity data in shear, elongation and oscillation

3. The difference between the total and viscous pressure drop is the pressure drop due to non-shear or extensional component. From this difference and a mean extensional rate in the wedge, the extensional viscosity is calculated.

Results and Discussion

Fig. 2 shows the results obtained done with the HAAKE X-die compared with the oscillation test with the HAAKE RheoStress 6000 rotational rheometer. With the HAAKE X-die a shear rate range from 10 to 10^4 s^{-1} can be covered, the complex viscosity data is identical and extend the shear rate range to 10^{-1} s^{-1}. The shear and complex viscosity data for both samples is identical. Only the extensional viscosity data shows a difference between the two samples and correlate with the performance in the foaming process.

Torque rheometer testing is similar to production conditions showing differences between the batches. Different samples can be tested in a reasonable time by purging. After the first encouraging results with a new type of extensional die presented earlier [1], this paper presents a comparison of batches to determine the extensional viscosity. For statistical control, further experiments have to be done with other types of polymers - however the results are a promising step towards the standard use of extensional data in polymer processing especially for on-line applications.

Reference

[1] G. Chaidron, J. Bouton, Study of extension and shear viscosities of polymer melts combining a specific contraction flow and an analytic numerical simulation, Proceedings of the 18th Processing Society, Guimarães, Portugal, 2002

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