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In-line monitoring flow in an extruder die by rheo-optics

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ABSTRACT

A new modular slit die with optical windows in two different positions and three pressure transducers flush-mounted along the wall was built and coupled to the exit of a twin-screw extruder. Thus, the birefringence and the pressure drop of polystyrene were monitored inline during extrusion. Two experimental procedures were tested: steady-state and cessation of extruder feeding. The latter proved to be very useful in the case of polystyrene since the ratio between the birefringence and the pressure drop can be quantified for a wide range of steady-state conditions with a single experiment. In fact, down to relatively lower values of pressure drop, the birefringence proved to be a function of shear stress at the wall only, depending neither on the initial feeding rate nor on the aspect ratio of the slit die, for W/h down to 5, at least.

1. Introduction

Understanding the flow character of polymers during processing is of extreme importance to control the final properties of the material. Most rheological analysis to assess the material functions, aiming at the prediction of polymer processability, has been undertaken from offline experiments, either by the use of rotational or capillary rheometers, at low and high shear stresses, respectively. Despite the indisputable usefulness of these bench techniques, the quest for in-process assessment of thermo-mechanical histories imposed on the material and by extension structural changes has been a concern.

Over recent years, the inline monitoring of polymer extrusion by attaching a slit channel to the extruder die exit has received considerable attention. A relatively large number of experimental techniques have been used to monitor polymers and polymer composites flowing through a slit die [1,2]. It is well-known that the installation of flush-mounted pressure gauges along the slit die wall allows the assessment to viscous features up to relatively high shear stresses with no need of Bagley corrections. It is well established in the literature that slit dies with aspect ratios higher than 10 (W/h > 10) fall within the concept of parallel plates of infinite width. Thus, the flow may be looked on as unidimensional and the edge corrections can be neglected. Thus, the wall shear stress is customarily obtained by the equation [3]

$$\sigma_w = \frac{h}{2(1+h/W)}\frac{dp}{dx} \tag{1}$$

where h and W are, respectively, the height and the width of the slit, and dp/dx is the gradient of the outward-acting total normal stress at the wall measured with pressure gauges in the fully developed flow region of slit-die geometries. The corresponding wall shear rate, $\dot{\gamma}_w$, is given by

$$\dot{\gamma}_w = \frac{\dot{\gamma}_a}{3}\left(2 + \frac{d\ln\dot{\gamma}_a}{d\ln\sigma_w}\right) \tag{2}$$

where $\dot{\gamma}_a$ is the apparent shear rate, which is expressed by

$$\dot{\gamma}_a = \frac{6Q}{Wh^2} \tag{3}$$

with Q being the volumetric flow rate.

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Under flow, most polymer melts become optically anisotropic, creating the possibility of using non-invasive optical techniques to understand the chain conformation and orientation during flow. Moreover, for homopolymers and a relatively broad range of experimental conditions, it is possible to establish a relation between the stresses in the material and the optical anisotropy as given by the refractive index tensor, **n**. Therefore, assuming that the principal axes of the stress tensor, $\boldsymbol{\sigma}$, and the refractive index tensor are collinear, the stress-optical rule may be written as [4,5]

$$\mathbf{n} - \langle n \rangle \mathbf{I} = C\{\boldsymbol{\sigma} + p\mathbf{I}\} \tag{4}$$

where C is the stress-optical coefficient, $\langle n \rangle$ is the mean refractive index, p is the hydrostatic pressure and $\mathbf{I}$ is the unit tensor. One of the most common ways to measure the birefringence of a polymer in shear flow is to send a light beam in the velocity gradient direction (y-direction). It should be noted, however, that the application of equation (4) is not straightforward since the velocity gradient direction is neither principal axes of the refractive index tensor nor principal axes of the stress tensor. Nevertheless, it is possible to show that, for small values of birefringence, the following expression is valid [6]

$$\Delta n_{13} = C[\sigma_{11} - \sigma_{33}] \tag{5}$$

Additionally, for flow in a slit die, the light traveling in y-direction passes layers of varying degrees of molecular orientation and, therefore, only an average birefringence, $\langle \Delta n_{13} \rangle$, is measured. However, Wales [7] was able to deduce a relation between the average birefringence and the birefringence at the wall, $\Delta n_{13,w}$,

$$\Delta n_{13,w} = \langle n_{13} \rangle \left[1 + \frac{d \log \langle n_{13} \rangle}{d \log \sigma_w} \right] \tag{6}$$

which is analogous to the Rabinovich correction [Equation (2)].

Early measurements of polymer flow birefringence in a slit die were carried out in the 1960s by Wales [7]. In his master thesis [8], under supervision of Janeschitz-Kriegl, he reported no influence of the aspect ratio (W/h) of the slit die on the birefringence for $W/h > 10$. The influence of the entrance was also studied by Wales. He observed that, for a low density polyethylene flowing through a slit die the birefringence, $\langle \Delta n_{13} \rangle$, decreases with increases distance to the entrance. Indeed, a steady flow condition, as characterized by a birefringence not dependent to the distance to the entrance, is not even reached for reduced distances (l/h, with l being the distance to slit entrance) as large as 100 and at low apparent shear rates. However, for polystyrene, Wales observed no variation of the birefringence along the slit channel even for a reduced distance of 15. Obviously, the different behavior of LDPE and PS could stem from the large discrepancy between the relaxation times of the polymers.

From equation (5), it follows that, if one disregards the difference between $\sigma_{11}-\sigma_{22}$ and $\sigma_{11}-\sigma_{33}$, the birefringence measured in 13 plane can be directly related to the first normal stress difference, N_1. This quantity is an indication of the elasticity of the material and, in general, relates to the shear stress by the following expression [9]

$$N_1 = A\sigma_{12}^{a} \tag{7}$$

in which A and a are constants. It is well-known that, in the limit of the linear viscoelastic range, the value of the slope a is 2. Since the zero-shear viscosity of monodisperse, linear polymers varies with the 3.4 or 3.5 power of the molecular weight, the zero shear-rate first normal stress difference is expected to increase with molecular weight to about the power 7. Measurements of the birefringence on slit dies have shown that the constant A increases with the molecular weight increase even at relatively high shear rates [8,10]. Regarding the influence of molecular weight distributions, it appears that broadening the distribution of a linear polymer, while keeping the average molecular weight and the shear stress unchanged, results in an increase in the normal stress difference but a decrease in the exponent a [9,11,12]. Furthermore, if Equation (7) holds, the first normal stress difference versus shear rate or shear stress must follow time–temperature superposition. That superposition was observed for PS in a slit die up to shear stresses of 10^5 Pa by using the birefringence as a measure of N_1 [13].

Several authors [14–24] have used the birefringence fringes to visualize the stress state in a slit die. In fact, by counting the birefringence fringes it is possible to spatially map the stresses along a slit die. This technique has been extensively employed in order to study the flow in the entrance and exit of slit dies. However, the quantification of birefringence by counting the fringes is not practical and, more important, it cannot be realized at high stresses. In addition to spatial visualization, other authors [25–27] have done a pointwise quantification of the birefringence by employing a laser as the light source and a detector.

In this paper we aim to show that the more traditional approach of controlling the extrusion [28] or injection molding [29] processes by inline monitoring the pressure drop in a slit die can be significantly improved if the birefringence is simultaneously measured. Here, a new version of the rheo-optical system developed in a previous work [10] is presented. The flow birefringence of polystyrene was measured. The three flush-mounted pressure transducers and two optical windows allow establish correlations between the pressure drop and birefringence in two different regions of the slit channel. With this data, it should be easier to infer about variations in the temperature due to viscous heating. Also, the "cessation of extruder feeding" approach is presented and its potentialities analyzed. In some situations, it can be more convenient to use rectangular ducts with W/h < 10 aspect ratio. It will be shown that the rheo-optical information obtained in those channels can be easily extrapolated for channels that meet the traditional "infinitely wide condition", W/h > 10.

2. Experimental

2.1. Materials

A commercial grade of polystyrene (PS), referred to as N2560, from Innova was used in this work. Throughout this paper a value of 0.94 g.cm^{-3} [30] will be considered for the