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A calorimetric capillary for the measurement of viscous heating in tube flow of molten polymers

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Abstract: The design of a calorimetric capillary for use with a plunger-driven capillary rheometer is discussed. With this equipment it is possible to measure, during extrusion, the local radial heat flow out of the liquid through the wall of the capillary. From this, temperature gradients in the liquid can be derived. Results compare well (in case of PS) or

reasonably well (in case of PE) with predictions from an analytical description of temperature distributions in power-law liquids as developed by the authors elsewhere. In the geometry used, the pressure dependence of viscosity and the decompression heating effect play an important role in the predictions. Melt flow anomalies (pulsated flow and melt fracture) are related to the measurements with the apparatus presented.

Key words: Calorimetric capillary, viscous heating, tube flow, polymer melt

1. Introduction

When a molten polymer is forced through a duct, as in extrusion or injection moulding, high energy dis-



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sipation is often unavoidable due to the high viscosity of the melt. The resulting increase in temperature of the plastic causes a reduction in the viscosity of the melt, leading to a change in the flow pattern and an increase of the throughput. On the other hand, the required high pressure (typically 10^8 Pa) causes an increase in the plastic viscosity. Additionally, at such high levels of pressure difference, changes in temperature due to decompression cooling (typically 10 K) cannot be neglected. The relative importance of these counteracting effects is not obvious.

In [1] an analytical description for the flow of power-law liquids (with arbitrary power-law exponent) incorporating these effects was developed by the authors. Most published studies on the theoretical analysis of this problem lack a direct experimental verification. An important reason apparently is the difficulty of measuring temperatures in polymer melt flow. Inserting a probe induces a flow barrier which often influences the pressure upstream and the temperature downstream of the probe. In this study we present an apparatus, called a calorimetric capillary, with which it is possible to obtain information about the liquid temperature without disturbing the liquid flow.

2. The calorimetric capillary

The calorimetric capillary is shown schematically in figure 1. A stainless steel tube *A* (inner/outer diameter is 1.6/1.8 mm) was soldered into two cylinders of red copper

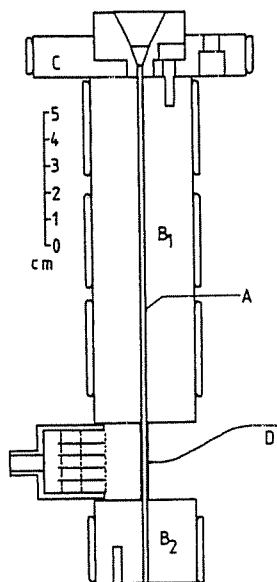


Fig. 1. Cross-sectional view of the calorimetric capillary

(B_1, B_2). Special care was taken to ensure optimal thermal and electrical contact between the steel and copper.

The capillary was used in the following way. After mounting it onto a plunger-driven extrusion rheometer using flange *C*, the capillary was controlled at a set temperature with electrical band heaters around the copper-walled sections and by heating the bare stainless steel section using a direct current flowing between both copper blocks. The temperature in this central, measuring section of the capillary was monitored with a miniature Chromel/Alumel thermocouple soldered onto the capillary.

When forcing a polymer melt through the capillary, viscous dissipation tends to heat the channel wall. In the copper-covered sections heating does not affect the wall temperature due to PID-thermocontrolled band heaters. In the measuring section the electrical current is reduced as much as is necessary to let the thermocouple regain its set-temperature. The reduction in electrical heating power in that section is exactly equal to the amount of radial heat flow in the melt at the channel wall. This is what we called the calorimetric principle [2]. Using accurate data for the heat conductivity of molten polymers [1], the radial temperature gradient in the plastic at the capillary wall is known. In this way substantial information on the temperature field in capillary flow is obtained without disturbing the flow.

By application of forced air cooling of the measuring section, the measuring range was extended drastically to higher heat flows through the wall, i.e. higher shear rates. Care was taken that the measuring section was isothermal within 1°C . The equipment was designed in such a way that it could be fitted to the rheometer with both ends of the capillary thus enabling measurements of radial heat fluxes both near the entrance and the exit.

3. Results and discussion

Figure 2 shows a typical result of the measurement of radial heat fluxes. The reduction in electrical heat-

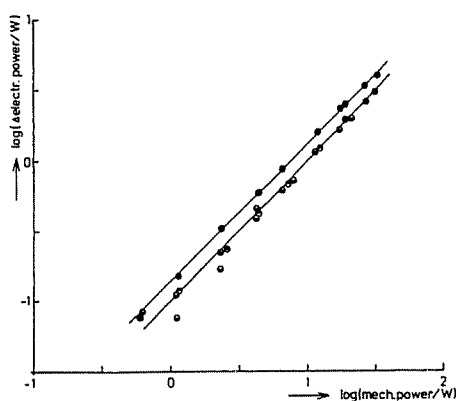


Fig. 2. Radial heat flux measurements with LDPE (Stamylan, ex DSM) at 192°C . The reduction of electrical heating power in the measuring section is plotted versus the mechanical power input in the capillary. Filled symbols refer to measurements near the entrance. Open symbols indicate measurements near the exit. Different symbols refer to measurements taken several months apart

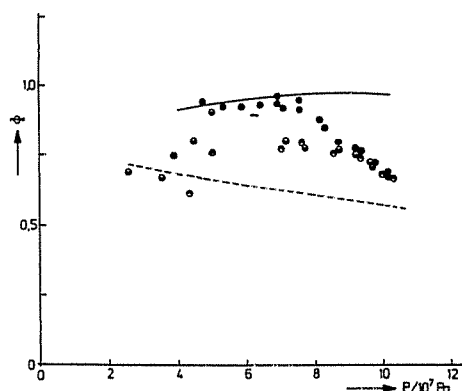


Fig. 3. Dimensionless radial heat flux for polystyrene (Hostyren N4000-01) at 192°C as a function of the pressure drop over the capillary. Filled symbols refer to measurements near the entrance. Open symbols indicate measurements near the exit; different symbols refer to measurements taken several months apart. Solid (dashed) lines are theoretical predictions for the location near the entrance (exit)

ing power in the measuring section is plotted as function of the mechanical power input (pressure drop times throughput rate) for a low density polyethylene. Over the two decades in power covered, straight lines are usually found. The filled symbols in the figure are measurements taken near the entrance of the capillary; the half-open circles refer to a location near the exit and show series taken several months apart for checking reproducibility. The main reason for the filled circles having the higher values is that the isotropic pressure and thus the viscosity, at a fixed shear rate, is higher near the entrance. In most of our results the pressure dependence of viscosity overrules its temperature dependence, due to the relatively large capillary used.

In figure 3 results, in this case for a polystyrene, are plotted as a function of the pressure drop along the capillary; values between 250 and 1000 bar were useful for the calorimetric technique. The vertical axis shows the radial heat flux through the channel wall in dimensionless form. A value $\phi = 1$ would occur if all the mechanical power were converted to heat and it were uniformly to flow out radially. Again filled circles refer to measurements near the entrance; semi-filled ones were measured near the exit. The lines are predictions from our theoretical model mentioned earlier, using the best data available for the relevant physical constants [1]. Due to compression cooling both lines coincide for $P = 0$ not at $\phi = 1$ but at $\phi = 0.8$. The experiments at the lower pressures are predicted well in the case of polystyrenes. With polyethylenes predictions of ϕ generally fall somewhat

below the experiments. We surmise that this is related to differences in the entrance flow pattern between PE and PS.

An interesting point is that with PS, beyond a pressure drop of 600 bar, flow instability occurred. At 600 bar (at an average shear rate of about $1.2 \cdot 10^5$ Pa) the instability was only detectable as very slight oscillations (± 0.03 K) in the signal of the thermoprobe attached to the measuring section.

At high pressures and throughputs these oscillations became more pronounced; oscillations also became detectable in the reservoir-pressure transducer signal and in the (visually observed) flow rate. During the periods of low flow rate the reservoir pressure increased and the probe temperature decreased, both almost linearly. During the periods of high flow rate temperature increase and pressure decrease were observed. Beyond 760 bar the dimensionless radial heat flux near the entrance decreased. Slightly above that pressure, melt fracture became visible as distortions in the extrudate shape. To our knowledge such flow rate oscillations around a "forbidden" rate have not been reported before for polystyrene.

We shall not discuss the many explanations in the extensive literature, see e.g. [3], on the subject of these flow anomalies. However, we note that, in our opinion, the influence which temperature oscillations in the molten plastic (due to flow rate variations) may have on flow instability, especially on the existence of forbidden flow rates, needs further analysis. Especially at the low power law exponents involved (in our case ca. 0.17) viscous heating is concentrated very locally thus promoting instabilities.

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