On-Line Rheometry of Polymer Melts: Steady Shear and Entry Pressure Measurements

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ABSTRACT

An novel on-line rheometer has been used to obtain flow characterisations of polymer melts on instrumented extrusion lines. Results are presented for LLDPE and polypropylene in shear flow and entry pressure measurements, and comparisons made with a standard laboratory capillary rheometer.

INTRODUCTION

On-line rheometers have been used for a number of years in process measurement of polymer extrusion, to give an indication of some property of the melt. This method gives a fast analysis of the melt propery with short delay from the process, but most process rheometers have been limited to a continuous shear viscosity measurement. The Rosand On-Line rheometer is designed to provide full melt characterisation over a range of processing rates in both shear and extension, in addition to the monitoring of shear viscosity. Research is being carried out to develop the instrument and to define it's capability in melt characterisation, and to study the effect the testing process has on the rheology of the melt.

RHEOMETER DESIGN

The prototype Rosand On-Line Rheometer is a computer controlled instrument which uses a precision gear pump to abstract melt from an extruder and force the melt through capillary dies at known flowrate, using a sensitive pressure transducer located above the capillary to determine melt pressure. Standard capillary rheometry theory is used to determine capillary wall shear rate and shear stress. The rheometer has the novel capability of automatic die changing during a test, allowing various capillary die geometries to be used, and hence offering entry pressure determination in addition to standard shear flow data. The dies are in the form of a 'cartridge block' which is moved (indexed) electro-pneumatically during an automated test. A schematic diagram of the equipment is shown in Fig.1.

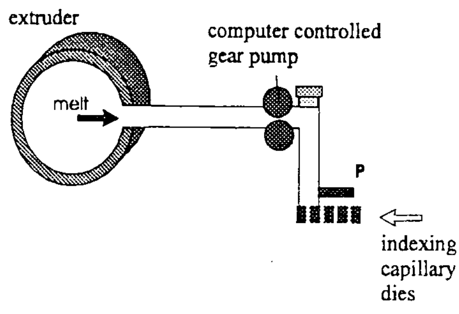

Figure 1. Schematic Diagram of the Rosand On-Line Rheometer

Shear flow evaluation by the rheometer can be undertaken using a single capillary die, by stepping the gear pump in programmable manner to obtain a range of shear rates. This is similar to other on-line rheometers, but incorporates the Rosand algorithm for automatically detecting when steady state pressure has been reached. Entry pressure can be measured using 'zero length' orifice dies and from this Bagley corrections may be applied to the shear data and extensional and elastic properties examined. Capillary wall shear rates of over $5000s^{-1}$ are achievable with 1mm bore capillaries. The flexibility of the die block system allows any combination of long and short die pairs, and dies of differing capillary diameter. This in turn allows various studies to be carried out for example the investigation of wall slip velocity.

EXPERIMENTATION

The Rheometer was connected to a Betol BTS40 twin screw extruder with screw diameter 38mm. The extrusion line is fully instrumented allowing melt temperature and pressure to be monitored up to a frequency of 1000Hz, and in-process rheological measurements are possible using an in-line rheological slit die. For comparison purposes, a standard off-line laboratory capillary rheometer was used - a Rosand RH7 twin bore instrument.

Two dies were used during the on-line rheometer tests, a 16x1 mm bore long die, and a 0.25x1 mm short die, whose L:D ratio is sufficiently small for it to be treated as an orifice die[1] . A Dynisco 5000 p.s.i. pressure transducer was used for melt pressure drop measurement, although the rheometer is designed to allow transducers to be readily changed so an appropriately sensor can be selected to suit test conditions.

Tests were set using Rosand software running on a dedicated PC. Shear rate stages are pre-set and the digital encoder controlled gear pump is run to give the desired melt output rate. The test is first run for the long die before the block is automatically indexed and the test repeated for the orifice die. Experimental results are calculated automatically by the test software and can be examined in tabular or graphical form. Two materials have been used in the tests - a linear low density polyethylene supplied by BP Chemicals and a polypropylene supplied by Shell. Shear and entry flow data was then compared with results from the off-line RH7 rheometer, in which flow is generated by pistons.

RESULTS

Experimental results were plotted in the form of shear stress (Bagley corrected) versus apparent wall shear rate, and entry pressure versus apparent wall shear rate. Graphs of results for LLDPE are shown in Figures 2 and 3.

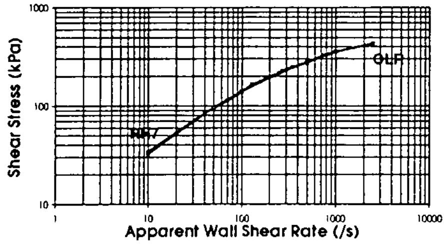

Figure 2. Shear stress-shear rate data for LLDPE at 200°C: OLR and RH7

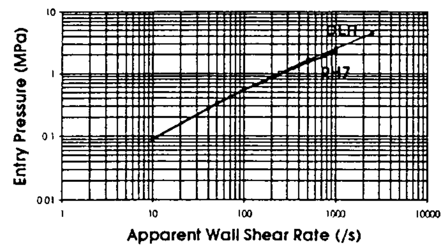

Figure 3. Entry Pressure data for LLDPE at 200°C: OLR and RH7

Figure 2 indicates excellent agreement between on-line and off-line measurements (within 5% maximum deviation). Figure 3 also shows very good agreement (within 10% maximum deviation), although entry pressure drops measured on the on-line rheometer are slightly higher than those from the RH7 at higher shear rates. This may be attributed to increased energy input into the melt by the gear pump at these higher rates.

PRESSURE FLUCTUATION

In addition to the standard rheological tests, studies have been made into the typical variation in melt pressure in flow generated by the on-line rheometer gear pump compared to in-process flow (measured using in-line rheological slit dies[2]) and the laboratory rheometer. Such measurement of statistical variation provides an assessment of accuracy of measurement with respect to process noise.

Both rheometers and the extrusion line were fully computer monitored, so pressure signals could be logged at various frequencies and variation measured. The results of these experiments are summarised below in Table 1.

Instrument	Coefficient of Variation (%)
On-Line Rheometer	1.92
RH7 Rheometer	0.58
In-Process Die	3.36

Table 1. Pressure fluctuation data for on-line rheometer, RH7 and in-line die

As expected, the in-line die pressures showed greatest variation due to screw rotation effects; the on-line rheometer pressures showed a small variation, reflecting a small effect of gear pump rotation, but a significant dampening of process noise.

To further examine the fluctuation in the on-line rheometer melt flow, Fourier analysis has been carried out on pressures logged at high frequencies. Melt flows at different gear pump output rates were analysed in an attempt to find out whether the action of the pump had an effect on the stability of flow. Using Fast Fourier Transforms, it was possible to see if process noise occurred in a cyclic nature, and whether the frequency of the cycles could be correlated to the gear pump speed and/or meshing of individual gear teeth.

Results showed that the pressure fluctuation did appear to occur in cycles and that the frequency of these suggested that the pump speed was a contributing factor. However, the magnitude of the process variation was relatively small and it is thought that the slight process noise will have negligible effect on the final rheological results.

CONCLUSION

These studies have provided a good indication of the usefulness of the Rosand On-Line Rheometer in carrying out both shear and extensional melt characterisations on-line to the extrusion process, with excellent correlation being found with an off-line rheometer. This, and the flexibility which the rheometer tests permit, gives the potential for obtaining a depth of melt characterisation previously limited only to off-line instruments.

Pressure fluctuation in the on-line rheometer melt flow was slightly higher then that of the laboratory rheometer, and was attributed to the action of the gear pump, although the magnitude of variation was not judged to be significant in determining the final rheological melt properties.

REFERENCES

1. Cogswell, F.N. (1981) Polymer Melt Rheology: A Guide for Industrial Practice, Godwin, London, pp 24-31.

2. Coates, P.D., Rose, R.M. and Wilkinson, B., (1989), Proc. 3rd Int Conf Polym Proc Machinery, **13**, 1-9