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The gas-assisted extrusion of molten polyethylene

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Synopsis

This paper reports for the first time our preliminary findings of a new gas-assisted extrusion process. We have discovered that if gas is injected at a metal die/molten polymer interface at a low flow rate, it is possible to establish a stable gas layer at the interface, which can give rise to an essentially full slip wall boundary condition. We report experimental optical observations, flow birefringence data, pressure difference, and die swell data for both a slit and rod geometry extrusion. We also match some of the experimental results with a viscoelastic numerical simulation. The introduction of wall slip induced by the presence of the gas layer has a profound effect on the magnitude of the die swell observed for polyethylene processed using gas-assisted extrusion. The experiments demonstrate, without ambiguity, that wall boundary conditions can play a crucial role in the overall extrusion flow of high viscosity viscoelastic fluids, such as polyethylene. © 2001 The Society of Rheology. [DOI: 10.1122/1.1332786]

I. INTRODUCTION

The rheology and processing behavior of molten polymers, and polyethylene in particular, has been studied extensively for many decades. Sophisticated constitutive equations such as the Wagner integral equation [Wagner (1976)] and more recently, the McLeish–Larson (1998) Pom Pom model have been developed to capture the complex rheological behavior of polymers such as polyethylene. Recently reported work for example by Bishko et al. (1999) show that both extensional and shear rheological responses can be matched for different polymers using data from the materials linear viscoelastic spectra and with the appropriate choice of nonlinear parameters.

Recent advances in numerical computation [see for example Crochet and Walters (1993), and Baaijens (1998)] has also meant that it is now possible to simulate the flow behavior of these rheologically complex fluids within relatively complex flow geometries such as capillaries and slits. For most polymers under mild processing conditions the no slip wall boundary condition has proved adequate for an impressive fit to be found between numerically predicted stress fields and experimentally obtained flow birefringence stress fields [see for example Ahmed et al. (1995)].

There is, however, now a growing body of evidence to support the view that some polymers can slip at the wall under certain processing conditions. The idea of slip is of course not a new one and can be traced back to the classic work of Mooney (1931). There is now extensive literature on wall slip in relation to the effect itself [see for example...]

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Vinogradov and Ivanova (1967), Piau et al. (1994 and 1995), Hazikiriakos and Dealy (1991 and 1992), Brochard and de Gennes (1992), and Person and Denn (1997). In addition wall slip has also been related to die swell effects [see for example Kalika and Denn (1987) and extrusion surface instabilities (see for example Wang and Drda (1997)].

Processing aids [Chan and Feng (1997)] and liquid lubricated dies [Pendse and Collier (1996)] have also been used as a way of enhancing the processibility of certain polymers. Up to this point, it does not, however, appear that gas lubrication has been used for enhanced polymer extrusion, although gas injection is a well developed technology in the field of injection moulding where in particular hollow mouldings are required [Liang (1996) and Potente and Hansen (1993)]. In this paper we report for the first time experiments and some matching simulations where we form a stable gas layer at the polymer/wall interface during extrusion flow.

II. EXPERIMENTAL MATERIAL AND APPARATUS

A. Materials

The molten polymer extruded was a commercial extrusion grade polyethylene HDPE Rigidex 5502XA supplied by BP Chemicals. This is a medium molecular mass polymer with a melt flow index of 0.2 g/10 min and a density of 954 kg/m³. It has good mechanical properties such as rigidity, impact strength, and environmental stress cracking resistance for applications of containers, sheet, and pipes.

The material was rheologically characterized in terms of an integral equation Wagner model in the way reported in previous papers [Mackley et al. (1994)]. The rheological measurements were carried out on a Rheometrics RDSII rheometer with a pair of Φ25 mm parallel plates at a gap of 1 mm and a temperature of 180 °C. The material parameters determined in simple shear were described with a group of relaxation time spectrum \((λ_i, g_i)\) and a damping function coefficient \(k\). Simple shear \((λ_i, g_i)\) and \(k\) for HDPE Rigidex at 180 °C are listed in Table I.

<table>
<thead>
<tr>
<th>(λ_i(s))</th>
<th>(g_i(Pa))</th>
</tr>
</thead>
<tbody>
<tr>
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<td>227690</td>
</tr>
<tr>
<td>0.009868</td>
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<tr>
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<td>40494</td>
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<td>17276</td>
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<tr>
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<td>2138.7</td>
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<tr>
<td>28.295</td>
<td>537.61</td>
</tr>
<tr>
<td>155.66</td>
<td>831.28</td>
</tr>
</tbody>
</table>

\[ k = 0.26±0.01 \]

B. Experimental apparatus

A Betol extruder with a screw of diameter 18 mm, L/D ratio 20, and compression ratio 2:1 was used and the polymer melt flow rate was stabilized by a melt gear pump. The melt flowed through the extruder barrel, to the melt pump, followed by a connection arm, and finally into and out of an interchangeable flow cell. The temperature settings of the extruder barrel from the feed section to the melt pump inlet were 160, 175, and 180 °C.
Five additional band heaters were used to heat the melt pump, connection arms, and the flow cell. These band heaters were set in order to maintain the flow cell tested at a uniform temperature of 180 °C. The extrusion pressure was measured from a pressure transducer installed on the flow cell. The melt flow rate was controlled by choosing appropriate melt gear pump speed ranging from 0.1 to 1.0 rpm. The corresponding melt mass flow rate was obtained by weighing the extrudate within a certain period of time at a given melt pump speed, giving a range from 0.034 to 0.21 g/s.

The flow cell was specially designed to enable gas to be injected into the melt flow stream. Nitrogen was used as an inert gas in the experiment. The nitrogen from a storage cylinder flowed first through a pressure reduction valve to reduce the pressure from about 200 to 40 bar and then through three flow rate control valves and another pressure control valve to achieve a consistent gas supply of pressure about 5 bar. The gas was introduced into a chamber inside the flow cell, where the gas was heated up and injected into the extrusion die slot in the inner wall of the die with a width of 100 μm. The injected gas flowed downstream together with the melt stream and out of the die. A schematic diagram of the experimental set-up for the gas-assisted extrusion is shown in Fig. 1. Different gas flow rates were tested. It was found that the gas should be injected at such a flow rate that the injected gas would not interfere with the flowing melt and a stable laminar gas layer was developed at the die land/polymer melt interface.

Two flow cells were designed, manufactured, and used. One flow cell was inserted with a pair of slit dies. Figure 2 shows the schematic diagram of the slit die and the gas injection position. The melt from the melt gear pump enters the upstream part of the die and flows through and out of the die. The extrudate swells after flowing out of the slit and emerging into free space. The slit die has an upstream width 15 mm, slit gap 3.5 mm, slit length 14.5 mm, and a depth of 15 mm. The gas was injected into the slit from a slot of 0.1 mm at a position of 7.5 mm from the slit entrance, or 7 mm from the slit exit. The gap

![Schematic diagram of the experimental setup for the gas assisted extrusion showing extruder, flow cell, and gas system.](image)
of the lower part slit where gas injection occurs was extended to 3.7 mm. This 0.1 mm step was designed to assist the development of the gas layer at the interface and reduce the influence of the injected gas on the flowing melt. The gas injection slit was across the slit depth and thus the injected gas flows only along the two sidewalls. The slit dies were constrained between two glass windows in the direction of the depth so that the injected gas flow could be observed. In order to measure the die swell ratio of the extrudate, the glass windows were cut at 2.5 mm downstream from the slit exit and the die swell ratio for the slit die was conveniently measured 2 mm downstream from the slit exit. In addition, rheo-optic measurements of the flow field within the glass window were carried out with this flow cell. A light of single wavelength 546.1 nm was used to enable flow birefringence observation. The principal stress difference (PSD) distribution was determined from flow birefringence in terms of the stress optical rule using a stress optic coefficient $1.839 \times 10^{-9} \text{ m}^2 \text{N}^{-1}$, giving a PSD increment for each fringe of $1.98 \times 10^4 \text{ Pa}$.

A second flow cell was a rod die assembly and the sectional view of the rod die extrusion flow channel is shown in Fig. 3. The rod die channel consists of separate top die and bottom die. The top die has a length of 77.4 mm and a diameter of 9.5 mm while the

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**FIG. 2.** Schematic diagram of the slit die and the gas injection position. All dimensions are in millimeters. Diagram not to scale.
bottom die has a length of 25 mm and a diameter of 9.7 mm. There is the gap of 0.1 mm between the top die and the bottom die, forming a circular slot where the gas is injected into the die. The melt flows along the die and then out of the die into the free space. The gas flows along the circular surface of the bottom die and then out of the die into the atmosphere. The die swell ratio for the rod die was taken from photographs at 20 mm downstream from the die exit. The gas enters both the slit and rod cell at ambient temperature; however, the gas will be heated within the flow cell. At present, we do not have precise information on the gas temperature at the injection point into the polymer, however, we believe the temperature to be much nearer that of the melt than ambient temperature.

III. SLIT DIE EXTRUSION

A. Optical observation of gas evolution

The transparent optical characteristics of the molten polyethylene and the design of the flow cell offered an excellent opportunity to observe the gas injection into the die and the
possible interaction of the injected gas with the flowing melt. Figure 4 shows the visual formation of a gas layer at the polymer melt/the metal die interface. The melt is flowing from the top to bottom through the slit gap and then out of the die. The melt is transparent but strong optical contrast occurs at the polymer gas interface due to the large refractive index difference between the polyethylene and the nitrogen. It can be seen from Fig. 4 that a gas layer develops after gas injection begins [Fig. 4(a)]; the gas layer initially forms a tongue shape within the melt [Fig. 4(b)] and then the gas layer folds back towards the die surface [Fig. 4(c)]. Subsequently a stable, uniform gas layer is formed between the metal die surface and the flowing melt [Fig. 4(d)].

Figure 4 demonstrates that stable gas injection into the flowing melt stream is possible and the gas injection occurs uniformly across the injection slot. The injected gas flows
downstream adjacent to the wall and then out of the die. In order for this to occur the gas pressure needs to be adjusted to an appropriate level. The gas can initially only be injected into the die when the gas pressure is higher than the melt pressure at the gas injection point. However, if the gas pressure is much higher than the melt pressure at the gas injection point, the gas can penetrate into the upstream melt and interfere with the melt flow, causing an unsteady melt extrusion. Experimental observation shows that the gas can successfully be injected at the interface and form a gas layer perfectly situated at the interface.

Observation of the removal of gas pressure is also instructive and Fig. 5 contains a group of pictures showing a surface wetting process of the slit die wall on cessation of gas injection.

FIG. 5. Surface wetting process of the slit die wall on cessation of gas injection. (a) Gas layer on the surface with gas injection; (b) the melt has partially wet the surface instantly after stopping gas injection; (c) the melt has mostly wet the surface after stopping gas injection; and (d) the melt has fully wet the surface.
gas injection. Figure 5(a) shows an existing gas layer on the metal die surface before the gas injection is stopped. After switching off the gas supply, the residual gas gradually disappears and the surface is partially wetted by the melt [Fig. 5(b)]. The residual gas further escapes to become less and less and the surface is mostly wetted by the melt [Fig. 5(c)]. Finally, the metal die surface is completely wetted by the melt [Fig. 5(d)]. The process takes only a few seconds.

B. Flow birefringence

The technique of flow birefringence has been used to demonstrate the way in which the stress field within the flowing polyethylene melt has been modified by the presence of a gas layer. The experimental techniques were those previously described in Ahmed et al. (1995). Figure 6 shows three flow birefringence stress fields relating to 6(a), upstream, no gas, 6(b) downstream, no gas, 6(c), downstream, with gas. Figures 6(a) and 6(b) show the classic stress patterns for molten polyethylene flowing within a slit. Stress buildup in the entry region is observed and the development of shear stresses near the wall can be clearly identified. The fact that the fringes are essentially parallel to the slit wall within the slit indicates that the shear stress along the wall is constant. At the exit, stress concentration at and near the surface can also be seen. The gas inlet step can be identified in Fig. 6(b) approximately half way down the photograph. At this point, a near wall fringe cusps into the surface. A key feature of Fig. 6(b) is that the wall shear stress is maintained essentially up to the exit of the die.

When gas is applied, the entrance upstream flow birefringence pattern was observed to be essentially unchanged, however, downstream of the gas inlet, major differences could be identified. Figure 6(c) shows the situation for gas flow and it can be immediately seen that stress relaxation near the walls rapidly occurs. By the time the polymer reaches the exit, most but not all of the stress (fringes) have disappeared. The gas layer is apparently acting as a very low viscosity lubricating layer and the flow in the slit is reverting to essentially plug flow.

In order to test the assumption of full lubrication at the wall, a Polyflow simulation was carried out to match the experimental observations. Using methods described previously in Ahmed et al. (1995) a two-dimensional (2D) Polyflow numerical simulation was carried out and preliminary results are shown in Fig. 7. Simulations were carried out with a sufficiently fine mesh to obtain mesh invariant results. 2D simulation was only available for the type of integral equation used and previous work by us Ahmed et al. (1995) has shown that for the geometry used here this is not an unreasonable approximation. In Fig. 7(a) the normal no slip boundary conditions are solved for a Wagner type integral constitutive equation using the rheological parameters given in Table I. The small step was not included in the simulation and the overall matching of simulated stress field to experimental result was reasonable. In Fig. 7(b) the same simulation was carried out but allowing a full slip boundary condition below the position of the gas inlet. The full slip condition thereby mimicked the presence of the gas, although the geometry step and the finite gas layer were not taken into account. The modification in the simulated stress field, whilst not exactly matching what was observed experimentally, captures most of what occurs. The wall stress level rapidly decays within the slit and the level of numerically predicted die swell decreases in line with experimental observation.

From these experimental observations and supporting numerical simulations we conclude that the gas layer acts as a “near perfect lubricating boundary layer.”
C. Die swell

Experimental die swell ratio data obtained from the 2D slit die are listed in Table II. The melt flow rates tested were read from the melt pump speedometer. The corresponding melt mass flow rate and wall shear rates are also listed for reference. The die swell ratio is taken at 2 mm downstream from the slit exit. It can be seen from Table II that the die swell ratio slightly increases with the increasing melt flow rate in both cases without gas injection and with gas injection. However, the die swell ratio is significantly reduced by gas injection.

The numerically predicted die swell ratios can be obtained from Fig. 7, giving a die swell ratio of 1.45 for the case without gas injection and a ratio 1.31 for the case with gas injection.
injection. These compare to the experimental data at the same melt flow rate with 1.53 and 1.38, respectively.

D. Extrusion pressure drop

Extrusion pressure measurements were taken from a pressure transducer positioned above the entry of the slit die as shown schematically in Fig. 1 and the extrusion pressure is plotted, in Fig. 8, as a function of melt flow rate for both with and without gas injection. In both cases the extrusion pressure increases with the melt flow rate, however, the normal extrusion has a significantly higher pressure drop than the gas assisted extrusion. There is an average reduction of 37.2% in the pressure drop between data of the normal extrusion and those of the gas assisted extrusion at different melt flow rates.

The reduction in extrusion pressure drop with gas injection is significant and it provides further evidence for the lubricating effect of the gas flow. The pressure drop with the gas assisted extrusion is reduced by a factor nearly equal to that anticipated by removing the part of the die downstream of the gas inlet and this offers further support to the belief that the gas layer is providing a full slip condition in this part of the die.

![FIG. 7. Numerical simulation of principal stress difference distribution of HDPE at a melt flow rate of 1.0 rpm from slit die. (a) Without gas injection; and (b) with gas injection.](image)

<table>
<thead>
<tr>
<th>Melt pump speed (rpm)</th>
<th>Melt flow rate (g s⁻¹)</th>
<th>Wall shear rate γw (s⁻¹)</th>
<th>Exp. die swell, Bᵃ</th>
<th>No gas</th>
<th>With gas</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.036</td>
<td>1.08</td>
<td>1.48</td>
<td>1.48</td>
<td>1.31</td>
</tr>
<tr>
<td>0.2</td>
<td>0.055</td>
<td>1.64</td>
<td>1.48</td>
<td>1.48</td>
<td>1.33</td>
</tr>
<tr>
<td>0.4</td>
<td>0.096</td>
<td>2.87</td>
<td>1.49</td>
<td>1.49</td>
<td>1.35</td>
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<td>1.52</td>
<td>1.52</td>
<td>1.35</td>
</tr>
<tr>
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<td>0.211</td>
<td>6.29</td>
<td>1.53</td>
<td>1.53</td>
<td>1.38</td>
</tr>
</tbody>
</table>

ᵃDie swell ratio (B) is taken 2 mm downstream from the slit exit.
IV. ROD DIE EXTRUSION

A. Experimental observation

Further experiments were carried out using a rod die extrusion cell; however, in this case direct flow visualization or flow birefringence within the cell was not possible. The effect of the gas-assisted extrusion was followed in terms of the form of the extruded product and the overall pressure drop along the length of the die.

For the rod die extrusion it was found that start up conditions were important. If the extrusion process was started by initially allowing polymer to flow in the die and then introducing gas at the slit, an extrudate of the form shown in Fig. 9(a) was often achieved. In this situation there appears to be a stationary polymer layer attached to the wall of the die. We believe that under these conditions the gas has penetrated into the polymer and left a “semisolid” layer of polymer at the wall. The temperature control of

![FIG. 8. Extrusion pressure drop as a function of melt flow rate from slit die with and without gas injection.](image)

![FIG. 9. Rod die gas injection start-up procedure. (a) Starting melt extrusion before gas injection, yielding a melt layer at the wall; and (b) starting gas injection before melt extrusion, yielding a uniform gas layer at the wall. Both at a melt flow rate of 0.4 rpm.](image)
the section of the die downstream of the slit is not very precise and it is possible that a low wall temperature was causing some solidification near the wall.

A second start-up procedure produced a much more satisfactory result. In this case the gas flow was started first and then the polyethylene was extruded into an initially polymer free die. The form of the emerging extrudate is shown in Fig. 9(b) and in this case, no wall layer was seen and a high quality extrudate was obtained. All further rod die experiments described in this paper will have been started using this second start-up procedure.

B. Die swell

The visual effect that gas assisted extrusion has on the rod die extrusion profile is shown in Fig. 10. Figure 10(a) shows the typical die swell behavior of polyethylene for a normal extrusion condition. If gas is applied the extrusion profile changes to that of Fig. 10(b) where the effect of die swell is essentially eliminated.

The effect is quantified in greater detail in Table III where die swell measurements are tabulated for different flow rates with and without gas flow. The magnitude of the swell

![FIG. 10. The effect of gas injection on the die swell behavior of HDPE at a melt flow rate of 0.4 rpm. (a) Without gas injection; and (b) with gas injection.](image)

<table>
<thead>
<tr>
<th>Melt pump speed (rpm)</th>
<th>Melt flow rate (g s(^{-1}))</th>
<th>Wall shear rate (\gamma_w (s^{-1}))</th>
<th>Exp. die swell, B(^a)</th>
<th>Frost line(^b) (cm)</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td></td>
<td>No gas</td>
<td>With gas</td>
</tr>
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<td>1.38</td>
<td>1.13</td>
</tr>
</tbody>
</table>

\(^a\)Die swell ratio (B) is taken 20 mm downstream from the die exit. No gas: no gas injection. With gas: with gas injection.

\(^b\)Frost line distance is the distance from the die exit position on the extrudate where solidification first occurs.
ratio is taken 20 mm downstream from the exit of the die. The table shows that with no gas flow, there is a finite die swell of order 1.35 and for the flow rates tested this has a very weak dependence on flow rate. If gas is applied at a low flow rate die swell is nearly completely removed and as the flow rate of the polymer increases a small die swell occurs which, however, is substantially less than that of the comparable flow situation with no gas.

In order to gain insight into the stress fields occurring within the rod die, Polyflow simulations were carried out for the rod die geometry in a similar way to the previous slit die. Again the small step at the gas injection slit entrance was not taken into account and the presence of the gas layer was modeled as a full slip boundary condition. Axisymmetric and isothermal boundary conditions were also assumed for the simulation.

The numerical simulations shown in Fig. 11 reveal a similar pattern to the slit geometry. For the no gas simulation shown in Fig. 11(a) the shear stresses in the capillary are sustained to the exit of the die and significant die swell occurs. If slip is introduced downstream of the gas injection point, the situation shown in Fig. 11(b) indicates that the stresses relax within the die and at the exit the stress free polymer shows essentially no die swell. The numerical die swell ratios for each case shown in Fig. 11 yields values of 1.30 for normal extrusion and 1.10 for gas assisted extrusion. These compare with experimental values for these conditions of 1.38 and 1.13, respectively.

C. Extrusion pressure drop

Extrusion pressure drop measurements also provide support for the belief that with gas injection an essentially full slip boundary is present below the point of gas injection. Figure 12 shows the experimental measured pressure drops for the rod die as a function
of flow rate. The pressure transducer for this die was positioned approximately 100 mm upstream of the gas injection slit. The addition of gas causes the overall pressure drop to decrease to close to a value for a die that does not have the bottom section attached. The 22% reduction in extrusion pressure matches the 24% reduction in extrusion surface area where the gas is present. This result offers further compelling experimental evidence to show that a full slip wall boundary condition is achieved with the gas injection.

Finally, we noted that with the addition of gas, the solidification frost line of the extrudate moved upstream towards the die exit. The frost line is the position on the extrudate where the material changes from an essentially transparent melt to an opaque semi crystalline solid. The experimentally observed position of the frost line is shown in Table III for a series of flow rates with and without gas flow. Without gas, the position of the frost line shows the expected trend of increasing length from the die exit with increasing mass flow rate. When gas is applied there is a substantial decrease in the position of the frost line for all flow rates. From these results and also our start up experiments we believe that the gas entering the die at the slit is not at the full melt temperature of 180 °C. The gas then has a surface cooling effect both within the die and downstream of the exit. This in turn enhances heat transfer to the surface of the polymer and promotes early solidification at the surface.

V. CONCLUSIONS

This paper has shown that it is possible to extrude molten polyethylene in a capillary or slit configuration whilst maintaining a stable gas film at the polymer wall boundary. The direct optical observation of the gas film formation shows without ambiguity, that it is possible to form a continuous gas layer at the boundary. At this stage we have not been able to experimentally measure the thickness of this gas layer, but tentatively would expect it to be of order of 100 μm or less. Amongst other factors, the control of gas pressure appears to be an important part of obtaining a stable extrusion. At start up and with polymer already in the die, if the pressure $P_g$ at the gas slit exit is below that of the normal extrusion pressure of the polymer $P_p$, clearly there will be no ingress of gas into

![Graph showing extrusion pressure drop as a function of melt flow rate from rod die with different flow boundary conditions.](image-url)
the molten polymer. If the gas pressure is say 10 bar above that of the extrusion pressure, the gas penetrates into the molten polymer in the form of bubbles and thereby produces an irregular flow and extrudate. If \( P_g \) is initially greater than \( P_p \) and then able to be adjusted, a stable film of gas can be formed as shown visually in Fig. 4. The effect is robust and could readily be achieved for both slit and rod die. The ability to form a stable gas layer is crucial for successful operation and this aspect will be sensitive to gas pressure, slit geometry, die geometry, and polymer rheology. All aspects need to be explored in the future and ideally modeled, thereby enabling optimum design to be achieved. In particular gas pressure and temperature measurements need to be made in the region of the gas boundary layer.

The formation of a low viscosity gas layer at the wall has a profound effect on the process rheology of the flowing melt. This can be seen with great clarity, from the flow birefringence shown in Fig. 6. With the injection of gas the wall shear stresses downstream of the injection point rapidly dissipate and as a consequence of this the overall stress level of the polymer at the exit of the die is substantially reduced. This in term has a major effect on the magnitude of the die swell of the polymer and demonstrates in a striking way that die swell itself is sensitive to both the polymer rheology and wall boundary conditions.

The near elimination of die swell and the ability to produce an essentially stress free extrudate are both aspects that should in the future provide process enhancement. In addition a precooiling effect of the gas at the wall which results in the polymer frost line moving upstream also should make the ability to form precision extrusion profiles possible.

The experimental configuration described in this paper offers exciting possibilities for systematically exploring the way in which wall boundary conditions can influence the process rheology of polymer and of other complex fluids. The optical transparency of molten polyethylene and the ability to follow stress fields using flow birefringence techniques provides an excellent system to carry out further detailed studies. In addition these experiments indicate an experimental way in which essentially stress free precision extrusion profile can be made. Because gas-assisted extrusion clearly alters the surface boundary conditions at the die exit it is also plausible to anticipate that this process might suppress or influence the onset of certain surface extrusion instabilities and thereby enable higher maximum extrusion rates to be achieved without a loss of extrusion profile quality.

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