3 = salt

Note: the term salt as used above also refers to the mixed salt pair collectively in those systems containing mixed salts

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On Intrinsic Errors in Pressure-Hole Measurements in Flow of Polymer Melts

CHANG DAE HAN

Department of Chemical Engineering
Polytechnic Institute of Brooklyn, Brooklyn, New York 11201

In order to investigate the possibilities of pressure-hole errors in the measurement of wall normal stresses in the flow of polymer melts, a 2.280-in. long slit die has been designed, having a slot with a rectangular cross section, 0.050 in. by 1.000 in. Three transducers were mounted on each of the two long sides of the rectangular slot along the longitudinal center line of the die and located so that each transducer was directly opposite another in the opposite side. The three transducers on one side were flush mounted. Those on the other side were mounted through pressure-holes with different diameters (0.017, 0.034, and 0.050 in.). Measurements of wall normal stresses were made with high density polyethylene and polypropylene melts at 200°C., following the experimental procedure described in the author's earlier papers. The present study indicates that pressure measurements made through pressure-holes are essentially the same as those made flush-mounted instruments, and that there are *no* pressure hole errors in the measurements of wall normal stresses, insofar as polymer melts are concerned.

In recent years several authors (*1* to *5*) studying the rheological properties of viscoelastic fluids have questioned the validity of normal stress measurement obtained by pressure transducers mounted on duct walls through pressure-holes. In most cases, such pressure measuring devices are connected to small pressure-holes. These holes are drilled through the wall normal to the surface mainly because the sensing device is much larger than the capillary diameter (or the gap between two plates).

Kaye et al. (*2*) reported that they found a systematic error in their pressure measurements using a cone-and-plate rheometer, and they attributed the error to the existence of small pressure-holes. They then presented an empirical correlation between the pressure-hole error and the shear stress by

$$p_H = -3.0\,|\tau_w| + 414 \qquad (1)$$

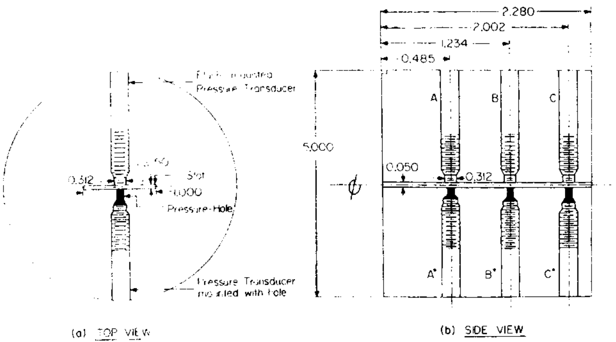

Fig. 1. Details of the slit die design: (a) top view; (b) side view.

in the range $200 < |\tau_w| < 500$ dynes/sq.cm.

More recently, Tanner and Pipkin (3) reported their experimental results, showing that the pressure-hole errors were about 25% of the primary normal stress difference, that is,

$$p_H = -0.25\ (P_{11} - P_{22}) \qquad (2)$$

Tanner and Pipkin (3) then presented an analysis using the second-order approximate constitutive equations which supports their experimental results. It should be noted here that the analysis of Tanner and Pipkin is strictly restricted to flows so slow that the second-order approximate constitutive equations are valid. For practical purposes, however, one is more concerned with the non-Newtonian flow regime where the elastic effects become most interesting.

It is further to be noted that the experimental work of Kaye et al. (2) and Tanner and Pipkin (3) dealt with dilute polymer solutions. However, polymer melts are perhaps more important than dilute polymer solutions from the standpoint of industrially useful processing materials.

In recent years, Han et al. (6 to 10), in an attempt to characterize polymer melts both in terms of viscous and elastic properties, measured wall normal stresses along the direction of flow in both circular tubes and rectangular ducts. Their pressure readings were taken with melt pressure transducers mounted normal to the solid surface through small pressure-holes. In one case, these authors (7) studied the pressure-hole errors by mounting the tips of the pressure transducers as close to the tube wall as possible and found *no* pressure-hole errors. Actually, it was not possible to mount the tips of the pressure transducers perfectly flush with the tube wall because the transducer tip is flat.

The purpose of this paper is to present some new experimental results of wall normal stress measurements of polymer melts, which have been obtained by the use of a newly designed slit die. Since the flat wall of the slit now made flush-mounting possible, wall normal stresses were measured both with and without pressure-holes and the two sets of measurements were compared.

TABLE 1. DIMENSIONS OF PRESSURE-HOLES
Sloth thickness (h) = 0.050 in.; Slot width (w) = 1.000 in.

Pressure-hole	Diameter, d_H, in.	d_H/h	Location from the die inlet, in.
A*	0.050	1.00	0.485
B*	0.034	0.68	1.235
C*	0.017	0.34	2.002

EXPERIMENTAL

Apparatus and Procedure

The apparatus consists of an extruder, a calming section, a reservoir section, and a slit die section. Polymer pellets, continuously fed to the extruder through a feed hopper, are melted, and the molten polymer is forced to flow into a calming section which is made of an 8-in. length of steel rod, drilled 0.75 in. in diameter. The reservoir section is a piece of brass rod 5 in. O.D. (1.15 in. I.D.) and 10 in. long bolted between the calming section and the slit die section. Molten polymer flows from the reservoir section into the die section, machined from a piece of aluminum bar, 5 in. in diameter and 2.280 in. long. The die section has a thin slot with rectangular cross-section, 0.050 in. by 1.000 in.

To measure wall normal stresses along the longitudinal direction, pressure transducers were mounted on the long side of the rectangle in two ways. (See Figure 1.) Three transducers (A, B, C) were mounted flush with the wall, so that there was no dead space between the flow channel (slot) and the tip of the pressure transducers. On the other side, three transducers (A*, B*, C*) were mounted through small pressure-holes directly opposite side to those mounted flush with the wall. In order for us to investigate the effects if any of the size of the pressure-holes, they had different dimensions as shown in Table 1. Figure 1 shows the side view of the slot and the location of each transducer with respect to the die entrance.

The transducers were of the bonded strain-gauge type. (Dynisco model PT 432.) The millivolt output of these instruments were measured by a potentiometer (Leeds and Northrup model, Type K-4) and Null detector. The transducers were calibrated against a dead weight tester and were found to give outputs repeatable to within ± 1% of the measured values over a range of 50-1500 lb./sq.in.g.

The temperature of the system was monitored at various positions along the calming section, reservoir section, and die section with iron-constantan thermocouples. Heat was supplied

TABLE 2. RESULTS OF TEST OF PRESSURE-HOLE ERRORS FOR HIGH DENSITY POLYETHYLENE AT 200°C.

	True shear rate, sec.$^{-1}$				
	123.2	161.6	205.8	250.6	329.0
Wall normal stress, lb./sq. in. gauge					
Transducer A	667.60	739.97	811.77	868.19	950.75
Transducer A*	669.98	739.62	812.71	864.71	949.46
Transducer B	393.68	435.78	476.53	509.26	560.34
Transducer B*	393.71	435.41	476.26	507.60	558.31
Transducer C	113.61	126.34	139.68	150.65	166.03
Transducer C*	113.01	126.68	138.42	150.43	164.72
True shear stress, lb./sq. in.	9.13	10.11	11.07	11.82	12.93
Exit pressure, lb./sq. in. gauge	12.69	14.29	16.29	18.58	22.29
Reynolds number	0.239×10^{-3}	0.375×10^{-3}	0.557×10^{-3}	0.771×10^{-3}	0.121×10^{-2}

* Transducer with pressure-hole. (See Figure 1 for the locations of pressure transducers.)

TABLE 3. RESULTS OF TEST OF PRESSURE-HOLE ERRORS FOR POLYPROPYLENE AT 200°C.

	True shear rate, sec.$^{-1}$				
	98.9	171.1	216.0	265.8	320.3
Wall normal stress, lb./sq. in. gauge					
Transducer A	467.93	582.14	635.00	683.21	725.84
Transducer A*	470.21	582.73	638.91	684.90	726.32
Transducer B	278.21	344.10	377.85	404.94	432.81
Transducer B*	277.06	344.20	378.02	404.83	432.44
Transducer C	73.07	94.02	102.73	111.74	119.52
Transducer C*	74.03	92.70	103.51	113.11	120.18
True shear stress, lb./sq. in.	6.51	8.04	8.77	9.42	9.99
Exit pressure, lb./sq. in. gauge	2.94	6.18	7.75	9.05	11.29
Reynolds number	0.218×10^{-3}	0.535×10^{-3}	0.780×10^{-3}	0.109×10^{-2}	0.148×10^{-2}

* Transducer with pressure-hole. (See Figure 1 for the locations of pressure transducers.)

by means of resistance wire wound on the whole system for uniform heat distribution. The entire apparatus was heavily insulated with asbestos. Temperature was controlled to within ± 0.5°F. by a thermistor-operated thermal regulator which consists of the regulator probe and an on-off type controller.

The apparatus is first brought up to a desired operating temperature, over a period of 4-5 hr. Once the system is in equilibrium, a volumetric flow rate of melt is set by the screw speed of the extruder and samples are collected over predetermined time intervals and weighed to calculate apparent shear rate.

Materials

1. High density polyethylene (DMDJ 4309) of Union Carbide Products. It has a polydispersity ($\overline{M}_w/\overline{M}_n$) of about 84, and a melt index of 0.2.
2. Polypropylene (Resin E115) of Enjay Chemical Products. It has a melt index of about 2.4.

RESULTS AND DISCUSSION

Test of "Pressure-Hole" Error

Measurements of wall normal stresses are given in Table 2 for high density polyethylene melts, and in Table 3 for polypropylene melts, both at 200°C. Note that transducers A, B, and C were mounted flush with the slit wall, and that transducers A*, B*, and C* were mounted with pressure-holes. It is clearly seen from Tables 2 and 3 that there is *no* discernible difference in pressure measurements between the transducers with and without pressure-holes.

Two things are worth noting about the measurements. First, pressure readings are repeatable to within ± 1% of the measured values over a range of 50-1500 lb./sq.in. gauge. Secondly, the pressure readings from transducers A, B, and C are the average values over the distance of 0.312 in., which is the diameter of the pressure transducer tip. On the other hand, the pressure readings from transducers A*, B*, and C* are the average values over the distance of 0.050 in. for A*, 0.034 in. for B*, and 0.017 in. for C*, respectively. (See Figure 1b.) With these two facts, one can observe from Tables 2 and 3 that the size of pressure-holes does not appear to affect pressure measurements.

Therefore one can conclude from the present study that there is *no* pressure-hole error in measurement of wall normal stresses, insofar as polymer melt is concerned. The conclusions drawn above are *not* in agreement with those reported in the literature (*1* to *3*) which dealt with dilute polymer solutions. In order to give some plausible explanation for the observed difference in the role of pressure-hole between polymer melts and dilute solutions, we shall next discuss both viscous and elastic properties of the polymer melts studied, using the pressure measurements given in Tables 2 and 3.

Determination of Viscous and Elastic Properties of Polymer Melts

As has been described by Han (*6* to *10*), measurements of axial pressure distribution permit one to determine both viscous and elastic properties of polymer melts, so long as measurements are taken in the region where flow is fully developed. A criterion for fully developed flow of viscoelastic fluids has recently been suggested by the author (*11*).

In Figure 2 are given representative axial pressure profiles for high density polyethylene melts at 200°C. Similar pressure profiles were obtained for polypropylene melts also. It is seen from Figure 2 that the three pressure measurements lie on a straight line for each given shear rate, and that extrapolating the straight line portion to the die exit gives rise to a non-zero gauge pressure, which is called the exit pressure (*6*, *7*). More will be said about the exit pressure later.

In order to determine the rheological properties of melts, the pressure profiles were represented by straight lines determined by means of the method of least squares. It was found that the root mean square errors lie somewhere

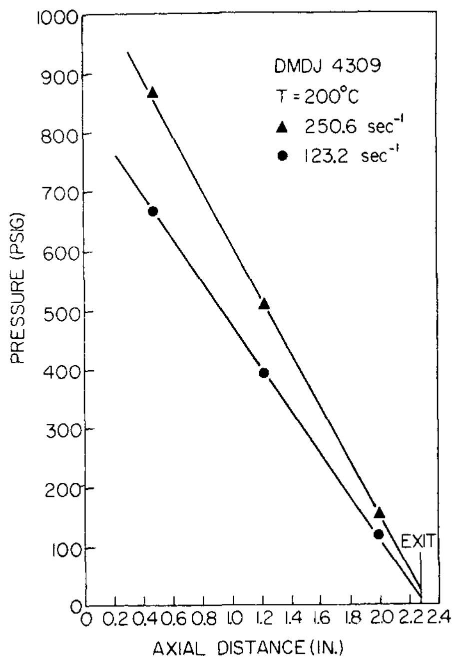

Fig. 2. Representation of axial pressure distributions for polyethylene melts at 200°C.

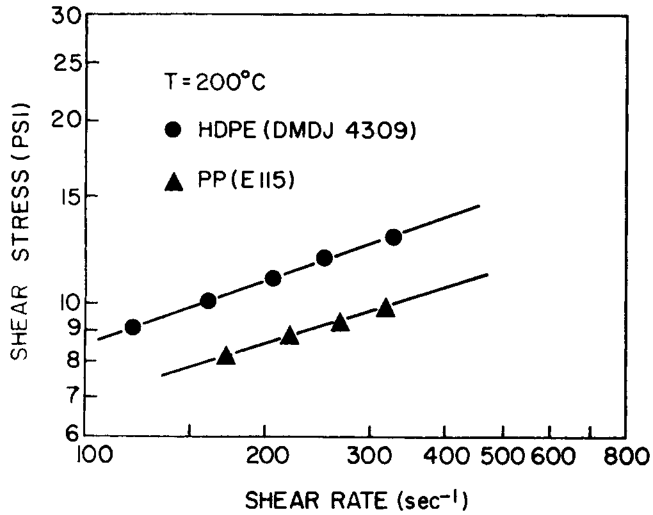

Fig. 3. Shear stress versus shear rate for polyethylene and polypropylene melts at 200°C.

in the range of 0.3 ~ 1.5 lb./sq.in. gauge and that the percentage error of each pressure measurement was within ± 1% in all cases.

One can calculate the true wall shear stress τ_w by

$$\tau_w = \left(\frac{-\partial p}{\partial x}\right) \frac{h}{2} \tag{3}$$

It is to be noted that the slit used has an aspect ratio (ratio of long side to short side of rectangle) of 20, and therefore that flow through such a thin slit can be assumed to be one-dimensional, instead of two-dimensional. This is because the thin slit may be considered to be a substitute for two parallel plates with infinite width. Note also that the first pressure transducer is located 0.485 in. from the entrance of the slit die section (see Figure 1b), which gives a slit length-to-thickness (L/h) ratio of about 10. This value of L/h ratio is sufficiently large to ensure that the axial pressure gradient becomes constant, considering that the same materials (both DMDJ 4309 and Resin E115) require a capillary length-to-diameter (L/D) ratio of about 2 for achieving a constant pressure gradient (*11*).

The apparent shear rate $\dot{\gamma}_w$ may be defined by

$$\dot{\gamma}_w = 6Q/wh^2 \tag{4}$$

The true shear rate $\dot{\gamma}$ with Rabinowitch-Mooney correction may be written as (*12*):

$$\dot{\gamma} = \frac{\dot{\gamma}_w}{3}\left(2 + \frac{d \ln \dot{\gamma}_w}{d \ln \tau_w}\right) \tag{5}$$

Using Equations (3) through (5) one can now construct flow curves (plots of shear stress versus shear rate) from measurements of the pressure gradient ($-\partial p/\partial x$) and volumetric flow rate Q. It is worth noting that use of Equation (3) does not require end-correction in constructing flow curves.

Figure 3 shows flow curves for both polyethylene and polypropylene melts at 200°C., from which it is seen that the materials used follow the power law

$$\tau_w = K \dot{\gamma}^n \tag{6}$$

over the range of shear rate studied. One can further construct viscosity curves, which are shown in Figure 4. These curves show that the materials used are in the non-Newtonian flow regime, suggesting further that the rheological behavior of the materials should be described by nonlinear constitutive equations, at least over the range of shear rate studied.

As stated above in connection with the axial pressure profiles, use of the exit pressure permits one to describe elastic behavior of materials. Note from Figure 2 that the exit pressure increases with shear rate. In Figure 5 are shown plots of exit pressure versus shear rate for both polyethylene and polypropylene melts, which further suggests the power law relation

$$P_{\text{exit}} = \alpha \dot{\gamma}^{\beta} \tag{7}$$

According to Han (*6, 7, 9, 10*), the exit pressure is approximately equal to the primary normal stress difference for polymer melts; therefore, Equation (7) may be rewritten as

$$P_{11} - P_{22} = \alpha \dot{\gamma}^{\beta} \tag{8}$$

Figure 5 and Equation (8) indicate that the materials used do not follow the second-order approximate constitu-

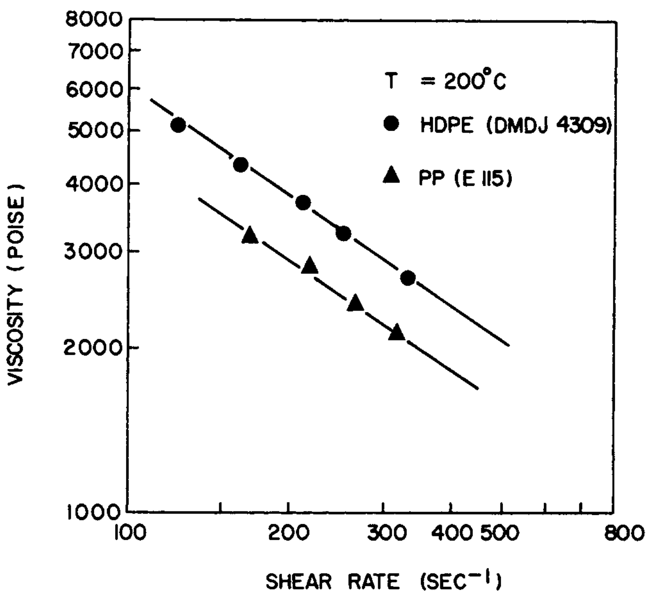

Fig. 4. Viscosity versus shear rate for polyethylene and polypropylene melts at 200°C.

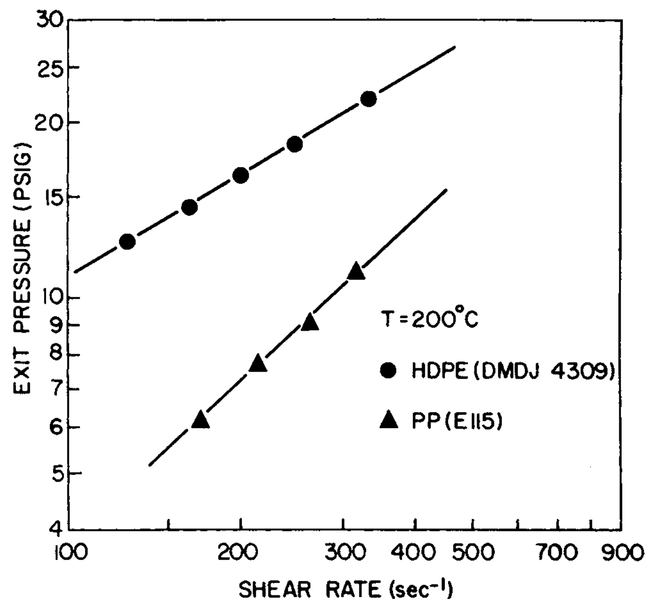

Fig. 5. Exit pressure versus shear rate for polyethylene and polypropylene melts at 200°C.

tive equations over the range of shear rate studied, because the constant β in Equation (8) is much less than 2.0.

Variables Which Could Affect Wall Pressure Measurement

About a decade ago, Shaw (*13*) presented an analysis to show that pressure-hole error may be related to shear stress, Reynolds number, and hole dimensions

$$p_H = \tau_w \, f(N_{Re}, d_H/D) \tag{9}$$

It can be seen from Equation (9), that Shaw (*13*) used the dimensional analysis for turbulent gas flow and presented experimental data to support his analysis. A few years later, Jackson (*14*) showed that the pressure-hole error approaches zero as Reynolds number approaches zero even in the flow of gas.

It is therefore interesting to calculate the Reynolds number for the polymer melts used, over the range of shear rate studied. For this, we use the generalized Reynolds number

$$N_{Re} = \frac{\rho \, h^n \, V^{2-n}}{K_p \, 8^{n-1} \left(\dfrac{3n+1}{4n} \right)^n} \tag{10}$$

Calculated values of N_{Re} are given in Table 2 for high density polyethylene melts and in Table 3 for polypropylene melts. Also given in Tables 2 and 3 are shear rate, shear stress, and the exit pressures. Note that calculation of N_{Re} requires information of K and n in the power law expression Equation (6).

Now then, it is clearly seen that Reynolds numbers of polymer melts are exceedingly low, lying somewhere about 10^{-4}-10^{-2} over the range of shear rate studied. Such low values of N_{Re} are characteristic of polymer melts in general, which is attributable to their very large values of viscosity. It is to be noted that for a given melt there is always an upper limit of shear rate, called the critical shear rate, beyond which flow instabilities occur. Therefore shear rates higher than those reported here would not appreciably increase the Reynolds numbers. One can now understand why the experimental results of the present study show *no* pressure-hole errors if one follows the analyses by Shaw (*13*) and Jackson (*14*) by use of N_{Re}.

Our results clearly indicate that the analyses of Tanner and Pipkin (*3*) and Kearseley (*4*), who used the second-order approximate constitutive equations to describe the pressure-hole effect of viscoelastic fluids, do not describe the experimental observations presented above. The reasons are clear from Figures 3 through 5 which indicate that the rheological behavior of the polymer melts used cannot be described by the second-order fluid model. First as shown in Figure 4 the polymer melts used show shear-dependent viscosity, while the second-order fluid predicts a viscosity independent of shear rate. Secondly as shown in Figure 5 the polymer melts used show a dependence of elastic behavior (in terms of primary normal stress difference) on shear rate much lower (about first power of shear rate) than the second power of shear rate, which is what the second-order fluid predicts.

It is to be noted that our study shows *no* effect of pressure-hole dimensions on wall pressure measurements over the range of pressure-hole sizes used: $d_H/h = 0.34, 0.67, 1.0$. (See Table 1.) On the other hand, it is quite inconceivable that anyone would drill pressure-holes as large as the capillary diameter for measurements of wall pressure in circular tubes. Based on the facts presented above, it *can be confidently said that all the previous measurements* by the author (*6* to *11*) are indeed free from pressure-hole errors. It happens that pressure-hole diameter used was about 25% of the capillary diameter.

Fig. 6. A schematic of the isovels in a slit having a side channel.

In this study, however, an additional attempt has been made, to investigate what would be the effect, if any, on wall pressure measurements when the dimensions of pressure-holes were even much larger than the slot thickness. For this, the flush-mounted pressure transducers (A, B, C as referred to in Figure 1) were pulled back slightly (about ⅛ in.) from the solid wall, thereby creating a dead space between the tips of the pressure transducers and the solid wall. The dead space will hereafter be called side-channel for an obvious reason, because it has a diameter of 0.312 in. (the diameter of the pressure transducer tip itself) which is 6.24 times the slot thickness, that is, $d_H/h = 6.24$.

Measurements of wall normal stresses in the presence of side-channels are given in Table 4 for high density polyethylene at 200°C. One can now make the following interesting observations by comparing wall normal stress data in Table 2 with those in Table 4. First, the presence of side-channels give rise to a substantial *decrease* in wall pressure readings, and hence pressure-hole errors. This observation is, in fact, consistent with that made by Tanner and Pipkins (*3*) who used dilute polymer solutions. It should be remembered, however, that Tanner and Pipkins had very small pressure-holes, and that our study shows *no* pressure-hole errors (see Table 2 and 3) with small holes.

The decrease in wall pressure readings observed above may be attributable to a pronounced flow disturbance caused by the "side-channels," as schematically shown in Figure 6. As given by Equation (9), the extent of flow disturbance may depend on both Reynolds number and the size of the pressure-holes. The present study seems to indicate, however, that in polymer melt flow the presence of pressure-holes introduces little disturbance in flow pattern and hence gives rise to no discernible errors, as long as their size is kept small. This means smaller than the slot *thickness in slit flow and smaller than 1/3 of the capillary* diameter in tubular flow. Reynolds numbers in polymer melt flow are exceedingly low, say below 0.01, in almost all practical situations. However, the situation becomes quite different in the flow of dilute polymer solutions for

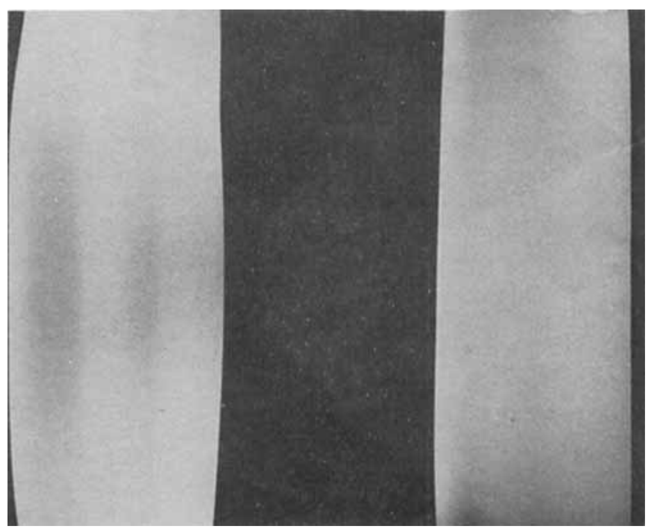

Fig. 7. Extrudate swell behavior from a slit die: (a) In the presence of side-channels; (b) In the presence of small pressure-holes.

TABLE 4. EFFECT OF SIDE-CHANNELS ON WALL NORMAL STRESS MEASUREMENTS FOR HIGH DENSITY POLYETHYLENE AT 200°C.

	True shear rate, sec.$^{-1}$				
	161.4	205.1	250.7	299.2	328.9
Wall normal stress, lb./sq. in. gauge					
Transducer A	730.0	792.7	850.0	908.2	936.6
Transducer B	423.8	459.9	493.7	527.5	543.8
Transducer C	110.6	118.8	130.0	140.3	146.7
True shear stress, lb./sq. in.	10.21	11.11	11.86	12.65	13.02
Exit pressure, lb./sq. in. gauge	−3.06	−4.63	−2.16	−0.97	1.08
Reynolds number	0.356×10^{-3}	0.529×10^{-3}	0.738×10^{-3}	0.989×10^{-3}	0.116×10^{-2}

which Reynolds numbers can be made very high. Therefore it is quite conceivable that for a given size of pressure-holes, one would observe errors with dilute polymer solutions where none would be visible with polymer melts.

An interesting observation has been made of extrudate swell behavior with and without side-channels, as shown in Figure 7. Noting that the ratio of side-channel diameter (d_H) to the width (w) of the slot is 0.312, it is seen from Figure 7 that the extrudate in the *presence* of side-channels exhibits excessive swell in the middle, whereas the extrudate in the *absence* of side-channels does not. The extrudate pictures clearly show the fact that the polymer melt in the presence of side-channels has undergone a deformation process different from that without side-channels.

Now then, a natural question may arise as to the effect of side-channels on the rheological properties of melts, in particular the exit pressures. For this, Table 4 also gives calculated values of true shear stress, exit pressure, and Reynolds numbers. It is seen from Table 4 that the presence of side-channels gives rise to *negative* exit pressures. This is a consequence of the lower values of wall normal stress readings. It should be emphasized once again that the *negative* exit pressures given in Table 4 are due to the presence of large side-holes, referred to here as side-channels, whose diameter is 6.24 times the slot thickness (that is, $d_H/h = 6.24$), whereas only *positive* exit pressures have been obtained in the absence of side-channels. (See Tables 2 and 3 and references *6-11*.) This seems, then, to explain why *negative* exit pressures have been reported (*15*) in the measurement of wall normal stresses for dilute polymer solutions flowing through circular tubes, having pressure-holes much smaller than the tube diameter. However, a further study is necessary to accurately determine how small the pressure-holes should be in order to avoid such errors when testing dilute polymer solutions.

CONCLUDING REMARKS

Wall normal stresses of polymer melts have been measured by the use of a newly designed slit die. In order to test the possibility of having pressure-hole errors in the flow of polymer melts, the slit die was designed to provide for measuring wall normal stresses both with and without pressure-holes.

The results of the present study indicate that there is no pressure-hole error in the measurement of wall normal stresses, insofar as polymer melt is concerned. Explanations are then given for the difference in behavior of polymer melts from that of dilute polymer solutions, which may give rise to substantial pressure-hole errors. The present study therefore will give comfort to those who wish to measure wall normal stresses of polymer melts by drilling small pressure-holes in the wall of a flow duct. Such practices are very common in polymer processing industries, such as plastic extrusion and fiber spinning, etc.

NOTATION

d_H = diameter of pressure-hole, in.
D = tube diameter, in.
g_c = conversion factor, 32.2 (lb.$_m$) (ft.)/(lb.$_f$) (sec.^{2})
h = the slit thickness (short side of rectangle), in.
K = fluid consistency defined in (6), lb.$_f$/(sq.in.) (sec.$^{-n}$)
K_p = defined as g_cK, lb.$_m$/(in.) (sec.$^{2-n}$)
N_{Re} = Reynolds number defined by (10), dimensionless
n = flow index defined in (6)
P_{exit} = exit pressure, lb.$_f$/sq.in.
$-\frac{\partial p}{\partial x}$ = pressure gradient, lb.$_f$/cu.in.
$P_{11} - P_{22}$ = primary normal stress difference, lb.$_f$/sq.in.
p_H = pressure-hole error, lb.$_f$/sq.in.
Q = volumetric flow rate, cu.in./sec.
V = average fluid velocity, in./sec.
w = the slit width (long side of rectangle), in.
ρ = fluid density, lb.$_m$/cu.in.
τ_w = shear stress, lb.$_f$/sq.in.
$\dot{\gamma}$ = true shear rate defined by (5), sec.$^{-1}$
$\dot{\gamma}_w$ = apparent shear rate, sec.$^{-1}$
α, β = constants defined in (7)

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