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Hydrostatic extrusion behaviour of high density polyethylene

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The solid state processing of a high density polyethylene by hydrostatic extrusion at room temperature has been investigated. The extrusion pressure for a given extrusion ratio is found to depend on the pressure fluid used, the die angle and the velocity of extrusion. The strain hardening behaviour of the resulting extrudates is found to be independent of the extrusion ratio. An effective flow stress equation which accounts for the dependence of the flow stress of high density polyethylene on strain, strain rate and hydrostatic pressure is developed and used in an analysis of the extrusion process based on the upper bound approach. The calculated values of the extrusion pressure as a function of the various extrusion variables are in reasonable agreement with the experimental results.

1. Introduction

The application of the process of hydrostatic extrusion to polymers is a subject of active interest. The first measurements appear to be those of Pugh and Low [1] on the hydrostatic extrusion of polyethylene and PTFE at room temperature. Since then, studies on a number of polymers have been reported [2–14].

The mechanics of hydrostatic extrusion of metals are well developed [15, 16]. However, such methods cannot be employed directly in the analysis of hydrostatic extrusion of polymers because of the occurrence of significant strain hardening [17, 18], pressure hardening [19–22], and strain rate sensitivity [23–25] in polymers. A few investigators have analysed the extrusion process for polymers using approximate techniques derived from plasticity theory. Taking strain hardening into account, Imada and co-workers [26–28] analysed the conventional ram extrusion of several polymers including high density polyethylene and found reasonable agreement between calculated and observed extrusion pressures. The approaches employed by these authors included the pressure balance scheme [26], upper bound analysis [27], and slip line analysis [26]. Davis [11] analysed the hydrostatic extrusion of a linear polyethylene taking into account the flow stress dependence on strain and hydrostatic pressure but failing to account for friction and shear losses. An analysis of hydrostatic extrusion of polymers based on Sach's analysis [29] where the flow behaviour of the polymer with regard to large deformations, hydrostatic pressure and varying strain rates while passing through the extrusion die has recently been developed by Capaccio *et al.* [30].

In the present study, relationships between process variables in the hydrostatic extrusion of high density polyethylene, in particular, the effect of the extrusion ratio, extrudate velocity, die angle and pressure fluid on the extrusion pressure, are reported. The measurements are analysed in terms of Avitzur's [15] upper bound theory taking into account the strain, strain rate and pressure sensitivity of the flow stress of the polymer as given by recent models of thermally activated deformation.

2. Extrusion analysis

The upper bound approach has been used in many applications for estimating forming pressure. In the extrusion process a velocity field within the extrusion die is assumed and the pressure required

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to maintain this flow field, based on the yield behaviour of the material being extruded, is determined from the total applied power, J, [15].

$$J = \dot{W}_{\mathrm{I}} + \dot{W}_{\mathrm{R}} + \dot{W}_{\mathrm{F}} \qquad (1)$$

where $\dot{W}_{\mathrm{I}}$ is the power required for internal deformation as the billet passes through the die, $\dot{W}_{\mathrm{R}}$ is the redundant power loss associated with the velocity discontinuities at the die entrance and exit boundaries, and $\dot{W}_{\mathrm{F}}$ is the frictional power loss along the die surface.

Assuming proportional straining (spherical velocity field) through the die, $\dot{W}_{\mathrm{I}}$ for materials obeying von Mises yield criterion may be expressed as [15]

$$\dot{W}_{\mathrm{I}} = \frac{4\pi v_{\mathrm{f}} R_{\mathrm{f}}^2}{\sin^2\alpha} \int_{R_{\mathrm{f}}}^{R_0}\int_0^{\alpha} \sigma_{\mathrm{f}}(1 - \tfrac{11}{12}\sin^2\theta)^{1/2} \sin\theta \, \mathrm{d}\theta \, \frac{\mathrm{d}R}{R} \qquad (2)$$

where σ_{f} is the flow stress, v_{f} is the extrudate velocity, R_0 is the radius of the polymer billet to be extruded, R_{f} is the radius of the die exit and α is the semi-cone angle of the die.

The redundant power loss at the die entrance and exit boundaries is given by [15]

$$\dot{W}_{\mathrm{R}} = \frac{2\pi v_{\mathrm{f}} R_{\mathrm{f}}^2}{\sqrt{3}\sin^2\alpha} \int_0^{\alpha} [\sigma_{\mathrm{f}(R=R_0)} + \sigma_{\mathrm{f}(R=R_{\mathrm{f}})}] \sin^2\theta \, \mathrm{d}\theta \qquad (3)$$

where $\sigma_{\mathrm{f}(R=R_0)}$ and $\sigma_{\mathrm{f}(R=R_{\mathrm{f}})}$ are the flow stresses at the die entrance and exit respectively.

If Coulomb friction is assumed, the frictional power loss may be expressed as:

$$\dot{W}_{\mathrm{F}} = 2\pi v_{\mathrm{f}} R_{\mathrm{f}}^2 (\cot\alpha)\mu \times \int_{R_{\mathrm{f}}}^{R_0} \sigma_{\mathrm{f}(\theta=\alpha)} \left[1 - \ln\left(\frac{R_{\mathrm{f}}}{R}\right)^2\right] \frac{\mathrm{d}R}{R} \qquad (4)$$

where the zero-friction normal die stress approximation [29] has been used.

Substituting Equations 2 to 4 into Equation 1, with the relation $J = \pi v_{\mathrm{f}} R_{\mathrm{f}}^2 P_{\mathrm{ex}}$, gives the extrusion pressure as:

$$P_{\mathrm{ex}} = \frac{4}{\sin^2\alpha} \int_{R_{\mathrm{f}}}^{R_0}\int_0^{\alpha} \sigma_{\mathrm{f}}(1 - \tfrac{11}{12}\sin^2\theta)^{1/2} \sin\theta \, \mathrm{d}\theta \, \frac{\mathrm{d}R}{R} + \frac{2}{\sqrt{3}\sin^2\alpha} \int_0^{\alpha} [\sigma_{\mathrm{f}(R=R_0)} + \sigma_{\mathrm{f}(R=R_{\mathrm{f}})}] \sin^2\theta \, \mathrm{d}\theta + 2(\cot\alpha)\mu \int_{R_{\mathrm{f}}}^{R_0} \sigma_{\mathrm{f}(\theta=\alpha)} \left[1 - \ln\left(\frac{R_{\mathrm{f}}}{R}\right)^2\right] \frac{\mathrm{d}R}{R} \qquad (5)$$

In most applications of upper bound theory to extrusion σ_{f} is assumed constant and Equation 5 solved in closed form to give the extrusion pressure. In polymeric materials, which exhibit flow properties which are anisotropic and significantly dependent on strain, strain rate and pressure, it is clear that a more accurate estimate of the extrusion pressure may be obtained by taking into account the variation of σ_{f} through the die. Since the deformation is mainly tensile in character the variation of σ_{f} may be estimated from tensile measurements. Such measurements also reflect the development of anisotropy with strain. In principle the above analysis only applies to isotropic materials, however, for tensile deformation Hill's [31] anisotropic yield criterion, when applied to materials exhibiting planar isotropy, reduces to the same form as the von Mises criterion.

Previous studies taking into account flow stress variations through the die appear to be limited to Avitzur's linear strain hardening treatment [32], work on the extrusion of superplastic materials in which strain rate sensitivity of σ_{f} was accounted for [33], and the previously mentioned analyses of Maruyama *et al.* [27] and Capaccio *et al.* [30].

Measurements of the effect of strain, strain rate, and hydrostatic pressure on the tensile yield behaviour of polyethylene indicate that the flow stress increases exponentially with increasing strain [18], linearly with hydrostatic pressure [19], and logarithmically with strain rate [24]. Li *et al.* [34] have shown that the yield behaviour may be rationalized using rate theory. If plastic deformation occurs by a thermally activated process, the strain rate may be expressed as:

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp(-\Delta F/kT) \qquad (6)$$

where ΔF is the activation free energy associated with the rate controlling process and $\dot{\epsilon}_0$ is a constant. The effect of stress and pressure in ΔF may be expressed as [23, 34]

$$\Delta F = \Delta F_0 + P\Omega - V\sigma_{\mathrm{f}} \qquad (7)$$

where $\Omega = -kT\,\mathrm{d}\ln\dot{\epsilon}/\mathrm{d}P$ is the pressure activation volume and $V = kT\,\mathrm{d}\ln\dot{\epsilon}/\mathrm{d}\sigma$ is the stress activation volume. Substituting Equation 7 into Equation 6 gives

$$\sigma_{\mathrm{f}} = \frac{1}{V}[\Delta F_0 + P\Omega + kT\ln(\dot{\epsilon}/\dot{\epsilon}_0)] \qquad (8)$$

Measurements of V [25] in linear polyethylene have been shown to decrease with strain as $V =$

$V_0 \exp(-\beta\epsilon)$ where V_0 and β are constants. Li *et al.* [34] have suggested that the decrease in V results from an increase in the number of obstacles to the rate controlling process, thus accounting for strain hardening. Substituting the expression for V into Equation 8 the strain, strain rate, and pressure dependence of σ_f becomes

$$\sigma_f = (s + wP + m \ln \dot{\epsilon}) \exp(\beta\epsilon) \qquad (9)$$

where $s = (1/V_0)(\Delta F_0 - kT \ln \dot{\epsilon}_0)$, $w = \Omega/V_0$, and $m = kT/V_0$.

The use of Equation 9 for estimating extrusion pressures requires knowledge of the variation of strain, strain rate and pressure through the die. Avitzur [15] has shown that for a spherical strain field, the effective strain rate and strain are given by

$$\dot{\epsilon} = 2v_f R_f^2 \sin\alpha(1 - \tfrac{11}{12}\sin^2\theta)^{1/2}/R^3 \qquad (10)$$

$$\epsilon = 2(1 - \tfrac{11}{12}\sin^2\theta)^{1/2} \ln(R_0/R)/\cos\theta \qquad (11)$$

The variation of pressure through the die may be approximated from the zero-friction equilibrium stress distribution [29] in the die

$$P = \sigma_f[\tfrac{2}{3} - \ln(R_f/R)^2] \qquad (12)$$

Substitution of Equations 9 to 12 into Equation 5 allows the extrusion pressure to be evaluated numerically.

In a subsequent section experimental measurements of the extrusion pressure as a function of velocity, extrusion ratio and die angle are compared with values calculated from Equation 5.

3. Experimental details

The material studied was a high density polyethylene of density $0.96\,g\,cm^{-3}$ and melt index of 16 g/10 min (ASTM method D1238), supplied in pellet form. Specimens for tensile testing and extrusion were machined from injection moulded rods.

The hydrostatic extrusion apparatus used in the present investigation was similar to apparatus employed previously in studying extrusion behaviour of polymers [5, 11]. The pressure was generated by a Harwood high pressure system capable of delivering pressures of up to 1380 MPa. Pressures were measured using a manganin coil pressure gauge.

The extrusion dies employed had included cone angles of 10°, 20°, 30°, 45° and 60° and an output diameter of 2.51 mm. The dies had a parallel land length equal to twice the output diameter of the die. An extrusion die of included cone angle 20° with an output diameter of 5 mm was also used to obtain larger diameter extrudates for tensile testing.

The pressure fluids used were 1.0 cSt Dow Corning 200 silicone fluid, castor oil and glycerine. The effect of graphite lubricant in conjunction with these fluids was also investigated.

Both rapid and constant pressure extrusions were carried out. The rapid extrusions were performed in a manner similar to that used by Davis [11]. The polymer billet to be extruded was fitted on to the extrusion die and placed in the pressure vessel after which the pressure was increased at a rate of $17\,MPa\,min^{-1}$. The specimens extruded rapidly on reaching a critical pressure and the values of the rapid extrusion pressures subsequently reported correspond to the average of three such tests for a given set of conditions. The extrusion velocity for these tests was not measured. In the constant pressure extrusion tests a constant hydrostatic pressure was maintained and the steady state extrudate velocity was measured using a linear variable differential transformer located below the extrusion die such that the core of the LVDT was displaced by the sample emerging from the die. In these tests care was taken to ensure that the initial tapered portion of the billet had been completely extruded before making measurements.

Tensile tests were carried out on an Instron machine using a cross-head velocity of $1\,mm\,min^{-1}$. Strains were measured using a transverse strain gauge extensometer attached to the middle of the specimen gauge length, which was slightly tapered to define the location of initial yielding. All extrusions and tensile tests were carried out at 22 ± 1° C.

Results

For rapid extrusions up to extrusion ratios of 7 using an extrusion die of included cone angle 20° resulted in smooth extrudates with both silicone fluid and castor oil. Successful rapid extrusions were obtained for extrusion ratios of 9 using castor oil and less often for silicone fluid. The ends of the extrudates, however, were found to be fractured forming a closed spiral as has been observed by Davis [11] and Gibson and Ward [13].

Controlled extrusions using castor oil and

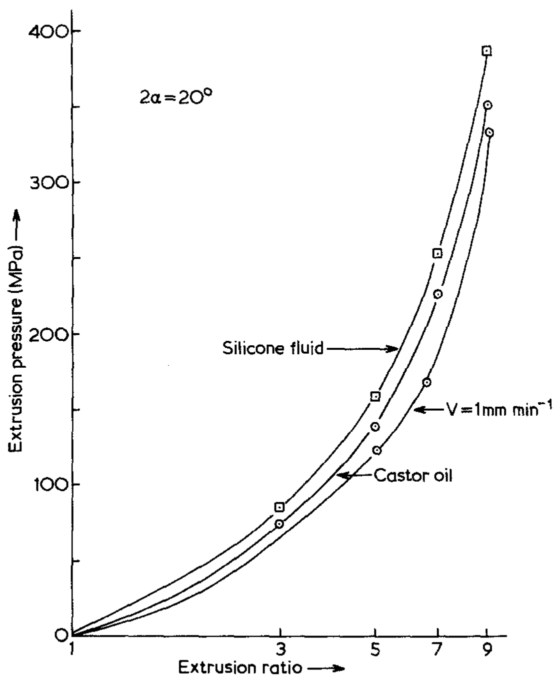

Figure 1 Effect of extrusion ratio in the rapid extrusion pressure using silicone oil and castor oil as pressure fluids and on the extrusion pressures for a constant extrudate velocity of 1 mm min^{-1} using castor oil.

a 20° die produced very smooth and flawless extrudates up to extrusion ratios of 11. The rates of extrusion were generally low (0.25 to 3 mm min^{-1}). For extrusion ratios above 11, the product was expelled violently resulting in a badly fractured product even though the rates of extrusion were small prior to the instability. Successful creep like extrusions up to an extrusion ratio of 13 could be carried out by maintaining a lower constant hydrostatic pressure for a few days. The product was found to be very stiff and highly transparent for such extrusions. Extrudates having an extrusion ratio higher than 13, however, could not be obtained even by this method.

Measurements of the extrusion pressure as a function of the extrusion ratio for both rapid extrusions and an extrudate velocity of 1 mm min^{-1} are shown in Fig. 1 for a 20° die. Rapid extrusion pressures were higher using silicone fluid compared to castor oil for all extrusion ratios. Rapid extrusion pressures were also found to be higher than the extrusion pressures recorded for an extrudate velocity of 1 mm min^{-1} for all extrusion ratios using the same pressure fluid of castor oil.

Measurements of the effects of pressure on the extrudate velocity for various extrusion ratios and die angles, using castor oil as the pressure fluid, are shown in Fig. 2. In all tests the logarithm of the extrudate velocity was found to increase linearly with the extrusion pressure. The slopes of the curves decreases with increasing extrusion ratio and die angle.

The effect of die cone angle on the extrusion pressure for $v = 1$ mm min^{-1} and $R = 6.75$ using castor oil is shown in Fig. 3. The extrusion pressure exhibited a minimum value at $2\alpha \simeq 20°$.

The influence of different pressure media and graphite lubricant on the pressure required for rapid extrusion was also studied. The results are given in Table I. Extrusions carried out using castor oil and glycerine exhibited similar extrusion pressures which were less than that observed for

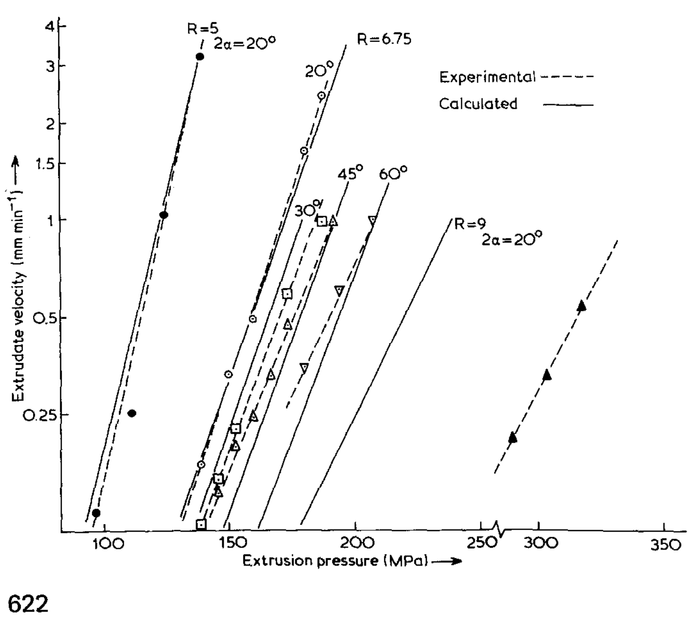

Figure 2 Relationship between logarithm of extrudate velocity and extrusion pressure for different extrusion ratios and die angles.

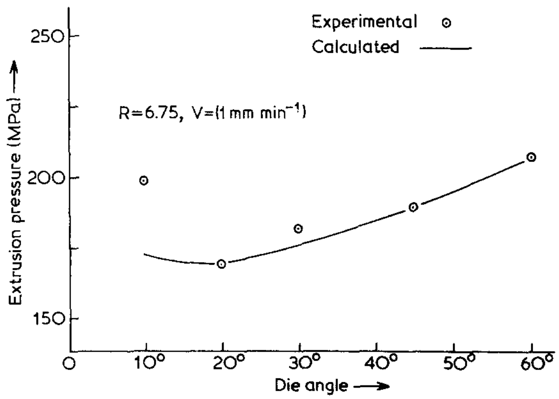

Figure 3 Effect of die angle on the extrusion pressure.

TABLE I Effect on the extrusion pressure using different pressure media and graphite lubricant. Die angle (2α) = 20°, extrusion ratio = 7

Pressure medium	Lubricant	Extrusion pressure (MPa)
Glycerine	–	210.3
Glycerine	Graphite	204.1
Castor oil	–	208.6
Castor oil	Graphite	212.8
Silicone oil	–	252.4
Silicone oil	Graphite	249.7

silicone oil. The application of graphite lubricant on to the billet did not have any significant effect on the extrusion pressure.

The results of tensile tests carried out on samples extruded to ratios of 3, 4 and 5 are shown in Fig. 4. True stress–strain curves of the extruded samples, plotted on the basis of total (extrusion plus tensile) true strain exhibited exponential strain hardening and a single true stress–strain relation was found for all samples. The slope of the curve in Fig. 4, which represents the strain hardening coefficient, β, was found to equal 0.75.

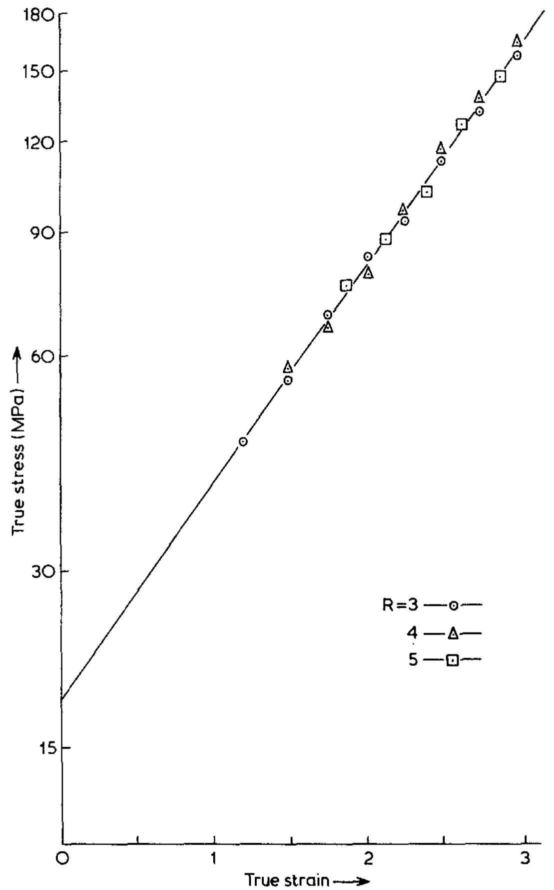

Figure 4 Relationship between logarithm of true stress and true strain for the extruded specimens of extrusion ratios 3, 4 and 5.

5. Discussion

5.1. Mechanical properties

The exponential strain hardening behaviour exhibited by the extrudates in Fig. 4 has been observed for many polymers [18] and has also been observed for polypropylene extrudates [5]. Similar behaviour has also been observed by Bahadur [18] for a range of polymers rolled to different strains. It appears that whatever the mode of deformation (tensile drawing, cold rolling, extrusion) there is a unique true stress–true strain relationship provided the starting material has the same structure and morphology. This is not surprising as the true strain determines the extent of molecular orientation which in turn determines the mechanical properties.

5.2. Extrusion behaviour

The effect of extrusion ratio on the extrusion pressure as shown in Fig. 1 is similar to that observed previously in HDPE [8, 11]. As well as the extrusion ratio, it is clear that the extrudate velocity and die angle are important variables determining the extrusion pressure. The linear $P_{ex} - \ln V$ relation observed for different extrusion ratios and die angles is similar to that observed in linear polyethylene by Gibson and Ward [13] for extrusion ratios less than 10. Examination of Equations 5 and 9 shows that such a relation would be expected since the flow stress exhibits a logarithmic strain rate dependence. For extrusion ratios larger than 10, Gibson and Ward found that a velocity limit is reached with increasing extrusion pressure where the velocity is independent of the extrusion pressure. Since the controlled extrusion velocities (0.25 to 3 mm min^{-1}) and the scale of the extrusion process (die exit diameter = 2.51 mm) were small, the extrusion process was assumed to take place under essentially isothermal conditions. The effect of scale on the deformational heating on the extrusion process has been discussed in detail by Gibson and Ward [13]. The velocity of rapid extrusions although not measured was high as has been mentioned. The possibility of such extrusions occuring under adiabatic conditions cannot therefore be ignored.

Calculations of the effect of the process variables on the extrusion pressure have been carried out using Equation 5 together with the flow stress expression obtained from Equations 9 and 12. The values of the pressure coefficient, w, was assumed to be equal to 0.094 as measured by Mears *et al.* [19] on HDPE and the coefficient of friction, μ, was taken to be 0.02. The values of $\beta = 0.75$ and $s + m \ln \dot{\epsilon} = 21.0$ MPa ($\dot{\epsilon} = 6.67 \times 10^{-4}$ sec^{-1}) obtained from the tensile tests were used, with m being the adjustable parameter.

The computed curves along with the experimental points assuming a value of $m = 2.6$ are shown in Figs. 2, 3, and 5. Excellent agreement between the computed and experimental extrusion pressures were obtained for the die angle of 20° and extrusion ratios of 5 and 6.75 over the range of velocities measured. However, the extrusion pressures for $R = 9$ is considerably higher than the computed values. The reason for this discrepancy is not clear.

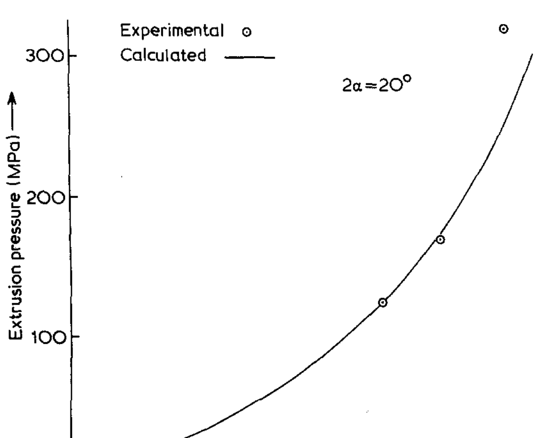

Figure 5 Relationship between extrusion pressure and logarithm of extrusion ratio.

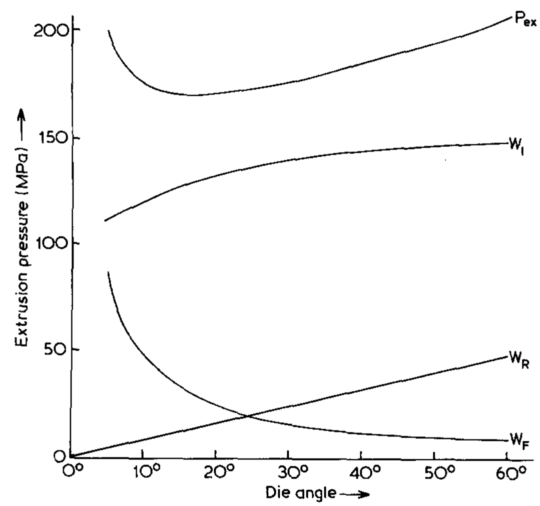

Figure 6 The calculated curves for P_{ex}, W_I, W_R and W_F as functions of die angle for an extrusion ratio of 6.75 and an extrudate velocity of 1 mm min^{-1}.

The computed extrusion pressure-die angle curve for the extrusion ratio of 6.75 and an extrudate velocity of 1 mm min^{-1} gives the optimum die angle equal to 16°. For a given set of extrusion conditions, the value of the coefficient of friction determines the value of the optimum die angle [15]. Since the calculated and experimental optimum die angles are similar, the value of $\mu = 0.02$ selected appears to be a reasonable assumption. This value of μ is within the range of Makinouchi's [35] experimental measurements for plastically deforming polyethylene and steel using different fluids as lubricants.

In Fig. 6 the calculated contributions to the extrusion pressure (P_{ex}) for an extrusion ratio of 6.75 and an extrudate velocity of 1 mm min^{-1} arising from internal deformation (W_I), redundant work (W_R) and friction (W_F) are plotted as functions of die angle. The value of μ assumed was 0.02. The contribution of W_R and W_F for increasing die angle to the extrusion pressure is found to be similar to that which has been calculated for non-strain hardening metals [15]. However, unlike metals, where W_I remains constant for increasing 2α [15], the contribution of the work of internal deformation to the extrusion pressure is found to increase with increasing die angle for the extrusion of a polymer because of dependence of the flow stress on the increase in strain and strain rate with increasing die angle [15]. Calculations of the effect of velocity on the individual terms indicate that the decrease in slope of the pressure–velocity curves with increasing die angle is due to the redundant work term.

5.3. Effect of pressure media

The effect of the pressure fluid on the extrusion pressure required for rapid extrusion indicates that lower coefficients of friction are associated with pressure fluids of higher viscosities. A similar result has been obtained by Nakyama and Kanetsuna [8] who found that extrusion pressures using castor oil and glycerine as the pressure fluids were lower than those using water and ethanol. In the hydrostatic extrusion process the pressure medium is dragged along the billet–die interface, as evidenced by the fact that the product emerging from the die is invariably covered with a thin layer of fluid. Hence, it is apparent that the frictional conditions existing at the billet–die interface and extrusion pressure are influenced by the pressure fluid used.

The observation that the coating of the billets with graphite lubricant had little effect on the extrusion pressure for the three fluids used suggests the possibility of a continuous fluid film separating the billet from the die as occurs with hydrodynamic lubrication. The sliding friction measurements of Makinouchi [35] indicate that graphite acts as a most effective lubricant between deforming polyethylene, having lowest coefficient of friction of a number of lubricants tested, including silicone oil. The adhesion of the graphite powder to the polymer billet was not affected by the pressure fluids as the extrudates were found to be uniformly coated by the graphite. If a continuous fluid film separates the graphite coated billet from the die then the graphite would have no effect on the frictional stress. The results of analyses [36, 37] of hydrodynamic lubrication during hydrostatic extrusion for constant flow stress materials show that hydrodynamic lubrication is dependent on establishing a sufficiently thick fluid film between the billet and die. Large film thickness are associated with high extrusion velocities and small die angles, such as used in these tests, as well as high fluid viscosities.

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