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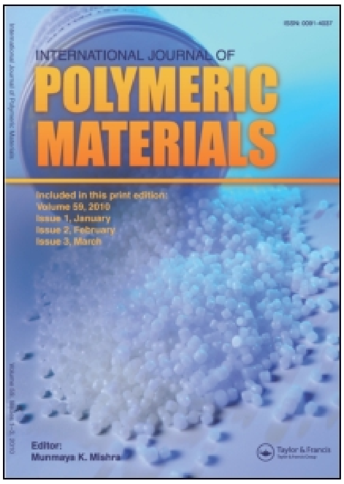

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Plastic Deformation of High Density Polyethylene in Solid State Extrusion

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An equation describing the process of solid state extrusion of high density polyethylene through a conical die with a taper of 20° was obtained by extending Sachs' equation to apply for polymeric materials which show considerable strain hardening. It was used to estimate the extruding pressure on the basis of the tensile data of the polymer. The agreement between observed and calculated values was satisfactory. Extruded polymers obtained at relatively low extrusion temperature, as low as 80°C, showed elastic after-effects, such as shrinkage at elevated temperatures. However, those extruded at relatively high temperatures, as high as 100°–120°C, showed extraordinary stability in their dimensions even at the temperatures as high as 120°C. Discussions on the basis of the slip line analysis were also made to interpret the discrepancies found between the calculated and observed results when the entrance angle of the die was increased.

1 INTRODUCTION

There have been some efforts to process polymers in the solid state.[1–5] However most of them were not successful. The materials were broken during deformation, or the products showed a tendency to restore their original shapes, especially when they were processed at room temperatures. These effects were understood to be due to the fact that the deformation at room temperatures was not perfectly plastic but that the elastic strain remained. The polymers of medium or high crystallinity, such as high density polyethylene,[4] isotactic polypropylene[6] or nylon 6[7] did not show these effects and deformation proceeded plastically, when the polymers were processed at relatively high temperatures such as from 80°C below the melting point up to 20°C below it. Thus, to deform the materials plastically and remove elastic deformation as far as possible is the prime requisite for the success in solid state processing of polymers.

The concept of "plasticity" is well understood in the field of metal processing.[8,9] Polymers, on the other hand, have been studied as viscoelastic bodies. The concept of the plasticity of macromolecules is not fully understood. What is the condition to deform the polymers plastically? Is it possible to deform the long chain polymers plastically by making each molecule slip away from elastic constraints? Or, what is the correlation between the plastic and viscoelastic nature of polymers? These are the problems to be solved in connection with the problem of how to succeed in the solid state processing of polymers.

Several processes used in the field of metals seem to be applicable to polymers. Deep drawing[1–3] and extrusion[1,2,4,5] have actually been applied to polymers. Deep drawing seems to be the most practical method. The process of deformation in this method is complicated and it seems to be quite difficult to be treated as a stationary state process. On the other hand, the method of extrusion seems less useful from the practical point of view. The advantage of the process of extrusion, however, lies in the simplicity of the deformation process. The stationary state extrusion can be realized at constant extrusion pressures.

Analysis of the extrusion process of plastic materials was attempted using different approaches. Some of them[8,9,13] have given fairly good results for metal extrusion with considerable preciseness: (1) the equations, such as Sachs' equation,[20] have been derived by assuming a simplified schema of stress distribution and pressure balance, and given estimations of extrusion pressures on the stress-strain characteristics of the material by using a hypothetical value of frictional coefficient, (2) slip line analysis[8,13] gives a complete stress and strain distribution by assuming a constant value of yield stress over a whole range of strain, of which the method is limited to the cases of plane extrusion, and (3) the upper and lower boundary theorems have an intermediate character between the methods 1 and 2.

In the present paper, we will derive first a generalized equation which will belong to approach 1. In the cases of polymeric materials the equation must take into account the remarkable effects of strain hardening. The equation will be examined in comparison with the observed extrusion processes by taking account of the stress–strain behaviour of the polymer. Discussions of stress and strain distributions in the material will also be made by analogy to the results of slip line analysis of plane extrusion. The two methods might be useful in a complementary way as in the cases of metal extrusion.

Preliminary report on super-structures of the extrudates has been published[4] and a more detailed report on the change of the super structure during deformation process will be made in the near future.[10]

2 EQUATION OF EXTRUSION PRESSURE

2.1 Equation based on Sachs' pressure balance schema

In the case of metal extrusion, Sachs' equation[8,15] gives a good estimation of extrusion pressure. The equation was deduced on the basis of the assumption that the material is purely plastic. Figure 1 shows the examples of true stress vs. true strain relationship of high density polyethylene. As shown in Figure 1, true stress of the polymer grows rapidly and seems to tend to infinity at a certain finite value of strain.[19] This feature differs from that of metals which is usually described by a simple formula of stress expressed by a power of strain with an exponent of rational number. In such cases, the stress will always take a finite value as far as the strain remains in finite values. Thus, the effect of strain hardening has a decisive importance to describe the deformation process characteristic of polymers. In such situations, it is not adequate to assign a constant value or an average value to the yield value all over the strain regions as made in Sachs' hypothesis. The yield stress of polymeric materials is rather adequately considered to be a function of true strain, and it increases towards the infinite value at a certain finite value of strain, as shown in our previous paper.[19] An extension of Sachs' equation thus is needed for the extrusion of solid polymers, which will be mentioned in the following part of the section.

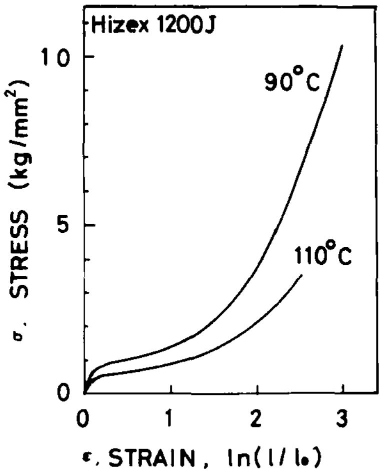

FIGURE 1 True stress vs. true strain curves of high density polyethylene.

Figure 2 shows the longitudinal section of the material in the die. The simplified pressure balance is represented by Eq. (1) referring to Figure 2.

$$\pi(r + dr)^2 (\sigma + d\sigma) = \pi r^2\sigma + 2\pi r\mu\sigma_r \frac{dr}{\sin\alpha} \cos\alpha + 2\pi r\sigma_r \frac{dr}{\sin\alpha} \sin\alpha \qquad (1)$$

where μ is the frictional coefficient between the die wall and the polymer, α is the aperture angle of cone-shaped die hole, σ and σ_r are the stresses normal to a cross section and die wall, respectively, and r is the radius of the die hole at the cross section. Tresca's yield criterion,[8] which gives the relation between two principal stresses, σ and σ_r, will be written by taking account of the strain dependence of the criterion as Eq. (2).

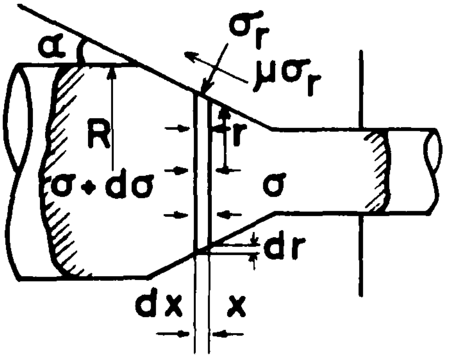

FIGURE 2 Pressure balance in conical portion of die.

$$Y(\epsilon) = \sigma(\epsilon) - \sigma_r(\epsilon) \qquad (2)$$

Where ϵ is the true strain, which is defined by Eqs. (5) and (6). From Eqs. (1) and (2) a differential equation, Eq. (3), is obtained,

$$\frac{d\sigma(\epsilon)}{d\epsilon} = (1 + B)Y(\epsilon) - B(\epsilon) \qquad (3)$$

where B is μ cos α. By integrating Eq. (3) with Laplace transformation or according to the conventional integration formula of linear differential equation of first degree, Eq. (4) is obtained.

$$\sigma_0 = (1 + B)\left(\frac{R}{r}\right)^{2B} \int_{2ln(R/r)}^{0} e^{-Bs}Y(2ln\frac{R}{r} - s)ds + \left(\frac{R}{r}\right)^{2B}\sigma(\epsilon) \qquad (4)$$

where σ_0 denotes the extrusion pressure at the entrance of the die hole, and $\sigma(\epsilon)$ is the additional stress at the exit of the die hole.

According to the criterion of Eq. (2), the value of yield stress, $Y(\epsilon)$, equals to that of the largest principal stress, σ_1, when the other principal stress is equal to zero, $\sigma_2 = \sigma_r = 0$. Admitting this approximation, we can easily estimate the yield stress as a function of strain by using tensile data of the polymer. The constant μ is introduced in Eq. (1) as the frictional coefficient between the polymer and the steel wall. It is difficult, however, to make direct measurements of frictional coefficient under the conditions at which the actual extrusion is conducted. In this study, the value of μ will be estimated by adjusting its value to give the best fit of the calculated extrusion pressure to the observed one.

2.2 Slip line analysis

Pressure balance schema as shown in Figure 2, being formulated by Eq. (1), is quite simple, since it is not considered to be realistic that the frictional force and reaction force act only at the wall surface and give no influence on deformation process in the die and that the boundary of plastic and rigid body is assumed to end by a simple flat plane at the exit. Real stress field and the resulting velocity distribution might be, therefore, more complicated. In such situations, it might be effective to deduce the detailed stress and strain distribution through the slip line analysis. However, it is only possible for the hypothetical cases that the materials which show no strain hardening are extruded in a flat plane instead of rod. Nevertheless, the results of this analysis might be useful to imagine the stress and strain distribution in the real polymeric materials being extruded through a conical die. The analysis can be conducted by following the descriptions of the standard books.[8,13] Analysis of stress fields and resulting velocity distributions for the materials which have frictional coefficients as high as those of polymeric materials has not yet been published. We tried the analysis with the values of frictional coefficients around 0.23. The calculation was conducted on the computer of Fuji-Tsushin Co., FACOM 230–60, installed in West Japan Computer Center at Kyushu University, as one of the experimental uses of the Time Sharing System. The methods and process of analysis will not be mentioned here as they are detailed in the books cited above. Therefore, we will cite only the results of calculation for the purpose of discussion.

3 EXPERIMENTAL

3.1 Sample

High density polyethylene used was Hizex 1200J, manufactured by Mitsui Petrochemical Co., Ltd. Polyethylene pellets were molded into the shape of a disk of 15 mm thick and 100 mm in diameter. Billets of 10 mm in diameter and about 50 mm in length were cut out from the disk. The densities of the billets were about 0.962 g/cm^3, and this corresponds to a crystallinity of ca. 71 %.

3.2 Extrusion device

Figure 3 shows a longitudinal section of the extrusion device. The liner diameter of the cylinder in which the billet is inserted, is $2R = 10$ mm. The piston fits the cylinder with clearances of 0.3–0.5 mm. A Viton (Kel-F) O-ring and Teflon back-up ring were set at the top of the piston to prevent the leaking of polymer.

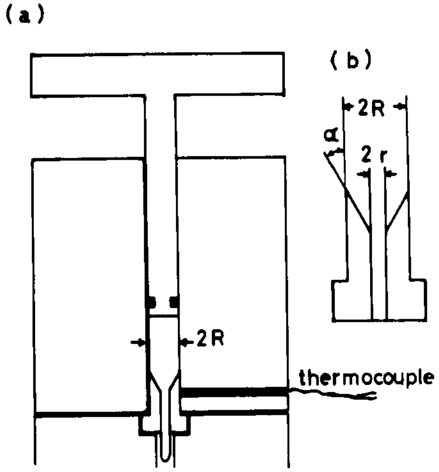

FIGURE 3 Longitudinal section of (a) extrusion device, and (b) die.

Figure 3(b) shows the longitudinal section of the die piece. The outer surface of the die piece was fitted to the cylinder, and the aperture angle was measured from the cylinder wall to the inner wall of the cone-shaped die hole. The angle α was selected among the values of 10°–60°. The diameter of the exit hole, ($2r$), was selected among the values of 2.0–4.0 mm.

The device was pressed by a hydraulic oil press. The cylinder was heated by a ribbon heater wound around it and the upper and lower hot plates of the press.

Forces exerted on the top of the piston were evaluated by reading the indication of a small oil gauge attached to the oil press and multiplying it by a factor previously determined by using strain gauge of full scale 5 tons.

Extrusion temperatures were measured with a thermocouple inserted into a hole drilled into the cylinder wall to attach the outer surface of the die piece.

3.3 Extrusion of the polymer

Silicone vacuum grease (Dow Corning Co., Ltd.) was used as a lubricant. The billet was kept in the device at the extrusion temperature for 1 hour before the beginning of extrusion. The pressure was raised rapidly up to the scheduled constant value and kept there by moving a hand pump lever to keep the indicator of the pressure gauge fixed at a constant value. The deviations of extrusion pressure from the average value during one shot of the experiment was less than 50 kg/cm^2. The quantity of the extrudate was evaluated by measuring the length of the extrudate which was extruded out of the die exit.

3.4 Evaluation of degree of deformation

There are some confusions in defining the degree of deformation of extrudate or more generally those of the products obtained through solid state deformation. Here we also will use several kinds of expression to denote the degree of deformation of extrudate as follows.

1) *By the use of the exit diameter of the die.* The diameter of the entrance of the die hole, which is equal to the diameter of the cylinder and also to the diameter of the original billets, is always taken as $2R = 10$ mm in this study. Therefore the ratio of the diameter of the exit and inlet hole of the die, $2r$ to $2R$, represents the degree of the deformation of the extrudate, when the diameters of the extrudates are not so different from that of the die exit, as was the case of high density polyethylene.

2) *By the use of the cross sectional ratio.* The ratio of the cross sectional area of the rod-shaped original billet, πR^2, and that of the extrudate, πr^2, is a good expression of the degree of deformation of extrudate, because it is comparable to the most conventionally used "draw ratio" of the drawn sample, when the sample volume is assumed to be constant throughout the deformation process. This expression may be substituted with more practical parameters of the ratio of πR^2 to that of the die exit, πr^2, when the diameters of the extrudates were not so different from those of the die exits. We will call this value as "Cross Sectional Ratio".

3) *By the use of the true strain.* The true strain is defined by Eq. (5)

$$\epsilon = \int_{l_0}^{l} \frac{dl}{l} = ln \frac{l}{l_0} \tag{5}$$

Under the assumption of constant volume and no die swell, we obtain another expression of ϵ as follows.

$$\epsilon = -\int_{r_0}^{r} \frac{dr}{r} = 2\,ln\,\frac{R}{r} \tag{6}$$

3.4 Measurements of stress–strain curve and shrinkage

The stress–strain measurements were made on thin sheets of 0.5 mm thick which were cut out from the extrudate or original billets. The effective sample length was 20 mm and the rate of stretching was 5 mm/min.

Shrinkage of extruded and drawn samples was measured with Vibron DDV–II made by Toyo-Measuring Co. Ltd., by applying small stress less than 10^6 dyne/cm^2 or so.

4 RESULTS

Figure 4(A) and (B) show the processes of extrusion $2r = 10$ mm and taper $\alpha = 20°$ at constant extruding pressures indicated in the figure. As the outset, extrusion proceeds rapidly, then after a few minutes the rate goes down to a steady state rate of extrusion. The greater the extruding pressure the faster the rate of steady state extrusion.

Figure 5(A) shows the steady state rates of extrusion conducted at 110°C, and Figure 5(B) at 90°C, by using die pieces of different exit diameters. As shown in Figure 5, a steep rise of steady state rates occurs at a certain value of pressure which depends on exit diameters. The rate grows remarkably in a narrow range of pressure values and below that the rate was negligibly slow. Exceptions for this general tendency were observed in the cases of extrusion with the die of extremely narrow exit such as $2r = 2.0$ mm. In those cases the slopes of steady state rate were not so steep. The pressure below which no remarkable extrusion is observed and above which steady state extrusion rate is remarkable will be called hereafter "extrusion pressure". And the term "extruding pressure" will be used for pressures applied on the piston without considering whether remarkable extrusion is observed or not. In Figure 6 extrusion pressures thus defined were plotted against degree of processing expressed in terms of true strain or $2\,ln(R/r)$.

Figure 1 is the stress–strain data of the polymer at 90°C and 110°C, which were plotted on true stress and true strain axes.

The structure and properties of solid extrudate of linear polyethylene thus obtained were reported in our previous paper.[4] For example, the extrudate obtained by using a die of $2r = 2.5$ mm, i.e. $(R/r)^2 = 16$, at 100°C showed a remarkable stability even at temperatures as high as 120°–125°C. The tensile modulus of the extrudate was about 3×10^{11} dyne/cm^2 at the stretching

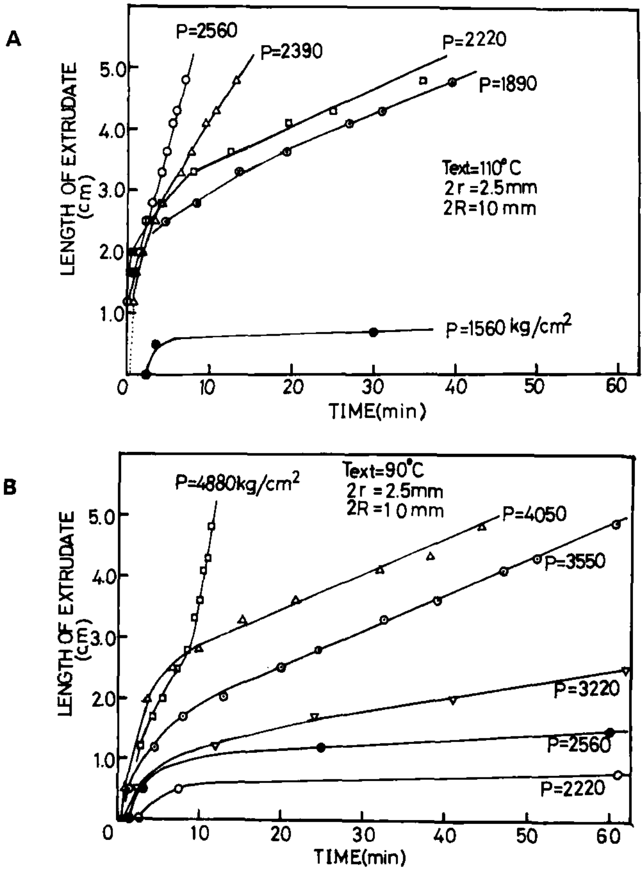

FIGURE 4 Length of extrudate in progress of extrusion at (A) 110°C, and (B) 90°C.

velocity of 5 mm/min for 20 mm original length at room temperature, being evaluated by initial slope of stress–strain curve.

Figure 7 shows extrusion pressure observed in extrusion of high density polyethylene at the temperatures of 90° and 110°C with the dies of entrance angles $\alpha = 10°–60°$ and the exit diameters $2r = 3.2$ and 2.6 mm. The extrusion pressure was increased gradually with increasing angle above 20°, whereas it seems to increase with decreasing below 20°–10°. Thus, the optimum entrance angle with which the extrusion was conducted at the smallest extrusion pressure was found between $\alpha = 10°–20°$.

Figure 8 shows distortion of a grid, which was originally marked in parallel to make square blocks. They were observed after the extrusion through a conical die of entrance angle $\alpha = 30°$ with cross sectional ratio $(R/r)^2 = 10$.

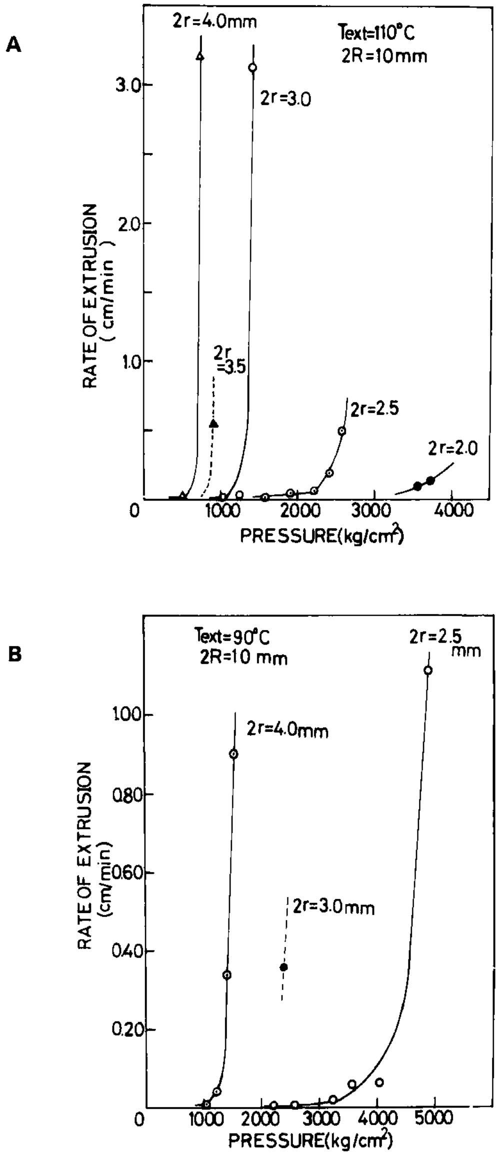

FIGURE 5 Steady state velocity of extrusion vs. extruding pressure at (A) 110°C, and (B) 90°C.

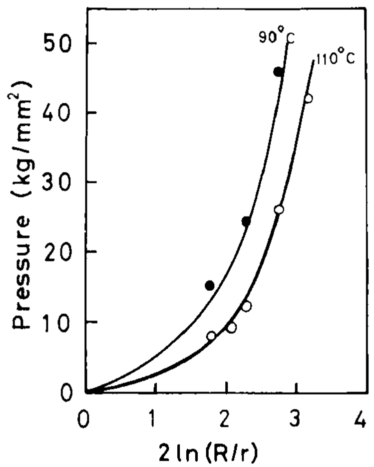

Figure 6 Observed extrusion pressures at 110°C (open circles), and 90°C (black circles) and calculated values (solid lines) with the value of $\mu=0.23$.

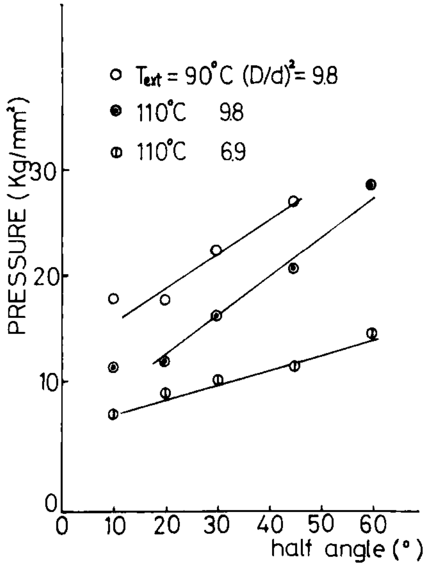

FIGURE 7 Extrusion pressure vs. entrance angle α.

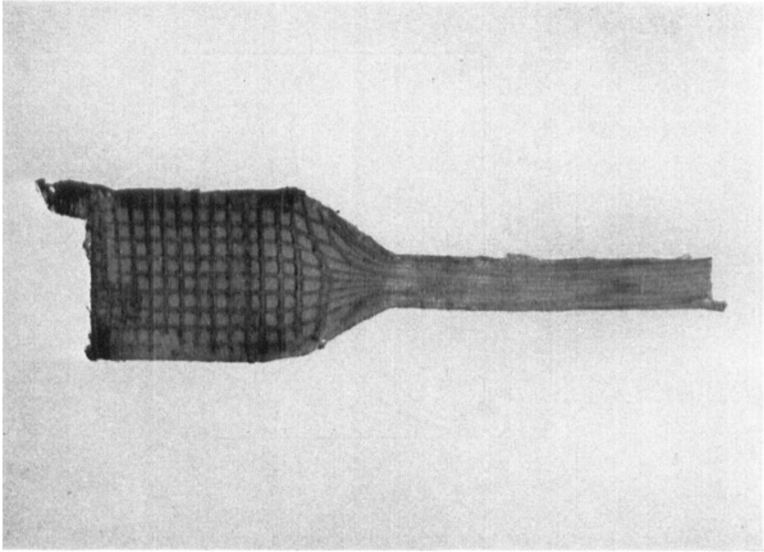

FIGURE 8 Distortion of square grid.

5 DISCUSSION

5.1 Analysis of extrusion data on the basis of tensile data

The extrusion pressure vs. true strain represented by 2 *ln* (R/r) curves are shown in Figure 6 with solid lines which were calculated by Eq. (4), by using the stress–strain data given in Figure 1. In this calculation a frictional coefficient μ was assumed to be 0.23 for both cases of extrusions at 90° and 110°C and the additional term of Eq. (4), the resistance against extrusion at the exit of the die was neglected. The agreement between the calculated and observed values is quite good. And the value of frictional coefficient assumed in the calculation is in the range of those of values reported in the literature[6] as the coefficients of the polymer and steel wall.

In the course of the extrusion, the pressure as high as 2 *k* bar or more was exerted on the material. According to the recent papers[17,18] the yield stress of polymers under hydrostatic pressure increases with increasing pressure. The deviations of the yield stress from the value at normal pressure are not negligible at the pressures employed in our extrusion experiment. When we take account of this fact, the agreement between the calculated and observed values shown in Figure 6 is unexpectedly good. The main reason of this agreement might be due to the nature of Eq. (4). In Eq. (4), the stress at large strain contributes more effectively to the extrusion pressure than that at small strain. In the case of high polymers, the effect of strain hardening is considered to be due to the origins different from the cases of metal deformation. The stress of polymeric materials at large deformation is due to the contracting force of largely extended molecular chains. In such a case the stress does not seem to be affected by hydrostatic pressure of the medium surrounding the polymer molecules at large deformation. The fact that the extrusion experiments were conducted at the elevated temperatures might be the second possible explanation of the results in Figure 6. Tensile data at elevated temperatures under high pressure have not yet been reported. The possibility of this explanation still remained in question. Third possible explanation is that the values of frictional coefficient might decrease with increasing pressures and this effect will cancel the effect of increased yield stress under high pressure.

To ascertain these possible explanations, measurements of true stresses as a function of true strain under high pressure and at elevated temperatures are necessary. Since such measurements are quite difficult to achieve, especially in the case of the sample which shows the necking phenomena, extrusion experiments with back pressure at elevated temperatures will be useful, instead of simple tensile testing under high pressure, to get some knowledge concerning the effect of hydrostatic compression on the deformation properties of macromolecular materials.

5.2 Effect of die angle

The increase of extrusion pressure with increasing die entrance angle as shown in Figure 7 cannot be explained by the use of a constant μ value in Eq. (4). Equation (4) predicts that the increase of die angle makes the decrease of the extrusion pressure, as far as μ is independent of the die angle. Distortion of the square grid after extrusion, shown in Figure 8, revealed a complicated strain distribution which was not forecasted in the preliminary assumption of pressure balance schema given in Figure 2. This could be the possible explanation for the effect of die angle as seen in Figure 7. To inspect this explanation, we conducted the slip line analysis after Hill.[8] Figure 9 shows the slip lines calculated for the plane extrusion through a slit die of the taper angle $\alpha = 30°$ with the frictional coefficient $\mu = 0.23$. As in usual slip line analyses the value of yield stress was assumed to be constant throughout the whole extrusion process. As shown in Figure 9 the frictional force at the wall surface exceeds the assumed yield stress of the material at the point indicated by the arrow. The point indicated by an arrow in Figure 9 corresponds with the point where $\beta = 0$, here β is the angle between the wall normal and the slip line intersecting wall surface at that point. At the high pressure side of

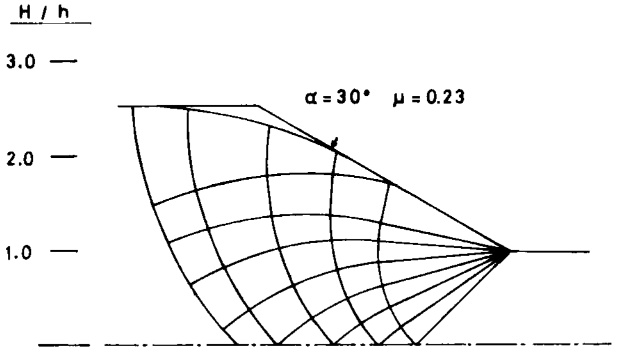

FIGURE 9 Slip lines for plane extrusion with the die of α = 30°.

this point (left side of the point) the material is adhered to the wall and immobilized. And it seems that the materials slip along the slip line which starts at the point of $\beta = 0$ toward the high pressure side and this slip line forms a natural boundary between the moving and dead polymer layer. By using this slip line field, a distribution of strain velocity in the die is obtained. By integrating the velocities, the map of distorted square grid in the die during the process of the extrusion was calculated. Figure 10 shows the calculated result. The result reproduces the observation of Figure 8 fairly well. By integrating the force along the wall, extrusion pressure was estimated on the basis of the slip line field of Figure 9. Figure 11 gives the extrusion pressures divided by the half value of yield stress, $k = Y/2$, which are plotted against the entrance angle α. As Figure 11 shows, the extrusion pressure increases with increasing α for different values of thickness ratio H/h, where H and h are the thicknesses of original sheet and extruded one, respectively. The results shown in Figure 11 correspond well with the experimental results obtained by the use of the

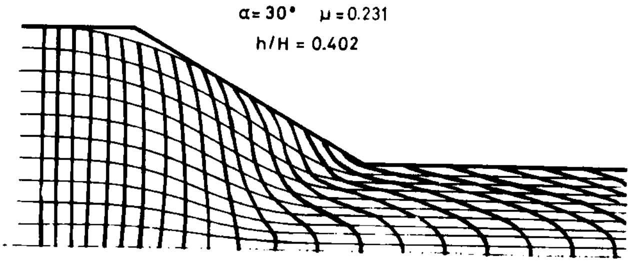

FIGURE 10 Distorted square grid calculated on the basis of slip line field of Figure 9.

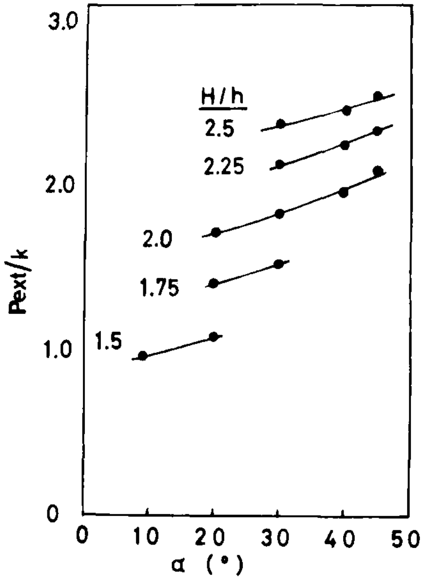

FIGURE 11 Calculated values of extrusion pressures vs. entrance angle.

conical dies as shown in Figure 7. The deviations of Eq. (4) from the observed value with increasing entrance angle were understood as due to the over simplification of the pressure balance of Figure 2.

In the present stage we have no methods to satisfy at the same time both the conditions of the strain hardening of the real polymeric materials, the realistic pressure balance, and the effect of hydrostatic pressure on the yield stress. Two methods used here conflict with each other in their basic assumption. However, these two different analyses confirm a simple but important conclusion that the deformation of solid polymers proceeds plastically while being extruded through the die.

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