

Unstable Flow of Molten Polymers: A Second Site of Melt Fracture

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INTRODUCTION

One type of instability in flow of molten polymers has already been described.¹⁻⁷ This instability was found to initiate at the inlet to a capillary and is thought to be the result of elastic failure of the viscoelastic melts. The phenomenon results in emerging polymer streams of grossly irregular shape. It is convenient to refer to this particular phenomenon as *inlet melt fracture* or *inlet fracture*, rather than by the previously used, more general term, *melt fracture*.

Instabilities in flow appear to occur at a site other than the capillary inlet for some samples of two types of polymers, linear polyethylene⁴ and copolymers of tetrafluoroethylene and hexafluoropropylene (TFE-HFP).¹⁸ The associated phenomena, their causes, and the relation to the earlier type of melt fracture are the subject of this paper.

DISCUSSION

Flow

The basic device used was a metal capillary viscometer or rheometer.¹ Pressure was applied by a piston, operating within the rheometer cylinder, activated by an external pneumatic cylinder. Flow rates were determined from the time for a given displacement of the piston.

Shear rate-shear stress data for linear polyethylene and the TFE-HFP copolymers are illustrated schematically in Figure 1 and given in Figures 2 and 3. The data in Figures 2 and 3 are uncorrected for entrance pressure losses and piston friction. Had such corrections been made, the lower parts of the curves corresponding to that on which *A* and *B* are indicated in Figure 1 would have been coincident. It is assumed in the following discussion that the lower sections of the curves are coincident as represented in Figure 1.

At shear stresses less than that at *A* in Figure 1, ordinary viscoelastic flow was observed: viscosity decreased with increasing stress or rate, and the emerging stream was smooth and regular. At and above the critical

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stress A but at stresses less than B , the emerging streams had a fine surface roughness similar to a close pitch screw thread (this has been described for linear polyethylene 4, 8). The fine-scale irregularity, along with the gross irregularity associated with inlet melt fracture, is illustrated in Figure 4 by specimens of linear and branched polyethylene, respectively. (It should be remarked that fine-scale surface irregularities have been noted with branched polyethylene⁴ in addition to the gross inlet fracture type of surface irregularity. The fine-scale roughness of branched polyethylene may or may not be due to that which causes the fine-scale roughness described in this paper, which initiates at a critical stress.)

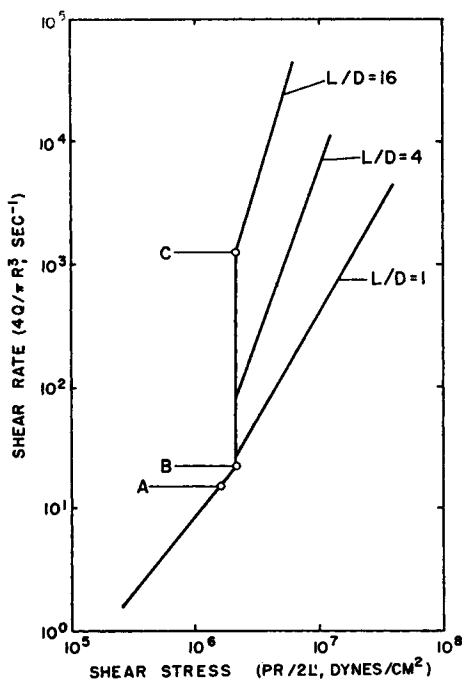


Fig. 1. Schematic flow data for linear polyethylene and TFE-HFP copolymers.

In the region A to B , Figure 1, the very first part of the emerging polymer streams had the fine-scale roughness. This contrasts with inlet fracture in which the gross irregularity of the emerging stream did not occur until emergence of polymer which had been in the inlet region at the time the stress was applied. (Application of the stress was nearly instantaneous. It was accomplished by quick release of a gate from the outlet of the capillary with the polymer at the desired pressure in the rheometer cylinder.)

The stress represented by B in Figure 1 was a second critical stress. At this stress the rate tended to be double-valued; that is, the rate at constant pressure in the rheometer oscillated between the rates corresponding to B and C in Figure 1. The magnitude of the higher rate was a function of

capillary length. For capillaries having a sixteen-fold length-to-diameter ratio ($16 L/D$) the higher rate was roughly tenfold larger than the lower rate. The rate difference, B to C , decreased with decreasing L/D ratio.

There were several unusual features of flow at rates corresponding to C in Figure 1. The most obvious was that of shape of the emerging streams. For the linear polyethylenes and the TFE-HFP copolymers used, the emerging streams were smooth and regular. As the flow rate oscillated between B and C , the specimens' surfaces changed accordingly, from rough to smooth (Fig. 5). The recurrence of smooth surfaces at rate C differs

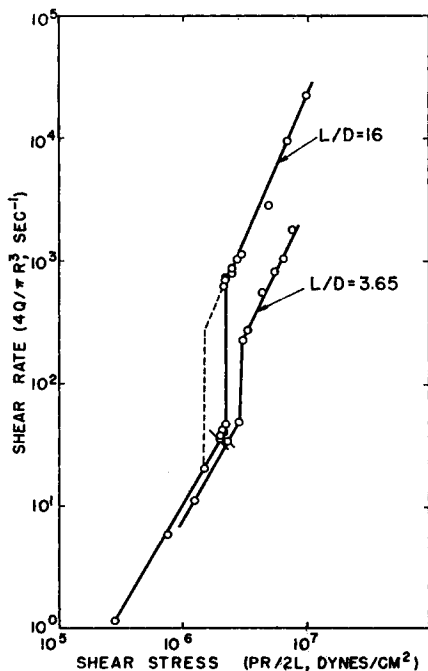


Fig. 2. Flow data for linear polyethylene, melt index 0.5, 0.958 density at 23°C ., at 150°C .: (---) "hysteresis loop" observed when stress is lowered from stresses above those at vertical section of curve.⁹

from the inlet type of fracture, for which increasing stress results in ever increasing irregularity.¹

In addition to the B -to- C type of rate variation noted above, there was a second type of nonuniform rate. The rate at C varied rapidly with time at a frequency of about 20/sec. A specimen and flow rate versus time record of a TFE-HFP copolymer is illustrated in Figure 5. Flow rate, as the velocity of piston motion, was recorded on a velocity meter and 1/100-sec. response time recorder. Figure 5 illustrates the low, relatively steady rate corresponding to B in Figure 1 and the 20-cycle/second oscillation of the high average rate, C . The lower of the rates during the 20-cycle/sec. oscillation was roughly that of B .

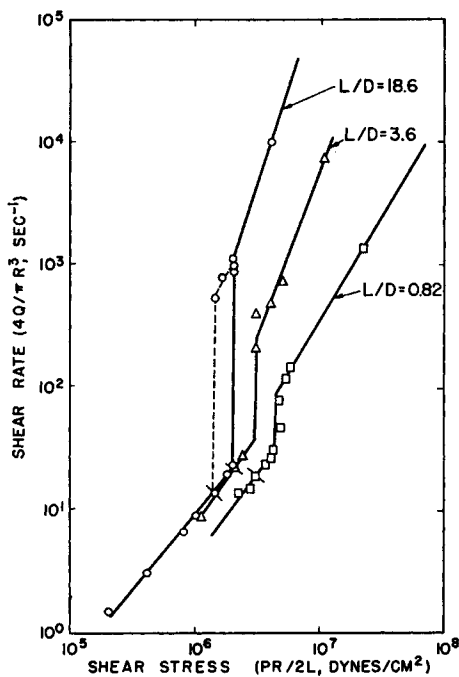


Fig. 3. Flow data for TFE-HFP copolymer at 380°C.: (---) "hysteresis loop" observed when stress is lowered from stresses above those at vertical section of curve.

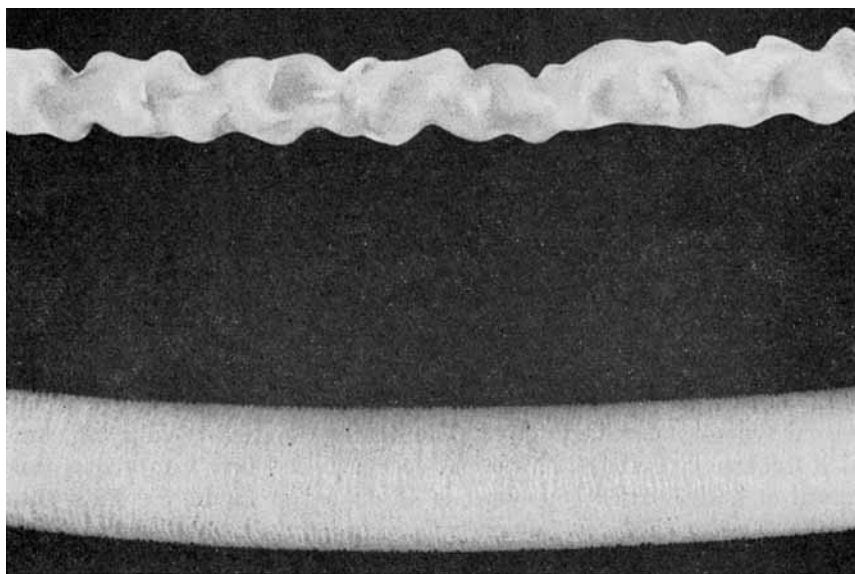


Fig. 4. Specimens illustrating large irregularity associated with inlet fracture (branched polyethylene) and the fine-scale surface irregularity (linear polyethylene). The specimens were obtained with capillaries of different radii.

The velocity meter was a Sanborn LV Syn device which consisted of a solenoid within which a permanent magnet was drawn by the rheometer piston. The resolution time of the device, about 1/1000 sec., was considerably faster than the full-scale deflection time of the recorder.

The flow curves at the critical stress B were not actually vertical. Over a stress range of about 5%, the average flow rate increased from that substantially at B to that at C . Within this 5% stress range the flow rates, averaged for a long time relative to the period of the B -to- C variation period, were between B and C . The fraction of the time of flow at rate B decreased from 1 to 0 over the 5% stress range. Conversely, the fractional time at rate C varied inversely from 0 to 1. Flow versus time curves from the high-speed velocity meter for sustained flow at the average rate C were continuous 20-cycle/sec. peaks.

A description of the dependence of critical phenomena on capillary geometry follows. The critical stresses corresponding to A and B in Figure 1 and the type of surface roughness of linear polyethylene and the

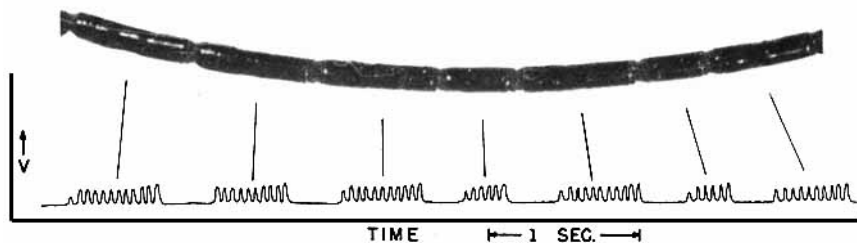


Fig. 5. Velocity as a function of time for flow oscillating between points B and C , Figure 1, for TFE-HFP copolymer at 380°C.: sections of the specimen are identified with corresponding velocity-time curve segments.

TFE-HFP copolymers were independent of (a) capillary diameter from 0.020 to 0.100 in. at a constant L/D ratio, (b) inlet geometries varying from flat or 180°-included angle entries to 20°-included angle cones, and (c) the materials from which capillaries were made, i.e., stainless steel (both polished and very rough), glass, graphite, and polytetrafluoroethylene which was used with linear polyethylene only.

One feature of flow in the graphite capillary was noteworthy. At rates corresponding to C in Figure 1, small flecks of graphite appeared on the surface of the emerging polymer stream.

The height and pitch length of the surface irregularity, but not the type of roughness, increased with increasing capillary diameter but were independent of L/D ratio. The critical rates at which oscillating flow began increased somewhat with decreasing L/D ratio. This is apparent in Figures 2 and 3, but is not indicated in Figure 1.

The magnitude of surface irregularity accompanying the earlier-described¹⁻⁷ inlet fracture increased with capillary diameter, decreasing L/D ratio, and increasing cone angle of the inlet.

The flow phenomena of linear polyethylene and TFE-HFP copolymers

suggest a different type of flow instability or a different site of incidence from that associated with inlet fracture. Specifically, the lack of dependence of the surface irregularity on the inlet taper or the L/D ratio of the capillaries and the immediate appearance of the roughness on application of stress suggest that the phenomena initiate in the straight section or land of a capillary or die. Initiation at the capillary outlet by some other mechanism is another possibility. The sudden yielding at the second critical stress and the 20-cycle/sec. oscillating rate suggest stick-slip flow along the upper branch of the flow curves. Slip at the capillary surface was strongly indicated by the graphite flecks on specimen surfaces.

Flow Birefringence

Some insight into the site and cause of the above flow instabilities was provided by observation of flow birefringence. Flow was observed through glass capillary assemblies attached to the bottom of the metal rheometer.

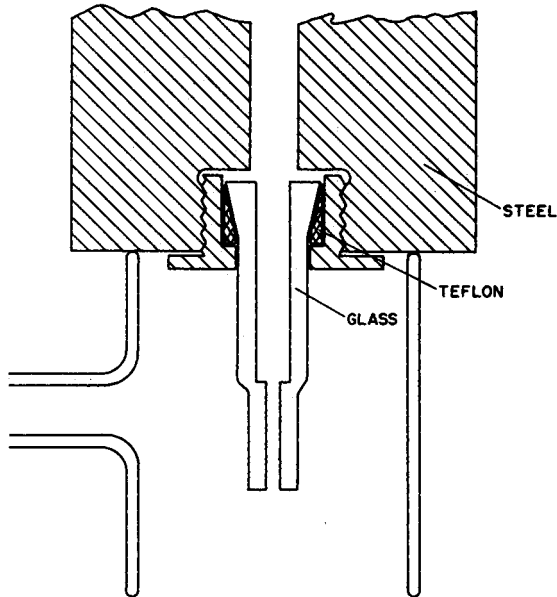


Fig. 6. Schematic drawing of attachment of glass tube and capillary to bottom of rheometer cylinder.

Capillaries were sealed to short sections of $1/4$ -in. I.D. hard-glass pipe. The standard tapered pipe ends were supported by a cushion of Teflon TFE fluorocarbon resin, as shown in Figure 6. Hot air flowing in the outer glass tube heated the capillary. The capillaries were backlighted by an intense incandescent light and observed or photographed between crossed polaroids. Photography on black and white film was done through a red "color separation" filter. Light and dark bands or isochromes similar to those obtained with monochromatic illumination resulted,

while the convenience of incandescent lighting was retained. (In full color, the appearance of the birefringence colors¹² was striking.)

The flow of branched polyethylene is illustrated in Figures 7 and 8. The contours of the isochromes are characteristic of occurrence of inlet fracture. The isochromes tend to form closed loops, with the top of the loop in the inlet and the bottom in the capillary. A maximum in birefringence occurs at the inlet. The number of isochromes at the inlet was found to be approximately linear with stress with an intercept on the stress axis at about 0.6×10^6 dynes/cm.². Inlet fracture, which was first apparent at about 3.0×10^4 dynes/cm.², is quite obvious at the highest stress in

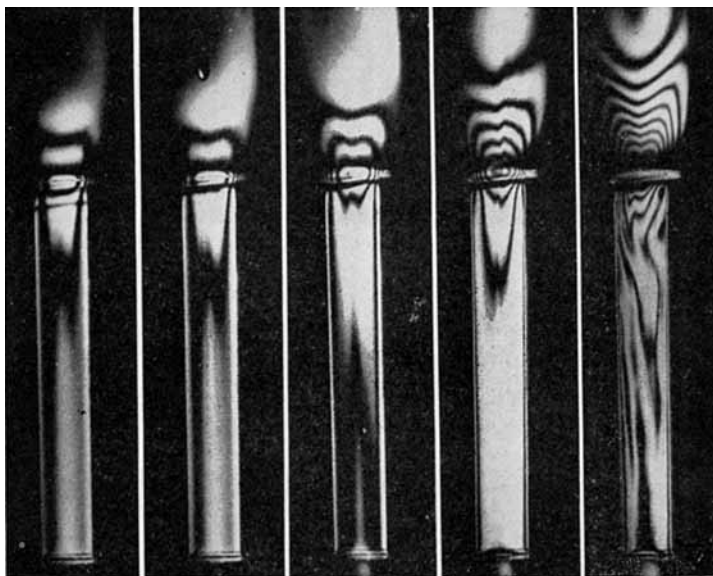


Fig. 7. Photographs through crossed polaroids of flow of branched polyethylene, melt index 2.0, 0.918 density at 23°C., in 1.0-in.-long 0.10-in.-diameter capillary having a flat inlet. That which appears as a horizontal disc at the inlet is an optical effect resulting from sealing the capillary to the glass pipe: stresses from left to right are, respectively, 0.9, 1.2, 1.8, 3.0, and 3.6, $\times 10^6$ dynes/cm.².

Figure 7. In Figure 8 at the highest stress, fracture resulted only in slight dissymmetry of the lowest isochrome in the capillary. Inlet fracture effects decrease substantially in magnitude with decreasing inlet angle.⁷

It appears reasonable to equate birefringence with elastic strain.¹⁰ If this is assumed, the following conclusions may be drawn for polymers which undergo inlet fracture. Elastic strain first increases and then decreases with increasing amounts of shear: the birefringence increases to a maximum in the inlet and decreases in the capillary. Inlet fracture occurs at the site of maximum elastic strain, i.e., at the inlet.

Flow birefringence of linear polyethylene, illustrated in Figures 9 and 10, was similar to that of branched polyethylene in that the birefringence in-

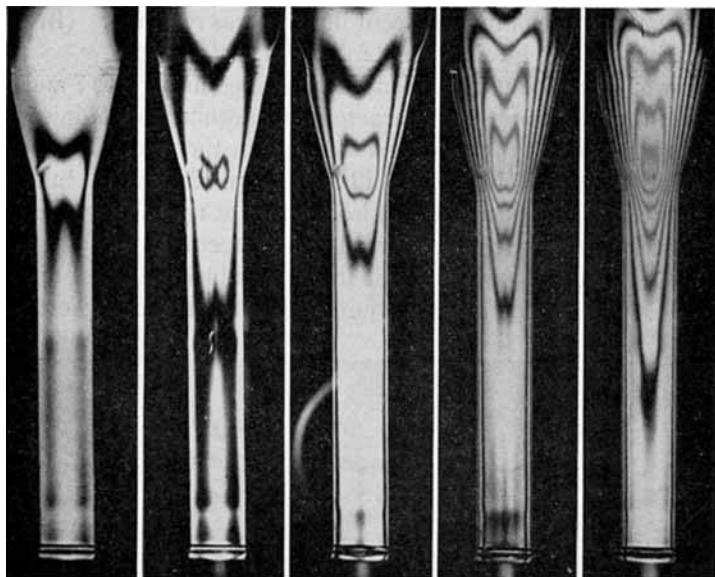


Fig. 8. Same as Figure 7 except that the inlet to the capillary is a 20°-included angle conc.

creased linearly with stress, but it increases at a lower rate. In other respects the birefringence patterns were quite unlike that of the blanched polyethylene. At the lower stresses and isochromes are quite uniform down the length of the capillary. The uniformity with capillary length indicates that elastic strain is independent of the amount of shear and is a function of

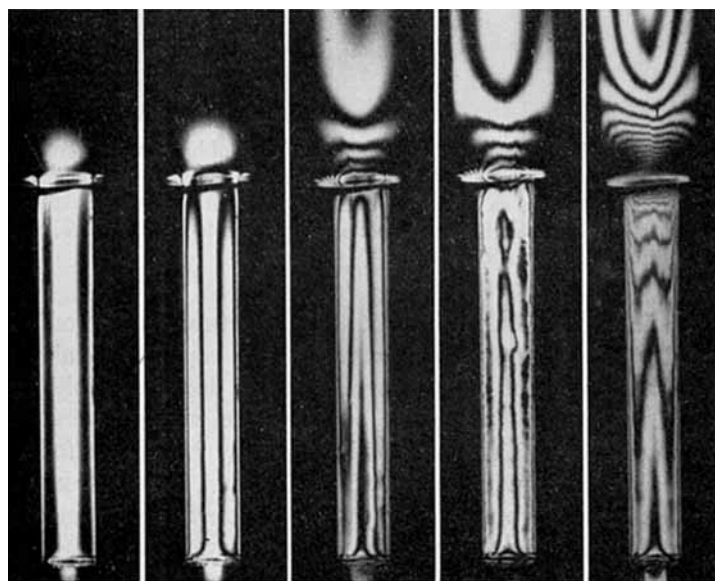


Fig. 9. Photographs through crossed polaroids of flow of linear polyethylene, melt index 0.5, 0.958 density (at 23°C.), at 150°C. in same capillary as used for Figure 7: stresses from left to right are, respectively, 0.9, 1.2, 3.0, 3.6, and 4.2, $\times 10^6$ dynes/cm.².

the shear stress or shear rate only. Linear polyethylene and the TFE-HFP copolymers are less complex in this respect than polymers in which inlet fracture occurs.

Successive isochromes appear at a distance from the center of the capillary of about seven-tenths of the capillary radius. This distance corresponds to the distance from the center, where the birefringence or retardation in a polymer flowing through a circular capillary observed perpendicular to the capillary axis is a maximum.

The distance from the axis at which the maximum retardation should occur may be estimated by assuming a linear dependence of birefringence on stress. Then, the retardation (ϕ) at any distance from the origin (x) is

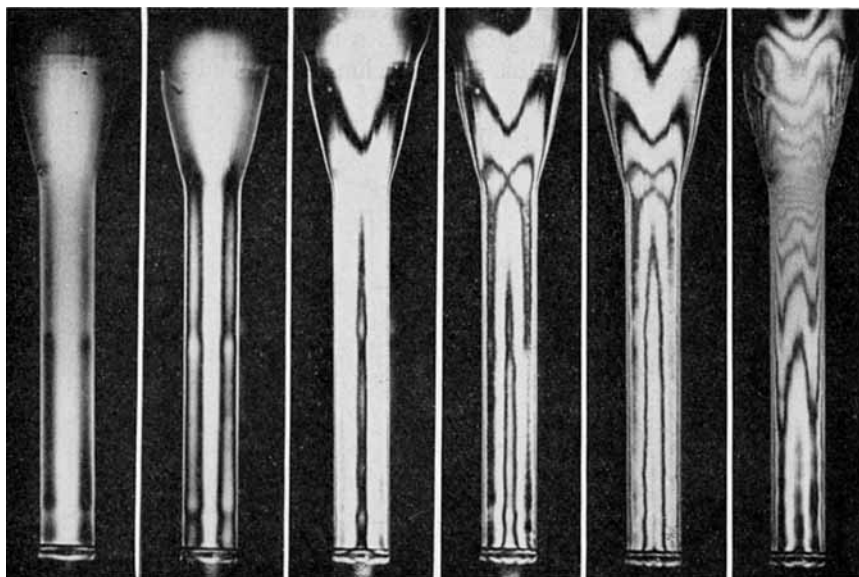


Fig. 10. Same as Figure 9 except that the inlet to the capillary is a 20° -included angle cone. The stresses are, respectively, 0.9, 1.2, 1.8, 3.0, 3.6, and 4.8, $\times 10^6$ dynes/cm.².

proportional to the sum of the products of differential thickness dt , times the stress at the radius $r(\sigma)$, corresponding to the location of element dt ; see Figure 11.

$$\phi = K \int_{-t}^{+t} \sigma_r dt$$

The stress at r is

$$\sigma_r = \sigma/R$$

where σ is the maximum stress at $r = R$, the radius of the capillary, and the radius r is

Substituting,

$$\begin{aligned}
 \phi &= K \frac{\sigma}{R} \int_{-t(x)}^{+t(x)} (t^2 + x^2)^{1/2} dt \\
 &= \frac{K\sigma}{2R} [t(t^2 + x^2)^{1/2} + x^2 \log (t + (t^2 + x^2)^{1/2})]_{-t}^{+t} \\
 &= \frac{K\sigma}{2R} \left[2tR + x^2 \log \left(\frac{R+t}{R-t} \right) \right] \\
 &= \frac{K\sigma}{2R} \left[2R(R^2 - x^2)^{1/2} + x^2 \log \frac{R + (R^2 - x^2)^{1/2}}{R - (R^2 - x^2)^{1/2}} \right]
 \end{aligned}$$

The value of the above expression has a maximum for a value of the fractional radius of six-tenths. This value corresponds closely to the

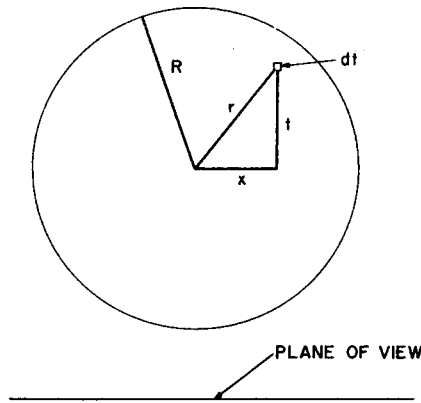


Fig. 11. Horizontal cross section of circular capillary viewed perpendicular to its axis, illustrating element of thickness dt at distance t from mid-plane at a radius r from the center, which appears at a distance x from the center from the plane of view.

fractional radius 0.7 at which maximum birefringence occurs in Figures 9 and 10. The variation between the observed and calculated fractional radii may be due to the lens effect of the glass walls of the capillary, or the lack of linear variation of birefringence with shear stress, or both.

With increasing stress, some concentration of birefringence occurred at the inlet. However, the first departure from laminar flow occurred within the capillary. Breaks in the isochromes near the capillary wall and a grainy structure of the isochromes were observed. These are illustrated in the fourth and fifth photographs in Figures 9 and 10, both.

The last photographs in Figures 9 and 10 show the birefringence at rates greater than those corresponding to C in Figure 1. At the critical stress where the rate was observed to vary between the upper and lower branches of the flow curve, the birefringence pattern varied simultaneously. At low

rates the appearance was like the second-last photograph in Figures 9 and 10; at high rates, like the last photographs.

At rates along the upper branch of the flow curves, the birefringence patterns are similar to those of materials exhibiting inlet fracture; i.e., the birefringence reaches a maximum at the inlet and decreases with increasing distance down the capillary. Extrudate irregularity similar to that associated with inlet fracture may occur along the upper branch of the flow curves. Motion pictures at about 600 frames/sec. of linear polyethylene flowing through a capillary with a flat inlet showed birefringence effects similar to those for inlet fracture. Birefringence within the capillary was not axially symmetrical. Rather than continuous isochromes, the birefringence occurred as random patches. This type of irregular flow appeared to occur just at the inlet, in the region of the seal made by the juncture of the glass pipe and the capillary, where observation was prevented. The isochromes above the seal were symmetrical about the axis; the effects of irregular flow did not propagate upstream from the seal at the capillary inlet.

The results of the flow birefringence studies may be summarized as follows. Elastic strain for linear polyethylene and TFE-HFP copolymer decreases but little with increasing amounts of shear. The first departure from laminar flow occurs within the capillary. Presumably, this first failure, which might be called "land fracture," results in the ripple type of surface roughness (the term land is used to mean the part of the flow channel of uniform cross section, in this case the capillary). Finally, at the stress at which shear rate tends to be double valued, the large increase in birefringence occurring at the inlet and the decrease with distance down the capillary are both indicative of slip or sharp yielding occurring within the capillary. This type of slip in flow of thermoplastic polymers may be similar to that observed in rubber.¹² Irregular flow, apparently of the inlet fracture type, may occur after onset of slip.

Instantaneous Modulus

Support for the hypothesis that the phenomena occurring within the capillary are fracture phenomena similar to those occurring at the inlet in the case of other polymers was found in instantaneous modulus data. Instantaneous modulus was measured in a cone-and-plate rotational viscometer after sudden small shear. The cone was rotated suddenly by driving a radium arm on the cone shaft against a stop. As the rate of shear was increased, the stress at the beginning of the relaxation approached a maximum. The ratio of the maximum stress to the strain was taken as the instantaneous modulus. The maximum instantaneous modulus so measured is compared with the stress at which fracture occurs in the capillary experiment in Figure 12.¹³ A linear relation is observed for which the slope is about 5. If the slope, stress/instantaneous modulus or stress/(stress/strain), is considered to be related to the elastic strain at onset of fracture, then fracture occurs at an elastic strain of 5 in polymers

that exhibit either *inlet* or *land* fracture. These results, along with those of Bagley,¹⁴ support the suggestion made by Tordella³ that fracture occurs at a critical amount of elastic strain. The results differ from those of Bagley¹⁴ in that he found the amounts of strain at fracture for linear polyethylene to differ from those for branched polyethylene and polystyrene.

CONCLUSION

Irregularity of the shape of molten polymer streams emerging from capillaries is associated with flow instabilities initiating at the capillary inlet, in some cases, and within the capillary itself, in others. Both effects appear to occur at the site of maximum elastic strain at a critical amount of strain of about 5 units. No positive evidence was found for the

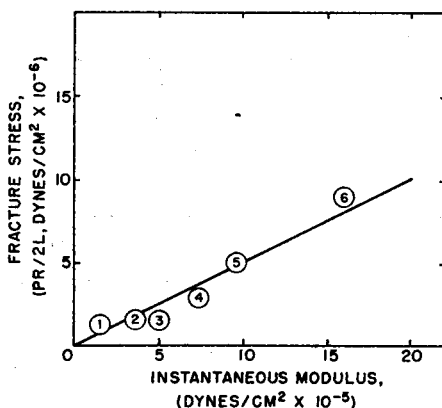


Fig. 12. Comparison of fracture stress using 3.65 length-to-diameter ratio capillary with instantaneous modulus for various polymers: (1) polyethylene at 150°C., melt index 2.1, density 0.917; (2) polyethylene at 150°C., melt index 2.0, density 0.923; (3) polyethylene at 150°C., melt index 2.0, density 0.914; (4) polyethylene, melt index 0.50, density 0.958; (5) polymethyl methacrylate 200°C.; (6) high polymer of formaldehyde, 200°C.

suggested⁴ initiation of roughness by differential elastic recovery between the less strained inner portions and more highly strained outer portions of emerging polymer streams.

The term *melt fracture* was coined to denote elastic rather than other types of fracture. Fracture at the respective sites of incidence may be denoted by the terms *inlet fracture* and *land fracture*. Polymers in which land fracture occurs slip intermittently at higher stresses, and may then undergo inlet fracture. Slip appears to be the ultimate result of land fracture. The lack of dependence of land fracture and slip on material of construction of the capillary indicates that the phenomena are predominantly dependent on polymer properties rather than on polymer-solid interfacial forces.

The decrease in birefringence in capillaries for inlet fracture types of

polymers suggests some kind of yielding within the capillary. However, slip does not seem to occur: the criteria for slip given by Mooney¹² are not followed, and an approximately parabolic velocity dependence on radius is observed, even after onset of inlet fracture. Further, the success of inlet pressure drop corrections⁹ indicates uniform viscosity within the capillary. The energy dissipation associated with the decreasing birefringence appears small.

The dependence of the flow rate after slip on the L/D ratio of the capillary follows reasonably from the observation that slip occurs. The proportional increase in pressure drop across the inlet at onset of slip decreases with decreasing L/D ratio. In addition, the total pressure drop at a given shear stress decreases with decreasing L/D . The combined effect is that of decreasing stress at the inlet at onset of slip with decreasing L/D ratio.

Use of the term $4Q/\pi R^2$ (where Q is volumetric rate of flow and R is the capillary radius) to indicate maximum rate of shear of molten polymers in capillaries is well recognized as an approximation. Its use for describing shear rate after slip occurs has very little meaning, but has considerable convenience in the presentation of data.

Land fracture and slip were well defined in some samples of linear polyethylene and copolymers of tetrafluoroethylene and hexafluoropropylene. Slip with inlet fracture has been observed for another polymer, polytetrafluoroethylene. Land fracture may occur for this material, but the very low rates of shear, $\sim 10^{-5}$ sec.⁻¹ at 380°C., prior to slip make observation of land fracture difficult. Slip behavior has been observed occasionally¹⁵ in nylon 66 and in high polymers of formaldehyde.¹⁶ These two latter materials exhibit birefringence of the type shown for linear polyethylene and TFE-HFP copolymers.

The cause of the different types of fracture behavior is not known. However, the response to shear of the two types of polymers are obviously different. The inlet fracture type of polymer reaches a maximum in elastic strain after relatively small amounts of shear. The land fracture type maintains constant strain within the capillary, independent of the amount of shear. This variation in response to shear may be the result of different degrees of molecular extension and/or intra- and intermolecular entanglement.

There is insufficient evidence to substantiate any proposed fracture mechanism. However, the following appears a reasonable initial hypothesis. The fracture-type flow instabilities result from local intermolecular disentanglement of molecules or clusters of molecules. Viscosity data indicate that molecules of branched polyethylene are more compact than those of linear polyethylene. At a given Newtonian viscosity the branched polymers have considerably higher weight-average molecular weights than linear polymers.¹⁷ Consequently, the molecules of the branched polymers must be more tightly coiled than those of linear polymers with less intermolecular entanglement. The compact branched molecules or clusters of

molecules apparently disentangle from their neighbors at relatively small amounts of shear with the observed results of decreasing birefringence in the capillary and inlet fracture. With the linear polyethylenes, disentanglement tends to occur at larger amounts of shear, so that fracture occurs within capillaries rather than at the inlets.

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Synopsis

Instability in melt flow initiates within capillaries for some samples of the two types of polymers linear polyethylene and copolymers of tetrafluoroethylene and hexafluoropropylene. This behavior contrasts with a previously described instability which initiated at capillary inlets. The instability initiating within capillaries results, initially, in fine-scale surface roughness and, ultimately, in slip or plug flow. Instabilities initiating at both sites appear to result from elastic failure or melt fracture at critical elastic strains of about 5 units. Data supporting these conclusions were obtained from capillary flow and flow birefringence, and rotational viscometry.

Résumé

L'instabilité constaté dans l'écoulement à l'état fondu, commence à l'intérieur des capillaires, pour certains échantillons de deux types de polymères, le polyéthylène linéaire et des copolymères de tétrafluoroéthylène et d'hexafluoropropylène. Ce comportement diffère de l'instabilité décrite précédemment et qui débutait à l'entrée du capillaire. L'instabilité qui commence à l'intérieur des capillaires est due initialement à la rugosité de la surface et finalement à l'écoulement laminaire ou houleux. Les instabilités qui débutent aux deux endroits semblent être dues au manque d'élasticité ou à la cassure lors de la fusion pour des tensions critiques d'élasticité d'environ 5 unités. Les résultats qui confirment ces conclusions ont été obtenus à partir d'écoulement capillaire, de biréfringence d'écoulement et de viscosimétrie de rotation.

Zusammenfassung

Bei einigen Proben von zwei Polymertypen, nämlich linearem Polyäthylen und Copolymeren von Tetrafluoräthylen und Hexafluorpropylen, entsteht eine Instabilität beim Schmelzfluss innerhalb von Kapillaren. Dieses Verhalten steht im Gegensatz zu einer früher beschriebenen Instabilität, die an Kapillaröffnungen entsteht. Die innerhalb der Kapillaren einsetzende Instabilität führt anfänglich zu einer feingradigen Oberflächenrauigkeit und schliesslich zu einem Fliesen unter Gleitung oder mit Pfropfenbildung. Instabilitäten, die an beiden Stellen entstehen, scheinen durch elastisches Versagen oder durch Schmelzbruch bei kritischer elastischer Verformung von etwa 5 Einheiten bedingt zu sein. Diese Schlussfolgerungen werden durch Versuchsdaten beim kapillaren Fliesen, bei der Strömungsdoppelbrechung und der Rotationsviskosimetrie gestützt.

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