

Solid-State Extrusion

[Abstract](#)

- [1. Introduction](#)
- [2. Extrusion with Changes in Billet Shape](#)
- [3. Extrusion Based on Simple Shear](#)
- [4. Applications](#)

[Bibliography](#)

[Figures](#)

Subject categories

[Processing and Finishing](#)

Solid-State Extrusion

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Encyclopedia of Polymer Science and Technology

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DOI: 10.1002/0471440264.pst343

Article Online Posting Date: July 15, 2004

[NEXT](#)

Keywords: polymer; solid-state extrusion; equal channel angular extrusion; twist extrusion; extrusion conditions; deformation mechanism; structure; properties

Abstract

Solid-state extrusion (SSE) is a method for attaining a highly oriented state of polymers, and is based on polymer deformation at temperatures just below the polymer melting temperature, or for amorphous polymers just above the glass-transition temperature mainly by extrusion through conical or slit dies.

SSE is used for shape forming and structure modification of various polymer materials (homopolymers, polymer blends, filled polymer composites) and makes it possible to produce fibers, films, pipes, shapes including those of large cross section and of different configurations, possessing high strength–stress characteristics.

In this article, much attention is given to the most known SSE versions—plunger extrusion and hydrostatic extrusion with the information on the influence of process controlling parameters on extrusion pressure, velocity of product exiting, value of extrudate swell, structure and properties of the processed material. There exists a region of controlling parameters where the process is stable; outside the region, the polymer flow is discontinuous, extrudate shape is imperfect and, finally, its failed.

Two new techniques of SSE are presented in brief, namely the equal channel angular extrusion and the twist extrusion which, in contrast to the conventional ones, are based on polymer simple shear, not drawing. These techniques are found to be promising for structure modification of polymers.

[NEXT](#)

[\[Home\]](#) [\[A to Z\]](#) [\[Subjects\]](#) [\[Search\]](#)

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[PREVIOUS](#) [NEXT](#)

1. Introduction

Solid-state extrusion (SSE) is one of the well-known methods used for attaining a high oriented state of polymers (1-4). It enables the production of fibers, films, pipes, shapes, including those of large cross section and of various configurations—all possessing high strain–stress characteristics. SSE is based on deformation at temperatures just below the polymer melting temperature, or for amorphous polymers just above the glass-transition temperature. The deformation is mainly by [extrusion](#) through conical or slit dies, it is used for shape forming and structure modification of polymer materials of different nature, such as homopolymers, polymer blends, filled polymer composites, etc (4-10).

For the production of high modulus high density polyethylene (HDPE), polytetrafluoroethylene (PTFE) and other polymers the solid-state extrusion is frequently used at the first stage of a two-step drawing process (11-16). The polymer is first drawn up to the limiting draw ratio at a fast rate and subsequently slowly superdrawn at a temperature that depends on the morphology. Extrusion draw ratio (EDR for short; it is defined as the ratio of die entrance to exit cross-sectional areas), pressure, extrusion rate, temperature, and die geometry are the parameters that effectively influence the process of the high oriented polymer production, the polymer structure and properties.

This article gives a brief information on different SSE methods and their influence on structure and properties of polymer materials. Attention is primarily paid to the most known methods, such as [plunger extrusion](#) and [hydrostatic extrusion](#). Two new methods of SSE implementation are analyzed in brief, which, in contrast to the conventional ones, are based on polymer simple shear, not drawing. Lately, they have been of high interest in view of the production of nanostructural metallic materials (17) and are now known as methods of severe plastic deformation (SPD).

The treatment by the scheme of simple shear under pressure results in the intensive refinement of metal crystallites with the formation of ultrafine grain substructures possessing unique properties (17). SPD of polymer materials has become the subject of investigation recently. The investigation results show that simple shear considerably affects the structure and properties of polymers (18, 19). In contrast to the conventional SSE, the solid-state extrusion based on simple shear does not result in final changes of billet shape and dimensions. That is why, the problems relating to the conventional SSE are included in the section “Extrusion with Changes in Billet Shape”, while the SPD methods for polymers and a brief analysis of the experimental results are given in the section “Extrusion Based on Simple Shear”.

[PREVIOUS](#) [NEXT](#)

[\[Home\]](#) [\[A to Z\]](#) [\[Subjects\]](#) [\[Search\]](#)

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PREVIOUS NEXT

2. Extrusion with Changes in Billet Shape

The SSE process based on changes in polymer-billet shape is, first of all, characterized by the EDR value. The limited EDR value is determined by polymer type, molecular weight and morphology. For example, for HDPE it may reach more than 40 (1). For polyoxymethylene (POM), polypropylene (PP), PTFE the maximum values of draw ratio equal 10, 6, and 4 (20-22), respectively. For polymethylmethacrylate (PMMA), polystyrene (PS), polycarbonate (PC), and other amorphous polymers they are even lower (21, 23). The extruded specimens are highly oriented and possess improved mechanical properties. In the case of semicrystalline polymers, the extrusion results in a considerable increase of tensile modulus and strength (1, 2). With amorphous polymers, a considerable increase of plasticity is observed alongside with increase of tensile moduli (21, 23).

2.1. Extrusion Methods

There exist two approaches to SSE implementation. These are the methods of plunger and hydrostatic extrusion. The first investigations by using the plunger extrusion were made on polyethylene. Extrudates were produced by melt crystallization under pressure and by orientation in a capillary rheometer of the Instron type (1, 24-26). Moreover, the crystallization of polymer took place at die entrance and in the rheometer cylinder. Such a technique made it possible to produce short transparent and strong fibers of less than 1 mm diameter or films of similar characteristics.

In more recent experiments, the polymer billet was formed prior to SSE. The extrusion process was implemented with both the capillary rheometer and by using a high pressure chamber and a pressing equipment (6-8, 25, 27), thus the potential applications of the method have become wider. Figure 1a is a schematic showing the process of plunger extrusion, which is the most frequently applied in practice. The polymer is extruded by a rod through the die of conical shape. With the dies of different deforming-channel sections (such as slot, round, square, screw ones) it is possible to produce high oriented fibers or films, to change the shape and to draw the polymer billet for having shapes of improved physical and mechanical properties.

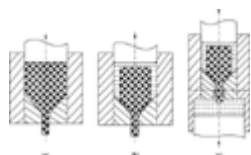


Figure 1. Schematic representation of plunger extrusion (a), hydrostatic extrusion (b), and hydrostatic extrusion with backpressure (c).

[Full View]

For the structural modification of polymers the method of solid-state coextrusion (SSCE) is used (28, 29). This is a technique for polymers which is difficult to draw, or if the highest draw is desired. The polymer to be drawn is extruded within a second more ductile polymer (within a sandwich arrangement). The process has been used for the extrusion of films (split-billet coextrusion) and fibers

(concentric-billet coextrusion). In particular, the SSCE technique was used for the production of highly oriented films of semicrystalline polyamides (28), poly(vinylidene fluoride) (30, 31), HDPE (25, 28, 29), poly(L-lactic acid) (32), and amorphous PS (29) and poly(ethylene terephthalate) (PET) (33). This technique makes it possible to coextrude polymers of different molecular weights at temperatures much lower than the melting temperature and to produce superthin films of high strength characteristics.

In the process of hydrostatic extrusion, the polymer billet is forced by a compressed liquid through a narrowing die. The high pressure develops due to rod travel, as shown in Figure 1a and 1b, or is generated by a high pressure compressor (the so-called “compressor version of the hydrostatic extrusion”). This processing method prevails over the plunger extrusion since there is no friction of the billet material on the channel walls in the high pressure chamber, and the friction between the polymer and the die is low because of liquid penetration into the gap. Besides, in the hydrostatic extrusion the effect of material plasticization is largely realized at the expense of high pressure action (26), so brittle polymers can be deformed as well (21, 23). If necessary to increase polymer ductility, the deformation with backpressure is carried out (Fig. 1c) (26). The first investigation of the hydrostatic-extrusion process as well as of the structure and properties of extrudates were done approximately at the same time, as for the plunger extrusion (34-36), and they were being actively developed later on (20-23, 25, 37, 38).

For SSE, monolithic or powder billets are used. In the latter case, the polymer powder is compacted, heated to the temperature close to the melting temperature and then extruded. The extrudates of ultra high molecular weight polyethylene (UHMWPE) (8), polymerization-filled polyethylene composites (7, 9) of polyetherketone (39), polyarylate (40) have been produced in this way. With the plunger extrusion of a powder billet, high EDR values are attained at temperatures much lower than in the case when a monolithic sample is deformed. The powder billet is also used for the production of high oriented polymers by the SSCE method and twostep drawing process (12, 13).

Polymer-billet geometry is defined by SSE scheme. In Figure 2, there are some variants of billets for the plunger (a-f) and hydrostatic (a, b) extrusion to be used in the described SSE techniques. The hydrostatic extrusion is of a lesser potential application as it is necessary to protect the compacted powder from liquid penetration and to seal the billet in the die to prevent liquid from leakage under pressure rise (coextrusion).

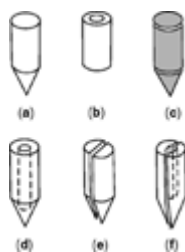


Figure 2. Billets for SSE (a, b, c), and shells for SSCE (d, e, f): (a) solid plug, (b) pipe billet, (c) powder billet, (d) concentric shell, (e) full width shell, and (f) half width shell. [Full View]

2.2. Extrusion Conditions

For a brief and clear presentation of the subject matter we make use of the approach of system analysis to represent SSE as a system having inputs and outputs, and set forth the relationships. The input variables $\{X\}$ are parameters controllable for the purpose of affecting the output variables $\{Y\}$. In the case of SSE, with a predetermined material of the billet, the former parameters are: the extrusion, draw ratio, die geometry, velocity of plunger motion for the plunger extrusion, and plunger hydroextrusion; pressure in chamber in the case of compressor version of the hydroextrusion; backpressure or drag force applied to the extruded product; temperature of chamber and die heating; structure of billet

material; dimensions and geometry of the billet; and lubricant and fluid (for hydroextrusion).

In SSE, the output variables are: extrusion pressure, velocity of product exiting, value of extrudate swell, degree of deformation and strain rate, and structure and properties of the processed material.

2.3. The Normal Solid-State Extrusion Process and Its Violations

The normal course of SSE is characterized by a nonstationary stage at the beginning of the process and a constant velocity of product exiting during the rest of the process. In this case, the extrudate has a regular cylindrical shape with smooth surface having no visible defects.

In the case of plunger extrusion, at the stationary stage of the normal process the velocity of the exiting extrudate v_e is described by the relation $v_e = R v_p$, where v_p is the velocity of plunger motion, R is the extrusion draw ratio. For the plunger version of the hydrostatic extrusion $v_e = R v_p S_p/S_b$, where S_b is the cross-sectional area of the billet, S_p is the cross sectional area of the plunger. For the compressor version of the hydrostatic extrusion, at the stationary stage of the process the velocity of extrudate existing is determined by the value of pressure (a more detailed explanation is given below).

The normal course of SSE is only realized at a definite range of input parameters. Outside the range there occurs some disturbances in the normal process: a discontinuous movement of the extrudate without an essential change of its shape (the stick-slip effect); the unstable flow of the polymer in the paraxial zone of the extrudate resulting in polymer turbidity; the pulsating flow with a considerable deformation of extrudate shape; and the occurrence of spiral-like cracks on its surface.

The above defects are due to different reasons. In the article (41), the stick-slip effect is related to self-excited oscillations initiated by the dependence of static friction–stress between the billet and the die at the time when they are in contact. The latter is connected with the viscoelastic properties of the rough billet surface and by lubricant squeezing out from the region of contact. Ward and co-workers (1) explain the stick-slip effect by the heating of billet during the deformation. The pulsatory flow is assumed to be due to a competition between the viscosity and high elasticity of polymers (42).

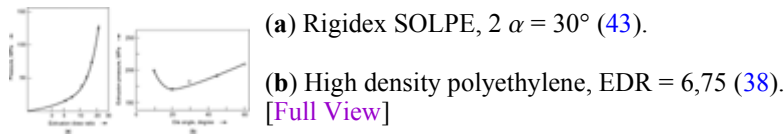
In accordance with different physical causes of interruption in the normal course of SSE (eg 41, 42), criteria have been obtained and the boundaries of the input-variable range have been determined for the normal regime of the process. For example, for the stick-slip effect the lower bound of the velocity of plunger motion has been determined in (41). The upper bound of the velocity region of normal process has been derived in (42) based on the Weissenberg (Deborah) criterion.

In (41, 44), different engineering approaches to stick-slip effect elimination have been proposed. The most effective are the application of a tractive force to the front end of the product being extruded.

2.4. Extrusion Pressure

The characteristic feature of the plot of extrusion pressure P vs the logarithm of extrusion draw ratio R is that it is convex to the axis of $\ln R$ (Fig. 3). In the case of the hydrostatic extrusion of metals, the dependence is practically linear (44). This difference is because with polymers there is a more intensive strain hardening, strain rate, and pressure hardening as compared to metals. At a qualitative level this is shown as follows.

Figure 3. Typical graphs of extrusion pressure versus extrusion draw ratio (log scale) (a) and die angle (b).



As the work of plastic deformation is equal to that of external forces, it follows, in the first approximation, that the hydrostatic-extrusion pressure P can be determined by the formula (45):

$$P = \int_0^{\varepsilon^* + \ln R} \sigma_s(\varepsilon, \dot{\varepsilon}, \bar{p}, \bar{T}) d\varepsilon \quad (1)$$

where $\sigma_s(\varepsilon, \dot{\varepsilon}, \bar{p}, \bar{T})$ is the strain resistance of polymer, ε is the strain, $\dot{\varepsilon}$, \bar{p} , \bar{T} are values of the strain rate, pressure, and temperature averaged with respect to the volume of deformation center, respectively; ε^* is the strain induced by the entrance and exit zones of the die (it depends on the value of die cone angle and does not depend on the extrusion ratio).

By double differentiation of relation 1 we have

$$\begin{aligned} \frac{d^2 P}{d(\ln R)^2} &= \left(\frac{\partial \sigma_s}{\partial \varepsilon} + \frac{\partial \sigma_s}{\partial \dot{\varepsilon}} \frac{d\dot{\varepsilon}}{d(\ln R)} + \frac{\partial \sigma_s}{\partial \bar{p}} \frac{d\bar{p}}{d(\ln R)} \right) \Bigg|_{\varepsilon^* + \ln R} \\ &+ \int_0^{\varepsilon^* + \ln R} \frac{d}{d(\ln R)} \left(\frac{\partial \sigma_s}{\partial \dot{\varepsilon}} \frac{d\dot{\varepsilon}}{d(\ln R)} + \frac{\partial \sigma_s}{\partial \bar{p}} \frac{d\bar{p}}{d(\ln R)} \right) d\varepsilon \end{aligned} \quad (2)$$

In view of polymer strain, velocity, and pressure hardening, all the derivatives of σ_s in the righthand part of relation (2) are positive. Besides, the average values of the strain rate and pressure at the center of deformation are increasing with extrusion draw ratio, ie the derivatives of $\dot{\varepsilon}$ and \bar{p} with respect to $\ln R$ are also positive. It follows that the right-hand part of relation (2) and, thus, the second derivative of P with respect to $\ln R$ are also positive, hence we have the convex plot of function $P = P(\ln R)$ with respect to the axis of R . According to (2), more intensive the effects, the higher the strain, strain rate and, stress hardening of the material.

On the dependence of extrusion pressure on die cone angle 2β there is a minimum (Fig. 3b). This is because of the competition of two factors that affect the extrusion pressure: contact friction and additional deformation ε^* at the input and output from the conical part of the die. The first and the second factor become significant with a decrease or an increase in cone angle, respectively.

In view of the fact that the polymer strain resistance depends much on strain rate and temperature, in plunger extrusion and plunger version of hydrostatic extrusion the extrusion pressure increases with the velocity of plunger motion and decreases with temperature rise. For the compressor version of the hydrostatic extrusion, pressure in the chamber determines the velocity of extrudate exiting.

The above-mentioned dependences of SSE pressure on input parameters can be taken into account within the framework of mechanical models of the process. In the most detailed ones (20), the relationships for calculation of pressure have been derived with a rather good agreement between

theory and experiment. The value of extrusion pressure can be estimated, in a first approximation, by formula (1), where $\varepsilon^* = (\pi/2) \tan(\beta/2)$ (46). The $\sigma_s(\varepsilon)$ dependence should be experimentally determined with the average, in deformation center, temperature and rate of deformation. It is easy to show that the latter is approximately equal to $\dot{\varepsilon} = \frac{2v_e R^3}{d_e} \tan \beta$ where d_e is the diameter of the extrudate.

2.5. The Velocity of Extrudate Exiting

It has been already pointed that the rate of extrusion is an output variable only in the case of the compressor version of hydrostatic extrusion. For the plunger processes, under the normal flow, the rate of extrusion is an independent quantity. If the working fluid is pumped to the chamber by a compressor, and in the chamber there is a control valve for releasing unnecessary liquid, then it is possible to maintain the extrusion pressure at a preset level. In this case, the velocity of product exiting depends on pressure and temperature. According to theoretical estimates, and to experiment (38), the extrusion rate exponentially increases with pressure.

2.6. The Swelling of Extrudates

In the case of SSE, a phenomenon of extrudate swelling at the die exit is observed. Taking it into account, the quantities of the actual extrusion draw ratio $R_d = S_b/S_e$ and the nominal extrusion draw ratio $R_n = S_b/S_d$ are introduced, where S_e is the area of the extrudate cross section, S_d is the area of the die exit. Parameter R_a correlates with the EDR value.

This phenomenon is related to a partial relaxation of viscoelastic strain in polymer during the extrusion. Its value is determined by both the polymer characteristic properties and the SSE conditions (1, 5, 23). With an increase in EDR value the swelling diminishes sometimes close to zero, the same as was observed for the linear polyethylene with $M_w = 1 \times 10^5$ (1). A considerable swelling is characteristic of semicrystalline polymers possessing high molecular weight, as well as of amorphous polymers. To reduce the swelling, the use is made of dies with a longer calibrating zone.

2.7. Peculiarities of the Mechanics of Powder Extrusion

Mathematical modeling of the SSE process by the method of continuum mechanics is based on defining equations relating the components of stress and strain tensors. Among them is the yield condition. For the isotropic monolithic materials it is in the Tresca, Mises or Hill form (47).

The mathematical description of the powder billet SSE cannot rely on the above conditions of plasticity, as they do not take the change in density of the extruded sample into account. Nor the conditions of plasticity used to model the compaction of metal powders are suitable for this case (48). They imply that with the increase in hydrostatic pressure the relative density of powder material tends to unity. However, in the case of hydrostatic compaction of polymer powders, the relative density of compacts tends to a value less than unity, which is typical of the polymer. The reason is in difference of compaction mechanisms for the metal and polymer powders.

For polymers, the compaction proceeds in two stages. The first stage consists in shear and turning of some particles relatively each other. They experience high elastic strain resulting in the increase of the area of real contact and forces of the interparticle friction. The second stage of compaction is mainly conditioned by high elastic–plastic straining of the particles that fill the free volume. After releasing the external pressure the polymer material decreases its density as the elastic deformation is reversible.

A condition of plasticity has been proposed (49) that makes allowance for a changing of the density of powder polymer during the deformation under pressure. It was the basis for the elaborated SSE theory

to predict the density of extrudates (50). It follows, in particular, that during the SSE a nonmonotonic dependence of the density of extrudates on the extrusion ratio is possible. The presence of the effect has been verified experimentally.

2.8. Deformation Mechanism

The deformation mechanism has been studied in more detail for the case of semicrystalline polymers, polyethylene, in particular (1, 2). There exist a number of models explaining the evolution of polymer structure at high draws. The models can be subdivided into two categories (2), namely, the microfibril plus the tie molecule model, as proposed by Peterlin and co-workers and the extended chain model, which is similar to Ward's crystalline bridge model.

According to the Peterlin's model (51, 52), the starting material consists of the packed lamellae with chain folds, while the completely extruded one consists of oriented microfibrils. In the lamellar structure, within the lamella there is a macromolecule that forms folds in direction normal to chain axis. In the fibrillar structure, the macromolecule is within the microfibril, but already in direction parallel to chain axis. Microfibrils consist of crystal blocks and noncrystalline regions connected by taut tie molecules. Under drawing, the lamellar structure changes to the fibrillar one at the expense of shearing, rotation, chain tilt, and slip processes taking place in lamella. The axial modulus is proportional to the number of load-bearing tie molecules that cross the noncrystalline regions to connect adjacent crystalline blocks along the microfibril.

Ward's model (53, 54) provides the presence of "crystalline bridges" formed from groups of crystallized taut intrafibrillar tie molecules between the crystallites. This morphology consists of fibrillar stacks of crystallites, linked by intercrystalline bridges, or of a continuous crystal containing disordered regions. In either case, the crystalline phase is essentially continuous in the crystallographic *c*-direction. The degree of continuity increases with EDR. This model is intermediate between a lamellar and an extended-chain structure (2).

The results of the electron-microscopy investigations of the oriented polyethylene suggest several consecutive stages of the deformation mechanism (2). At the first stage, lamellae move rigidly apart with the strain accommodated almost entirely by the interlamellae amorphous layer. At the second stage, the tie chains become highly extended and slip initiates in the crystalline lamellae. After this, strain-induced crystallization of the oriented amorphous fibers occurs. At the third stage, the blocks of crystal are pulled out of the lamellae; as block shear continues, the portion of lamellae that is below a critical size decrystallizes and contributes to strain softening. Finally, both the blocks form the crystal shear process and the strain-induced crystallization become aligned along the tensile axis.

The SSE process of semicrystalline polymers is highly influenced by their molecular weight. Extrusions of bulk crystallized polyethylene of high molecular weight proceed at slower rates and lower maximum EDR than lower molecular weight grades (2). The deformation resistance imposed by the higher degree of entanglement between molecular chains tends to inhibit high elongation.

2.9. Properties

The SSE of polymers results in the formation of a structural state possessing high anisotropy of properties, in particular, the strain–stress characteristics, thermal expansion, thermal conductivity, and gas permeability. This anisotropy is closely connected with orientation of chains and reconstruction of structure in crystalline and amorphous regions. For example, for semicrystalline thermoplastics, already at a comparatively low extrusion ratio, the chains in the crystalline phase turn out to be almost completely oriented in the direction of deformation. In the amorphous region, the chain organization is less perfect, but the degree of orientation continuously increases during the drawing. It depends on many factors: structure organization and molecular weight of the polymer, billet type, degree of

drawing, and conditions of the extrusion process.

2.9.1. Semicrystalline Polymers

Extrusion of semicrystalline polymers, in particular polyolefins, essentially influences their mechanical properties. The SSE can increase ten-fold their tensile modulus, as compared to the starting material. Solid plugs of HDPE deformed to EDR = 40 have tensile modulus of about 70 GPa (1). For low density polyethylene (LDPE) the limit draw ratio is less than 9 (55), which can be the result of difficulties in generation of a metastable ductile monoclinic form. As a result, the tensile modulus reaches a value of less than 1 GPa (55). The maximum value of the tensile modulus of the extruded PP is 17 GPa (34). Increase in yield strength and tensile strength occurs simultaneously with the increase of tensile modulus (22, 26, 56). With EDR increasing, the elongation decreases.

For equal EDR values, the values of tensile modulus and tensile strength are, as a rule, the higher, the lower the extrusion temperature (1, 55, 57). For these magnitudes, the dependences on die angle have been determined. For HDPE, they are extreme with a maximum in the range $2\beta = 15\text{--}30^\circ$ (58).

At compression, the effect of extrusion draw on the strain–stress characteristics of samples is not so pronounced (22). As shown (22), for PP, the increase in the modulus of elasticity and yield strength occurs under compression in a direction normal to that of extrusion. In the case of a load applied along the extrudate axis, there is a decrease in the above characteristics, as compared to the initial material.

The mechanical properties of other semicrystalline polymers similarly depend on the conditions of deformation process (4, 59, 60). The thermal properties of semicrystalline polymers subjected to SSE have a highly pronounced anisotropy. Thus, the measurements of thermal conductivity of linear polyethylene done in a wide temperature range in directions parallel (K^{\parallel}) and normal (K^{\perp}) to the direction of extrusion, have shown that $K^{\parallel} > K^{\perp}$ (1, 61). With EDR increase, the K^{\parallel}/K^{\perp} ratio increases significantly.

Upon heating the extrudates there occurs a recovery of shape (dimensions) due to thermal shrinkage (1, 56, 62). The larger the EDR, the more stable the dimensions of the oriented samples in a wide temperature range. In superdrawn samples of linear polyethylene, a noticeable shrinkage occurs practically near the melting temperature of the material (1). The beginning of the shrinkage process depends on the extrusion temperature: the lower the SSE temperature, the earlier begins the shrinkage. The value of the maximum shrinkage stresses increases with increasing EDR and decreasing extrusion temperature (62).

The extrusion of powder billets ensures a considerable improvement of some physical and mechanical characteristics (5). It is used in the case of materials for which the solid plug method is not efficient enough, eg UHMPE and PTFE (9, 63). For PTFE, the maximum value of the so attained flexural modulus at 24°C is 20 GPa at an EDR 40 (64).

As compared to the SSE method, the SSCE makes it possible to realize considerably higher extrusion ratios with semicrystalline polymers. Coextrusion is widely used in a two-stage draw technique for drawing of powders (12, 13, 65) and single-crystal aggregates of polymers grown from dilute solutions (66). For example, a two-stage draw technique was successfully applied to the superdrawing of PTFE virgin powder (13). The films, compression-molded from powder below the melting temperature T_m , were initially coextruded with draws till EDR of 20. These extrudates were further drawn by a second-stage pin draw in the temperature range 300–370°C that covers the T_m . For this, a small portion of the extrudate, gripped at both ends, was rapidly heated by contacting it with the surface of a hot metal cylinder. Then, the extrudate was quickly elongated to the desired length. During elongation, the

contacted area of the extrudate was continuously moved from one end to another end at a rate of ~ 1 m/min to heat and elongate a new sample portion successively. The maximum total draw ratio was 160. The maximum tensile modulus and strength achieved in this work were 102 and 1.4 GPa, respectively.

The aggregates of the solution-grown crystal mats of UHMPE and ultrahigh molecular polypropylene (UHMPP) are ductile to a draw ratio of 350 and 80, respectively. Tensile drawing and a combination of SSCE followed by tensile drawing give the highest and most efficient draw for mats of UHMPE and UHMPP. The tensile modulus of UHMPE of molecular weight $M_w = 8 \times 10^6$ for the limiting draw ratio reaches 230 GPa, and tensile strength—5.4 GPa. For UHMPP of $M_w = 8 \times 10^6$ these values are 36 and 2.5 GPa.

In article (15), films of atactic poly(acrylonitrile) were initially drawn uniaxially, by SSCE to EDR = 16, followed by tensile draw at different temperatures. For this polymer the highest tensile-modulus value was 28.5 GPa and strength—1.6 GPa.

2.9.2. Amorphous Polymers

As compared to semicrystalline polymers, the SSE exerts a lower effect on tensile modulus and tensile strength of linear amorphous polymers. It has been shown (23) that the room-temperature hydrostatic extrusion of PMMA gives an increase in the above parameters only by dozens of percent. A similar result has been attained during the three-point bending tests of the extruded PC (23).

The hydrostatic extrusion under elevated temperatures, when higher EDR values are attained, results in a significant effect. In the case of SSE at a temperature of 120°C with the EDR = 6, the tensile modulus and strength of high impact polystyrene become twice as large and equal to 3.8 GPa and 57 MPa, respectively. In the extrudates, the plasticity characteristics (elongation at break, impact strength) are higher than in the initial samples and there is a more than a twofold increase with the EDR and extrusion temperature (23). During the extrusion of a powder billet, eg polyarylate (40), there occurs a simultaneous increase in the strength and plasticity properties as well.

In article (67), where the peculiarities of SSCE of the atactic PS were studied, it is shown that the chain orientation and extension increase with EDR and molecular weight. For a sample of molecular weight 6×10^5 it was possible to achieve a maximum EDR of 27 by multiple draw steps. For the lowest molecular weight PS, the resulting films were brittle. The highest molecular weight sample, 1.8×10^6 , on the other hand, was drawable only to a EDR of 7. The results suggest (2) that in the amorphous polymer, chain entanglements provide the necessary intermolecular network, which facilitates molecular extension and the concomitant orientation. For very high molecular weight, the high viscosity prevents relaxation of the elongated chain. For low molecular weight samples the entanglement network is vastly reduced, and the resulting extensive chain slippage leads to less efficient orientation and extension.

In the case of cross-linked polymers (eg epoxy polymers), the SSE induces the increase in microhardness, tensile modulus, yield stress, and strength (68). Moreover, depending on EDR value these parameters are changing because of the competition between the processes of orientational hardening and mechanical destruction. SSE influences the structure and properties of polymers based on interpenetrating polymer networks similarly (68).

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Solid-State Extrusion

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Encyclopedia of Polymer Science and Technology

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DOI: 10.1002/0471440264.pst343

Article Online Posting Date: July 15, 2004

PREVIOUS NEXT

3. Extrusion Based on Simple Shear

3.1. Equal Channel Angular Extrusion

In this technique (69), a billet is extruded through a die containing two channels, equal in cross section, intersecting at an angle Φ (Fig. 4a). During extrusion the billet undergoes severe shear deformation but retains the same cross-sectional geometry so that it is possible to repeat extrusion for a number of passes. Between two consequent passes it is possible to rotate the billet around its longitudinal axis, creating different equal channel angular extrusion (ECAE) routes (69). Another factor that significantly affects the microstructural development is the channel-intersection angle, which determines the shear strain Γ from an individual pass (69).

$$\Gamma = 2 \cot \left(\frac{\Phi}{2} \right) \quad (3)$$

Smaller Φ will result in higher shear strain from each pass. Usually Φ is equal to 90° – 120°

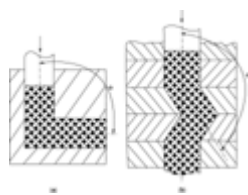


Figure 4. Schematic representation of equal channel angular extrusion (a) and equal channel multiple by angular extrusion (b).
[Full View]

Already in one of the first articles dealing with ECAE application to modify linear LDPE (LLDPE) structure (18) it has been shown that this technique is efficient in orienting the lamellar structure of semicrystalline polymers. Upon four ECAE passes the string-like lamellar bundles in LLDPE are composed of many well-aligned crystal lamellae. The sheaf-like crystal lamellar bundles tend to orient approximately along the extrusion direction. Signs of fragmented crystal lamellae are also observed.

The large strain plastic deformation induced by ECAE, not only generates a high degree of molecular orientation but also gives better molecular packing. As a result, the polymer extrudates can exhibit improved rigidity, strength, barrier properties, and impact resistance (19). In particular, the samples of one-pass extruded PC cut along the extrusion direction have flexural modulus and critical stress-intensity factor K_{LC} values that are higher than those of the annealed PC by 15 and 40%, respectively.

The above technique has been developed into a method of equal channel multiple-angular extrusion (ECMAE) (70). It is based on forcing a polymer billet through several pairs of channels having equal cross section. The channels can intersect with each other at different angles and in different planes as

well (Fig. 4b). A special feature of the ECMAE is that in an extrusion cycle with relatively low angles of the channel intersection (meaning that in each zone the deformation is low), the total deformation value per cycle is comparable with or exceeds the same for ECAE. At the same time, the implementation of any ECMAE scheme is accompanied by the change in shear direction including to the opposite one in a sequential zone.

ECMAE controls the deformation process more effectively and the expected result is attained quicker as compared to ECAE. The ECMAE equipment ensures the increase in manycycle extrusion output, a decrease in single deformation intensities for polymers of low plasticity, a possibility of providing a strict alternation of intensity values and shear directions for a guided structure forming. This has been confirmed by our experiments done on LDPE, HDPE, PTFE, nylon-6, and PC (71).

3.2. Twist Extrusion

In this technique, a billet is extruded through a die with a twist channel (Fig. 5) (72). The angle γ between the twist line and the direction of extrusion changes along the vertical axis of the die, furthermore, it is equal to zero on its starting and ending points.

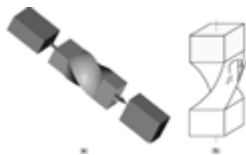


Figure 5. Schematic presentation of the twist extrusion process (a) and of twist channel [Full View]

The above properties of the channel geometry guarantee that the extrusion does not change the form of the workpiece, which allows to accumulate severe plastic deformations by iteratively extruding the workpiece through the die. Figure 5 shows a twist channel with a rectangular cross section. Generally, the cross section can be quite arbitrary, which allows one to use twist extrusion for obtaining not only semimanufactured article, but also prism-shaped end products. Note that by extruding on a mandrel, it is possible to get products with an inner channel.

The main factor that significantly affects the microstructural development is angle γ , which determines the shear strain Γ from an individual pass (72):

$$\Gamma = 2 \tan \gamma \quad (4)$$

Another factor that significantly affects the microstructural development is the route of billet deformation consisting of a successive use of dies having the right-handed and left-handed screw channel.

The results of our WAXS investigations of LDPE and HDPE subjected to twist extrusion show that there are changes in the intensities of corresponding diffraction peaks reflection from planes (110) and (200), as compared to starting materials. The same changes have been found in the corresponding diffraction peaks of nylon-6. Several passes of twist extrusion result in the accumulation of plastic deformation in the samples affecting the level of their mechanical properties, microhardness, in particular. In the case of the LDPE, the increase in the number of cycles results in 1.5 times increase of microhardness.

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DOI: 10.1002/0471440264.pst343

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[PREVIOUS](#) [NEXT](#)

4. Applications

Solid-state extrusion has various commercial applications. This method is used for the production of polyethylene ultrahigh-modulus fibers known under the trade name Tenflor (2). High-modulus ribbons and tapes produced from polyolefins by SSE are used as strappings and electrical insulation. In pipe production, the SSE makes it possible to manufacture biaxially oriented products possessing necessary properties in both the axial and tangential as well as radial directions (4). Owing to the suppression of cold flow (73), it is recommended to use fluoropolymer extrudates as efficient seals in structures operating in corrosive media. The thermal-shrinkage thermoplastics rods produced by SSE find their use as clamps in locking devices (74), as well as in various engineering structures (75).

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DOI: 10.1002/0471440264.pst343

Article Online Posting Date: July 15, 2004

PREVIOUS

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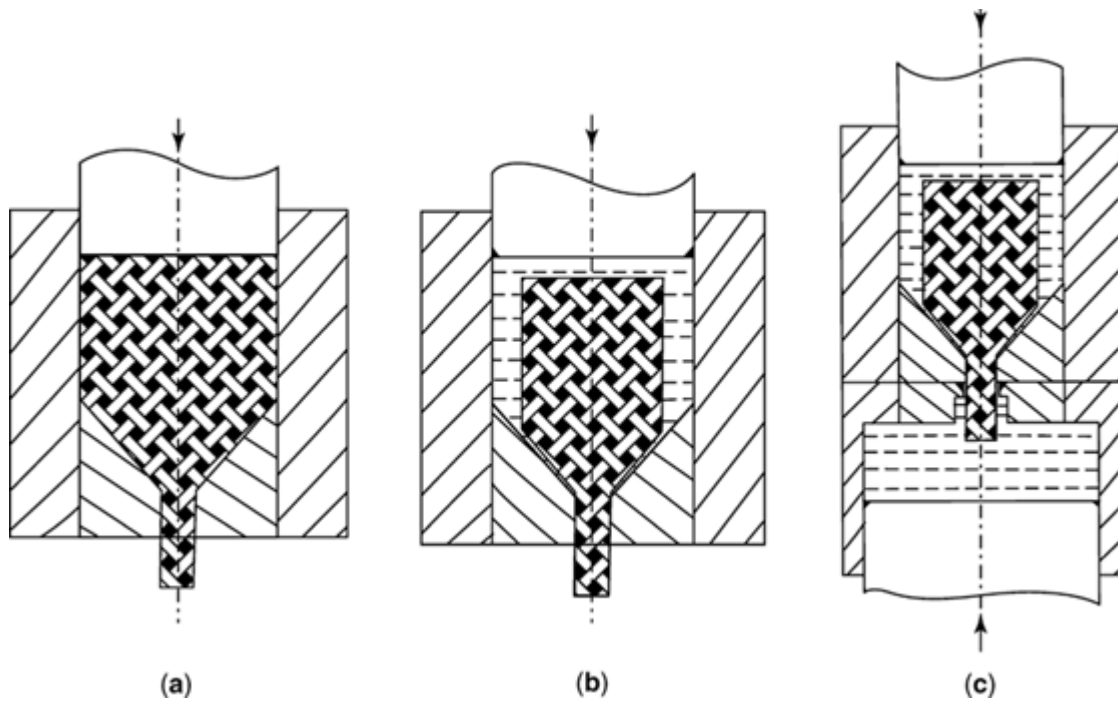
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Figure 1. Schematic representation of plunger extrusion (a), hydrostatic extrusion (b), and hydrostatic extrusion with backpressure (c).

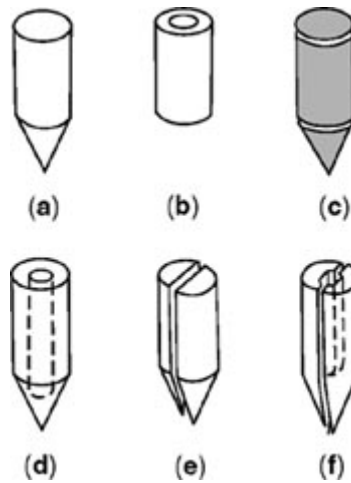


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Figure 2. Billets for SSE (a, b, c), and shells for SSCE (d, e, f): (a) solid plug, (b) pipe billet, (c) powder billet, (d) concentric shell, (e) full width shell, and (f) half width shell.



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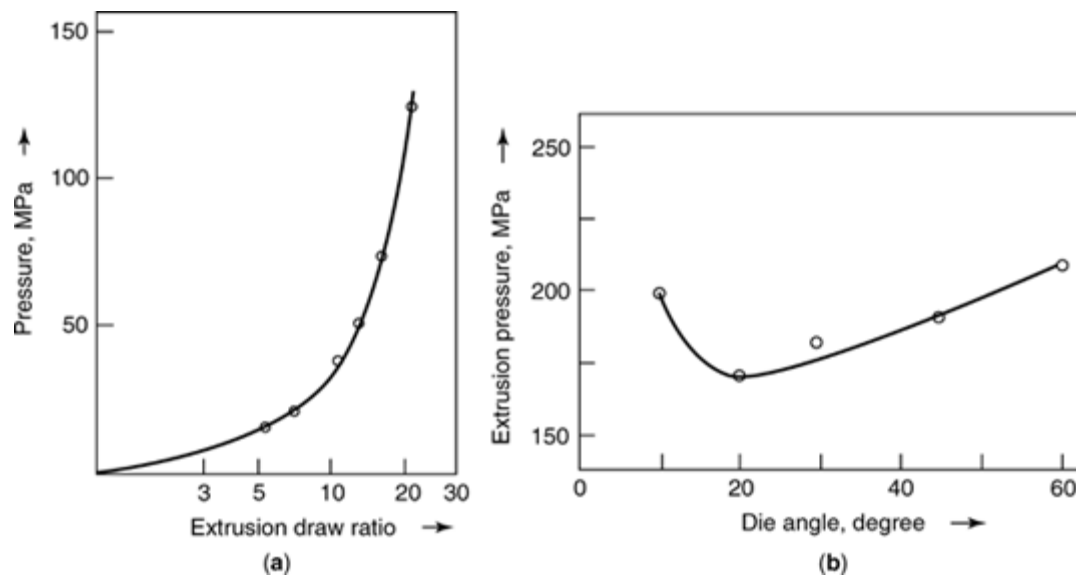
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Figure 3. Typical graphs of extrusion pressure versus extrusion draw ratio (log scale) (a) and die angle (b).

(a) Rigidex SOLPE, $2\alpha = 30^\circ$ (43).

(b) High density polyethylene, EDR = 6,75 (38).

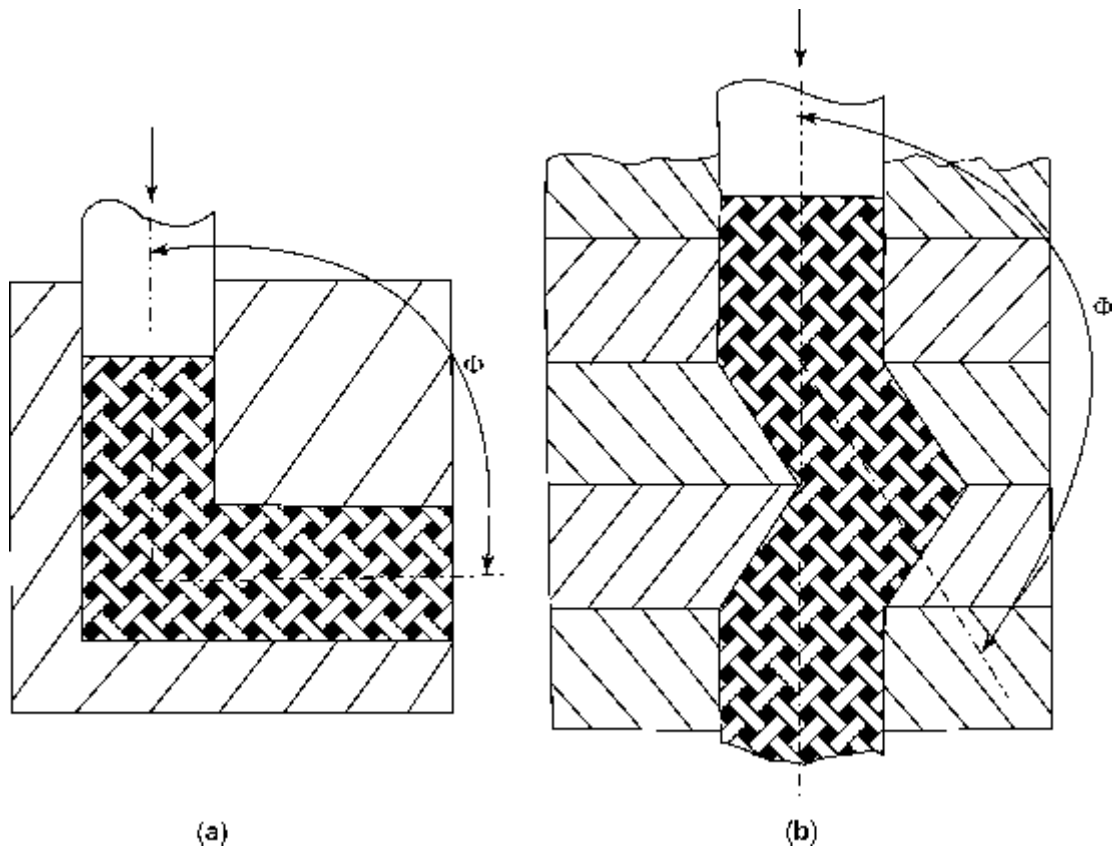


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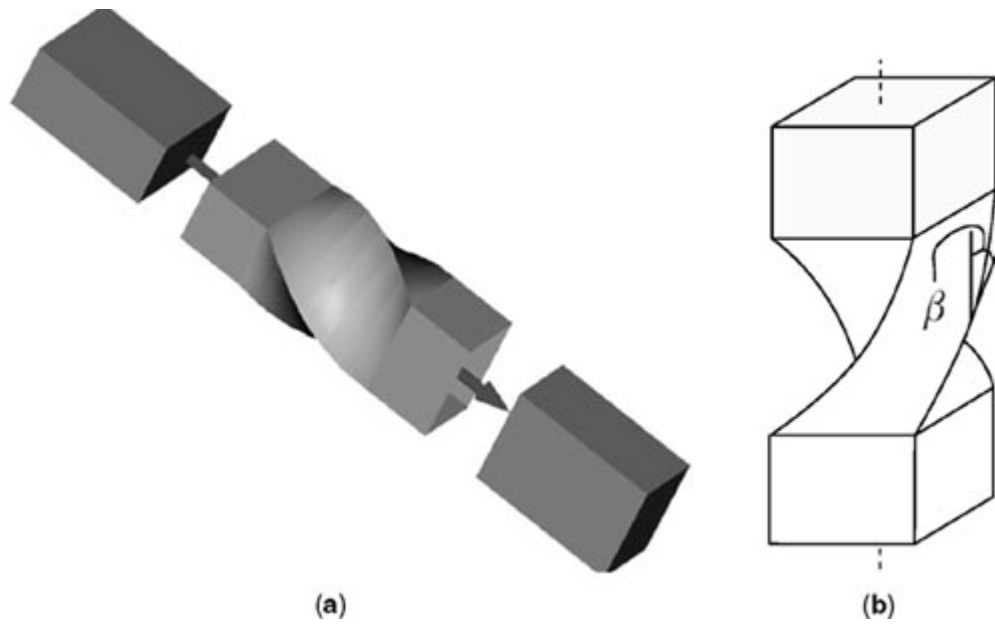
Figure 4. Schematic representation of equal channel angular extrusion (a) and equal channel multiple by angular extrusion (b).



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Figure 5. Schematic presentation of the twist extrusion process (a) and of twist channel (b).

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