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into general and special; the latter in no way represent a complete coverage of the literature, which is so large that the references could well have been extended to take up more space than the text. The references have been selected in such a way as to cite examples for all topics discussed rather than to present complete literature coverage of these topics.

The scope of the book is such that practical and technological aspects, e.g. polymerization processes, processing of resin, and applications are deliberately confined to a rather general discussion; the reliable information available in these fields is sparse in some respects and overwhelming in others due to the technological knowhow and sales policies of a great many large companies involved in polypropylene. It was felt that not much could be gained in a book of this rather small size by going into all these practical and sometimes controversial details.

In the discussion of the various topics some basic knowledge of polymer chemistry, physics, and technology is expected of the reader. It is hoped that in this form the book can usefully serve as an introduction to polypropylene, which is not only a fascinating polymer from a scientific viewpoint but has, as a comparative newcomer, already become accepted as one of the major thermoplastic materials in the plastics and processing industry.

The author wishes to thank the management of the Österreichische Stickstoffwerke A. G. for permission to publish this book and some of his colleagues for helpful discussion and criticism. The editor of this series, Prof. H. Morawetz, has greatly obliged the author by his thorough inspection of the manuscript and by his numerous constructive remarks and criticism. Special thanks are also due to the various industrial companies which have made technical information on polypropylene available to the author and which are listed in the text and references. The permission by various publishers (Hüthig & Wepf Verlag, Basel; Interscience Publishers Inc., New York; Plenum Press, New York; Reinhold Publishing Corp., New York; Società Italiana di Fisica, Milan; J. Wiley & Sons, Inc., New York) to reproduce photographs and diagrams is gratefully acknowledged.

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CHAPTER I

Introduction

Polypropylene (PP) is a crystalline thermoplastic polyolefin resin. It assumes a special position because of its stereoregular (i.e. isotactic) structure, being the first member of the growing family of synthetic stereoregular polymers to achieve industrial importance.

The scope of this book is essentially determined by this special feature of PP and is to cover the following main subjects: preparation and availability of propylene monomer; stereospecific polymerization of propylene and technological processes; physical and chemical properties of PP which are largely determined by its stereoregularity; processing and applications of PP.

PP is a rather novel thermoplastic; it entered the stage of large-scale production in 1957; since then PP has grown at a phenomenal rate, especially since 1963, after some initial difficulties had been overcome. As far as the near future is concerned, it seems to be a safe assumption that PP will still grow at a very high rate and will thus be by far the most important stereoregular thermoplastic material on the market. However, stereoregularity is by no means restricted to PP but is being utilized also in other polyolefins, such as poly-1-butene or poly-4-methylpentene-1, which are both also entering the stage of commercialization and will also be discussed in this book, if only very briefly. In order to stay ahead of these newcomers with basically similar properties and to compete successfully with other existing polyolefins such as low-pressure polyethylene (PE), PP is constantly modified and improved; this also applies to processing operations, and perhaps most important of all, to the search for improvement

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CHAPTER 6

Processing

Processing includes all those steps through which a polymer resin goes between polymerization and fabrication into plastic articles. The processing methods and properties of it. PP are on the whole very similar to those for linear PE, which have been described in great detail. This chapter will therefore be confined to a brief general discussion of processing methods and to some special aspects of PP.

Three stages may be distinguished in the processing of polyolefins: heating, forming, and cooling. The energy absorption in heating up to processing temperatures is somewhat less than that for linear PE because of the lower heat capacity (0.46 compared to 0.55) and the lower heat of fusion (approximately 40 cal/g compared to approximately 60). The forming process is determined by the rheological properties of PP melts (melt index, flow curves, see Chapter 4, section 3(c)). In the selection of a suitable processing temperature the oxidative and thermal stability of PP which is inferior to that of PE must be taken into account (see Chapter 5, section 3). During the cooling process crystallization takes place, depending on the rate of cooling (quenching: smectic structure; slow cooling: large spherulites, see Chapter 4, section 2).

1. Melt Blending

Melt blending may be performed for several purposes: homogenization, of PP resins, incorporation of various additives (e.g. fillers, pigments, stabilizers, etc.), blending with other polymeric materials (polyisobutylene, ethylene-propylene copolymer, etc.). The principles of melt blending are the same as in any liquid mixing process; the molten material is subjected to shear in

such a way that the interfaces intersect lines of flow thus increasing the interfacial area. The quality of mixing is proportional to the product of shear stress and time. Two main types of machines are used for melt blending: the intensive mixer (Banbury mixer) and most commonly various modifications of screw extruders which will convert powdered PP resin plus additives into pelletized material.

2. Extrusion

Extrusion is by far the most important processing method for polyolefins; extrusion processes lead to articles including pipes, rods, profiles, wire coating, sheets, films, filaments and fibers, etc.

Extruders serve to melt and homogenize the polymer and to deliver a constant rate of flow at constant pressure to the die. A number of technical terms for screw extruders are defined in the following (Fig. 6.1):

L/D ratio: L is the length and D the diameter of the screw; usual range 16–28

Compression ratio: ratio of volumes contained in one turn at the feed end and at the discharge end; usual range 3–4

Metering section: screw portion at the discharge end with small groove depth; usual range $2D$ – $14D$

Lead: distance between turns; usually about equal to the diameter.

A schematic illustration of a screw extruder is shown in Fig. 6.2. Extruder flow equations have been developed and were critically discussed.^{1, 2} They permit calculation of output as a function of

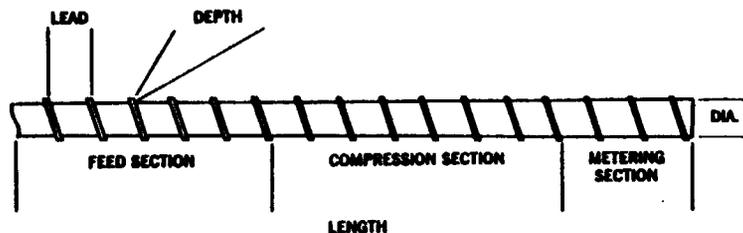


Fig. 6.1. Polyolefin extruder screw^A

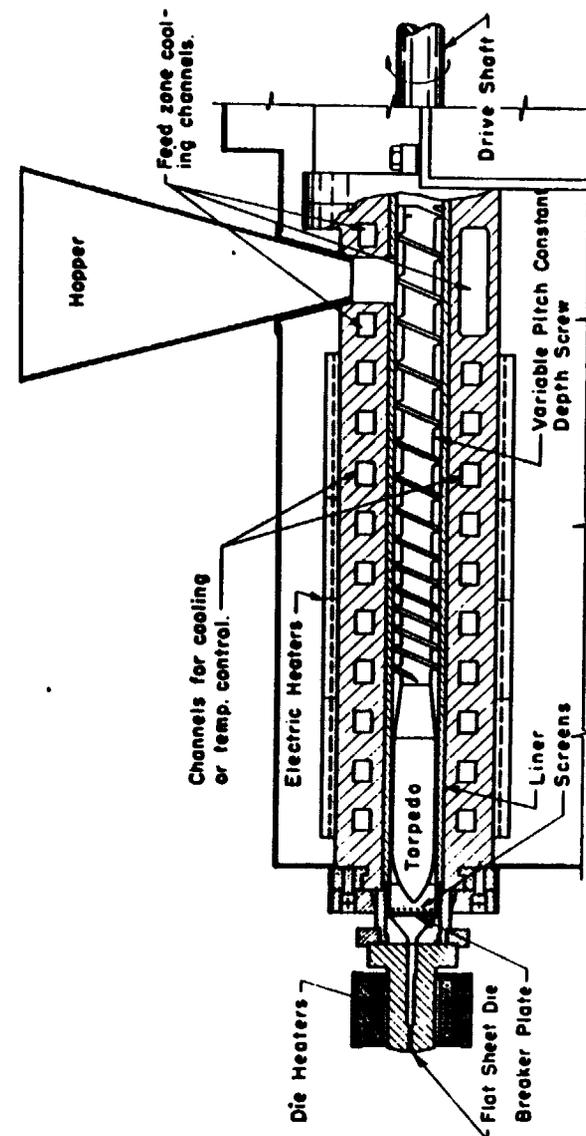


Fig. 6.2. Screw extruder (schematic). From Boundy and Boyer, *Styrene*, ACS no. 115, Reinhold Publishing Corp., New York, 1952.

screw diameter and speed. Under commercial conditions the approximate relation between extruder size, output and power requirement is shown in Table 6.1.³

TABLE 6.1
Relation Between Screw Diameter, Output, and Power Requirement

Screw diameter (in.)	2.5	3.5	4.5	6
Output (lb/hr)	200	250	500	625
Power (h.p.)	40	50	100	125

Output can be increased by higher melt temperatures and by increasing the L/D ratio which permits higher speed; there is, however, a speed limit because of the formation of excessive frictional heat. The quality and uniformity of extruded articles (particularly film) is superior at moderate outputs.

In most extruders external heat is supplied by electrical resistance heaters with temperature control by voltage regulation; a considerable amount of heat is generated internally by friction.

Screw designs recommended for PP are as follows:³

L/D 20–24, compression ratio 3.5–4, feed leads 9–11, transition leads 4–5, metering leads 7–8.

Lead depth for 2.5 and 6 in. diameter:

0.368–0.420 and 0.612–0.700 in. feed section.

0.105 and 0.175 in. metering section.

Screen packs of 80–100 mesh should be used to assure melt uniformity and retain gel particles. Long land lengths should be used (12 to 50 times the diameter) and the approach to the land should be long and shallow; a taper of 2–5° in the land seems desirable.⁴ The operating temperature range for extrusion operations of PP is between 200 and 300° C.^{3–5}

2(a) Pipes, Rods, Profiles, and Wire Coating

Pipes and tubing from PP can be extruded into sizes ranging from 1/8 to 8 inches in diameter. Barrel temperatures range

from 200 to 230° C. Long land dies are preferred with at least a length of 30 to 40 times the wall thickness. The most critical step in the extrusion of pipes and tubing is the sizing and cooling operation. The internal sizing method uses an internal cooled mandrel whereas the external method uses air pressure or vacuum to force or suck the outside diameter against a water cooled sleeve. Final cooling is accomplished by submerging the extruded pipe into a 10–20 feet long water bath. Biaxial orientation of pipes makes it possible to obtain a certain strength with less material.⁶ After cooling to about 95–120° C the pipe is reheated to about 130° C and stretched by internal pressure to the inside of a sleeve with a diameter 1.5 to 2 times the original pipe diameter; at the same time it is drawn longitudinally; typical stretching values are about 70% longitudinally as well as circumferentially. The strength of PP pipe is thereby increased about twofold and stiffened to more than double the original modulus.

The main point in the extrusion of rods is to prevent formation of voids: the operating temperature is kept at a minimum, i.e. about 200° C, low extruder speed and very high pressures are employed. The cooling process is performed very slowly (long cooling sleeve) and a brake is placed on the rod as it emerges from the cooling sleeve so as to maintain a back-pressure on the extruder; any voids that may have formed originally can be filled under the influence of the back-pressure.

In the extrusion of profiles maintaining uniform wall thickness in the entire cross section is the most critical design factor; profiles should be supported by cooled tools or air streams before final cooling in the water bath. A certain degree of draw (usually not exceeding 10%) is commonly practiced in extrusion operations.

For extrusion coatings of wire and cables^{3, 4} tapered land dies are recommended. The wire to be coated passes through a guider which corresponds to the pin of a pipe die. Preheating of the wire will improve adhesion of the coating. Draw-down of the coating from the die does not exceed 10% ordinarily. Gradual cooling is generally preferred: the first cooling bath is kept at about 95° C, subsequent cooling baths at lower temperatures. This procedure helps to prevent the formation of voids by shrinkage. The wire speed depends on the size of the wire and the type

and composition of the coating PP; speeds as high as 400 feet/minute can be achieved.

2(b) Sheet

PP is extruded into sheets (0.010–0.250 in. thickness)³⁻⁵ with equipment closely resembling that used for film extrusion (see section 2(c)). Melt temperatures between 220 and 250° C are suggested. For heavy gauge sheeting dies with a choker bar are used (adjustable restriction in the slot of the die) which is important for controlling sheet thickness. The sheet take-off equipment consists of three cooling rolls (Fig. 6.3). The temperature of the cooling rolls should be between 80 and 115° C.

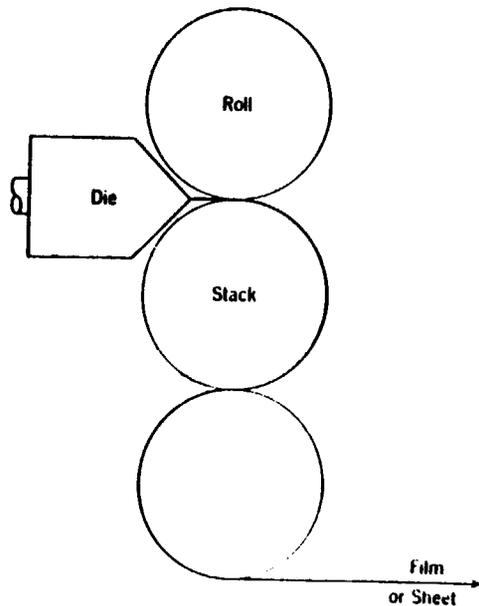


Fig. 6.3. Sheet extrusion (schematic)^A

2(c) Film

The blown-film process is shown schematically in Fig. 6.4. This process is most commonly used for branched ld. PE and less

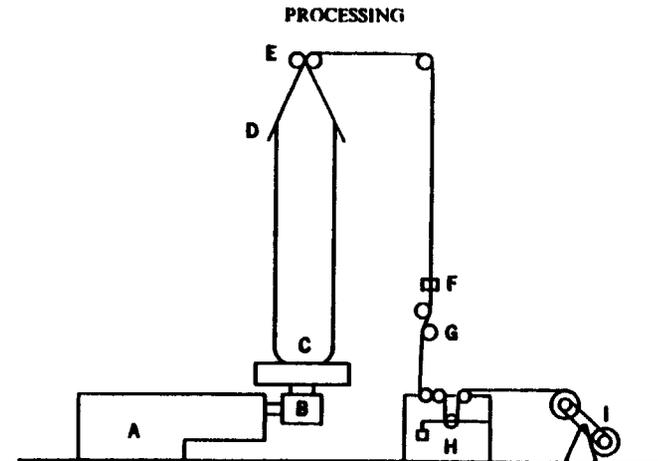


Fig. 6.4. Blown film process: A: extruder, B: die, C: air ring, D: collapsing guides, E: pinch rolls, F: edge sensing device, G: edge control rolls, H: tension control, I: roll up^A

frequently for linear hd. PE and it. PP. The not very effective cooling results in poor optical properties (haze; gloss and clarity imperfections) and the linear operating speeds are comparatively low. Only for heavy gauge, large diameter film will the production rate in lbs/hr be sufficiently economical in spite of low linear speed.

A great variety of dies are being used: side entry types as well as bottom entry types. The formed tube is cooled by a stream of air from an air ring; more rapid cooling may be effected by additional cooling from an internal mandrel. The pinch rolls fold the film bubble shut and regulate the take-off speed. Blown films will be oriented longitudinally by draw from the pinch rolls and transversely by blow-up.

Only heavy gauge PP-film (about 0.005–0.01 in.) and particularly polyblend films (PP with elastomer) are commonly produced by the blown-film method; extrusion temperatures range between 215–230° C.

The chill-roll process is the most practical method for the production of PP film;⁷ the film is formed by a die (0.001–0.01 in. thickness) and laid against a roll for quenching. This process

allows better gauge control than the blown-film process, produces better optical properties, and permits greater speeds (up to 500 feet/minute). Various designs for flat film dies are being used with variations in die lip and slot configurations; die lips should be tapered for close approach to roll surface, the land should be long with a gradual approach, die passage must be streamlined. Melt temperatures for film extrusion range between 245–300° C; die openings are usually set at 0.010–0.025 in. After the film has left the die lips it passes a very short draw distance (less than 1.5 in.) to the contact line on the chill roll which is cooled with water of a temperature not exceeding 15° C. Further improvement of film quality may be achieved by additional air knife chilling:⁸ air is fed through a duct and presses the molten polymer against the chill roll. This procedure is illustrated in Fig. 6.5.

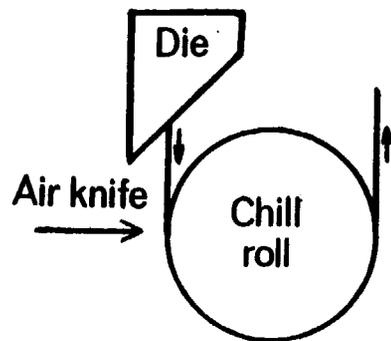


Fig. 6.5. Air knife chilling for PP-film (schematic)

Additional chill rolls may be used but only the first one is essential. Additional take-up equipment is similar to that used in the blown-film technique. The relation between a number of processing variables and film properties is shown in Table 6.2.

So far only unoriented films or films of low degree of orientation have been discussed. PP film is particularly suited for uniaxial and biaxial orientation, i.e., orientation longitudinally as well as transversely.¹⁰ The longitudinal (uniaxial) orientation is effected by passing the partly cooled film through a set of pull rolls into an

TABLE 6.2
Relation between Processing Variables and Film Properties:⁹
+ Increasing, - Decreasing, = Constant

	Gauge variation	Coeff. of friction	Clarity (%haze)	Tensile modulus	Gel count	Impact strength
Production rate	+	-	+	+	+	-
Draw distance	+	-	+	=	=	-
Melt temperature	=	+	-	=	=	=
Chill water temp.	=	-	+	+	=	-
Die opening	+	+	-	=	=	+

oven at 120–150° C and thereafter over another faster running set of pull rolls, thus drawing the film and orienting it (draw ratio 5–8). The transverse orientation is performed on a tenter frame (Fig. 6.6) consisting of two long, endless chains with a series of clips gripping the edges of the film. The tracks on which the chains are running diverge so that the clips pull the film sideways, thus producing transverse orientation (draw ratio up to 3); the tenter frame may be mounted in an oven to control the temperature at which orientation occurs.

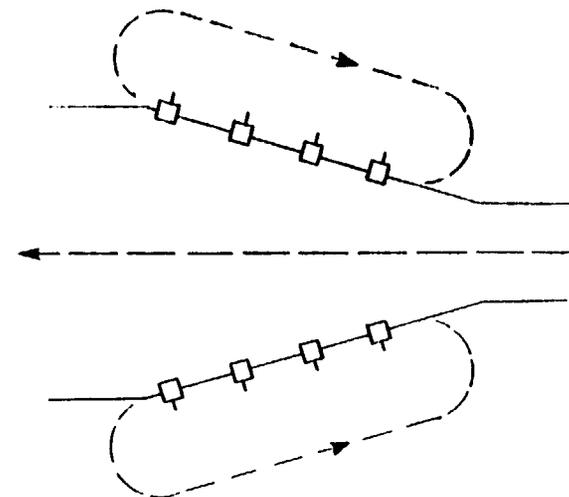


Fig. 6.6. Tenter frame for transverse orientation

Uniaxial orientation increases film strength about fivefold but only in the orientation direction; biaxial orientation increases film strength in all directions. Other changes are improved clarity, greater impact strength (particularly at low temperature), greater stiffness, etc.

2(d) Filaments

PP filaments are prepared by melt spinning which comprises two manufacturing stages: extrusion of filaments, subsequent thermal and mechanical stretching.

The extrusion processes employs ordinarily a round die with 60 to 80 orifices arranged in a circular pattern; for very fine filaments pump-type dies are used. The melt temperature is between 260 and 290° C. Screen packs are inserted in the extruder head to prevent foreign particles contaminating the extruded filaments.

The filaments are extruded downward into a quench tank (water filled) from where they go to pull rolls (godets), to a draw oven (125–160° C), to draw rolls, to an annealing oven (95–120° C), and finally to the wind-up. A schematic illustration of this set-up is shown in Fig. 6.7.

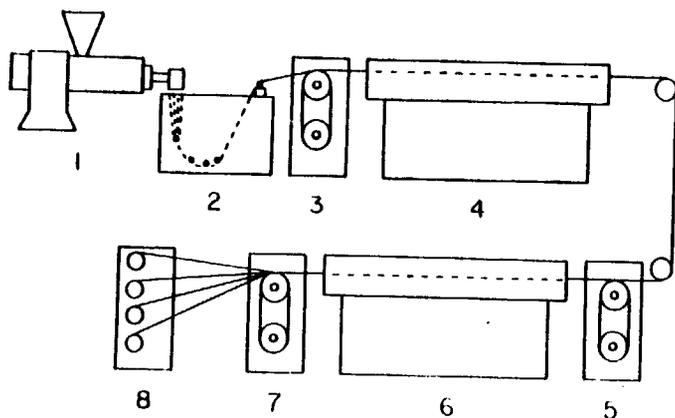


Fig. 6.7. Manufacture of PP filaments: 1: extruder, 2: quench tank 3: pull rolls, 4: draw oven, 5: draw rolls, 6: annealing oven, 7: relax rolls, 8: wind-up^B

The degree of orientation depends on the draw ratio, i.e. speed ratio of draw rolls to pull rolls, and is commonly chosen as 9/1. At this draw ratio the strength increase of the filament has reached a maximum. The subsequent annealing process relieves residual stresses and prevents shrinkage. The relations between a number of processing variables and filament properties are shown in Table 6.3.

TABLE 6.3
Relation between Processing Variables and Filament Properties^E
+ Increasing, - Decreasing, = Constant

Increasing	Tenacity (g/denier)	Elongation (ultimate)	Shrinkage
Draw ratio (4-30)	+	-	
Crystallinity (40-60%) [†]	-		
Molecular weight (melt index 20-0-6)	+	+	-
Melt temperature (220°-315° C)	-	-	-
Draw temperature (120-205° C)	-	+	-
Annealing temp. (100-160° C)	=	=	+

[†] Affected by varying efficiency of quenching.

3. Injection Molding

Injection molding is the most commonly used method for manufacturing formed articles of it. PP. PP can be molded in standard molding equipment. A schematic sketch of the essential parts of an injection molding machine is shown in Fig. 6.8.

The procedure in the course of a mold cycle is the following:¹¹ A charge of polymer material is allowed to drop into the rear part of the cylinder (ram retracted), the ram is then moving forward pushing hot molten polymer into the mold; as the mold fills the pressure builds up to a maximum. The ram is held forward until solidification occurs thereby sealing the mold. The ram is withdrawn and the pressure drops as the polymer cools; when the mold is opened the pressure has decreased to a small residual

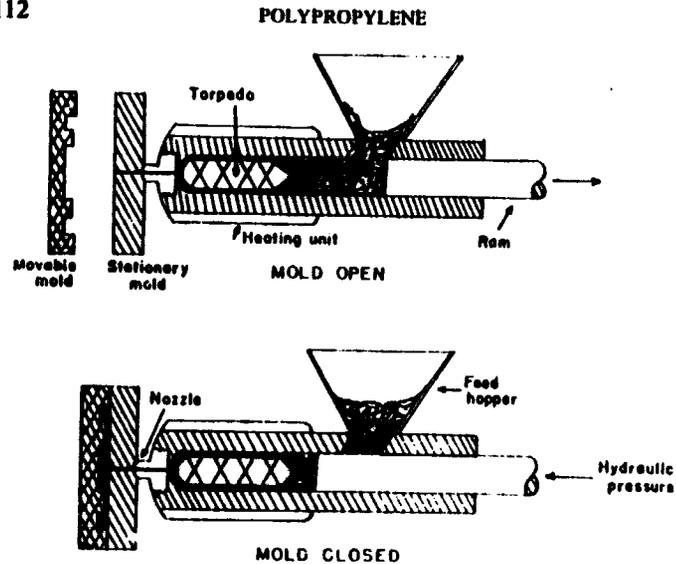


Fig. 6.8. Injection molding machine (schematic)

value. Injection time takes about half of the molding cycle; it begins at zero time and ends with the removal of the ram. Cure time lasts from there on till opening of the mold. Both together make up the molding cycle.

PP is molded at cylinder temperatures between 230–290° C, mold temperature is held below 65° C. Sufficient cure time must be allowed to cool the PP article below this temperature before opening the mold. The ram pressure is operated between 8000–15,000 psi, depending on the temperature (Fig. 6.9); the molding cycle lasts 20 to 40 seconds, depending on conditions. The main variables in injection molding are ram pressure, temperature of the polymer melt at the nozzle, molding cycle, and processability of the polymer material (melt index). The temperature–pressure relation is indicated on a typical molding area diagram (Fig. 6.9).

Linear mold shrinkage of injection molded PP articles varies between 0.5 and 2.5%; post shrinkage (after molding) is of the order of 0.4%. Other molding complications such as sink, warpage, voids, short shots, poor surface depend on processing variables and are shown in Table 6.4.

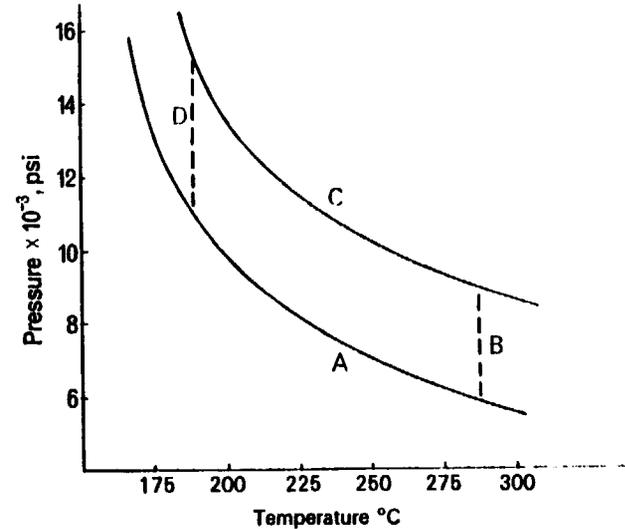
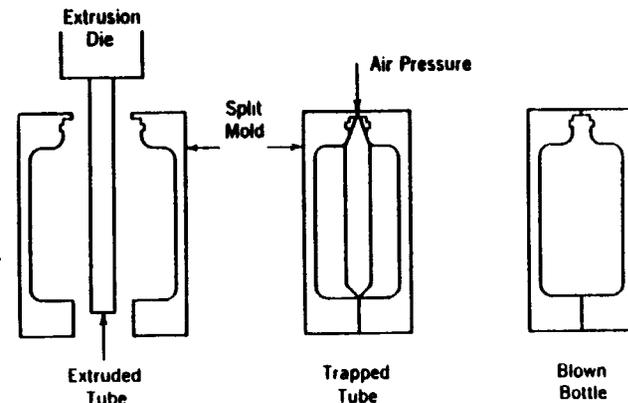


Fig. 6.9. Molding area diagram for it. PP; A: short line (incomplete filling of the mold), B: thermal decomposition, C: flow line (flow from the mold), D: flow marks, poor surface

Fig. 6.10. Blow molding process (schematic)^A

4. Blow Molding

The blow molding process¹³ is mainly used for the manufacture of bottles, containers, and other hollow articles; the equipment includes a conventional extruder and the blow molding set-up. A schematic illustration is shown in Fig. 6.10.

The parison, i.e. a molten tube of polymer, is formed from a heavy walled tube from a downward directed extruder die; the air can be blown through the die, or through the mold base, or through an inserted needle. Since extrusion is a continuous process while blowing is discontinuous, most machines are equipped with valves to direct polymer flow to other extrusion dies and molds while the first bottle is blown, cooled, and ejected. The extrusion temperature range for PP is 200 to 220° C, extrusion pressure is 2000 psi, compression ratio 3.5 to 4, L/D ratio 20 and over, blowing pressure 75-100 psi, mold temperature 15-25° C, shrinkage 2 to 5%. The molding cycle ranges between 10 and 30 seconds, depending on other processing conditions and it is shortened by increasing air pressure.

5. Miscellaneous Processing Methods

Other processing methods,¹⁴ which are to be mentioned only, include thermoforming (PP parts made from PP sheets in sandwich heaters), preparation of nonwoven fabrics (spun bonded PP material),¹⁵ lamination, fluidized bed coating, foaming,¹⁶ heat sealing of films,¹⁷ etc.

TABLE 6.4

Relation between Processing Variables and Molding Defects:¹²
+ Increasing, - Decreasing, = Constant

Increasing	Shrinkage	Sinks	Warpage	Voids	Poor surface	Short shots
Ram press. (8000-17,000 psi)	-	-	-	-	-	-
Stock temp. (230-315° C)	+	+	-	+	-	-
Mold temp. (25-95° C)	+	+	+	-	-	-
Injection time (5-40 sec.)	-	-	-	-	-	-
Cure time (5-40 sec.)	-	-	-	-	-	-
Gate size (0.05-0.2 in.)	-	-	-	-	-	-

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