

Foreword

The polymer and plastics industries have had a profound techno-economic impact on society for almost a century. In fact, it has been suggested that the advent and use of polymers and plastics products have represented a revolutionary technological change. They are used in packaging, furniture, construction materials, automotive, aerospace, sporting goods, biomedical, electronics, communications, and so on. More importantly, they have adapted to the ever changing social and technological demands. Thus, many of the current popular products, such as smart phones, computers, and other technological innovations would be difficult to contemplate in the absence of polymers. It does not seem likely that the foreseeable future will see a reduction in the important role that polymers and plastics will play in future technological development.

Cognizant of the role that polymers played and will continue to play in our lives, a group of polymer scientists and engineers from various countries around the world founded the Polymer Processing Society (PPS) in March 1985 at the University of Akron, Akron, Ohio, USA. According to its constitution, the goal of the PPS is to foster scientific understanding and technical innovation in polymer processing by providing a discussion forum in the field for the worldwide community of engineers and scientists. Thus, PPS has attempted to achieve this goal using the following mechanisms:

1. Organization of annual and regional conferences rotating among the various regions of the world and the dissemination of technical content of the conferences in the form of proceedings.
2. The publication of the International Polymer Processing (IPP) Journal.
3. The publication of the Progress in Polymer Processing (PPP) Series.

So far, these activities have allowed the PPS and its members to exchange information and ideas about the evolution of the principles and methods of polymer science and engineering and their application to the generation of innovative products, processes and applications.

Since the formation of PPS, eleven PPP volumes have been published. Four distinguished leaders in the polymer processing field have served as series editors: Leszek Utracki, Warren Baker, Kun Sup Hyun, and James L. White. Last summer at PPS 29 in Nuremberg, Germany, I was asked by the Executive Committee of PPS to serve as

PPP series editor. It is my hope, that with the help of the Advisory Editorial Board, our colleagues in the polymer processing field, and Hanser Publications, to publish at the rate of about one book every year. We already have two books under preparation. I encourage prospective authors to contact me or any of the Advisory Board members with their ideas and suggestions.

One of my first tasks has been to follow and expedite the completion of *Film Processing Advances*. This has given me the opportunity to refresh and expand my contacts with the editors, Drs. Kanai and Campbell, whom I have known for many years. As I have done some work in the area of film processing, I always benefited from reading their works and meeting them at conferences. Thus, it was easy to work together with them and with the publisher, Hanser, to set up the necessary mechanisms and procedures for a smooth and timely finish for this ambitious project. It is a real pleasure to have *Film Processing Advances* as the first PPP project completed during my first term as a series editor. Obviously, the credit goes to Professors Campbell and Kanai and to the contributors of the chapters for their tireless efforts. We also owe special thanks to the editorial staff at Hanser, especially Ms. Cheryl Hamilton, who handled the details of publication smoothly and efficiently.

As we all know, plastic films represent a major component of the polymer and plastics business. Plastic films are used extensively in packaging products. They have withstood and adapted to various pressures and requirements. Film processing technology continues to advance with the advent of improved extrusion and die design technologies, development of advanced film blowing and casting techniques; temperature, orientation, and crystallization control, and advanced computer simulation, monitoring and control systems. Thus, the publication of *Film Processing Advances*, by the same editors of the successful *Film Processing*, represents a timely technical update on the status of film processing technology.

Finally, on behalf of the Polymer Processing Society and the PPP Editorial Advisory Board, I would like to express our sincerest thanks and appreciation for Professor Gregory Campbell and Professor Toshitaka Kanai for the immense amount of effort, time, and dedication that they have contributed to the editing and preparation of this book. I also wish to thank the other authors for contributing their excellent chapters. Also, we owe a lot of thanks to Ms. Cheryl Hamilton and other Hanser staff for the organization of the copyediting of the book and timely completion of this project.

Musa R. Kamal
Series Editor

1

Extruder and Screw Design for Film Processing

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Single-screw extruders are the preferred machines for plasticating and metering resin to downstream film processes. The extruder must provide a homogenous and stable extrudate at high rates and at the target discharge temperature and pressure. Moreover, gels must be at a low and acceptable level. Gels are defined as any particle that creates an optical defect in the film. Because film products are typically very thin and in the range of 15 to 250 μm , very small particles can cause observable defects. In many cases, these particles are created in the extruder, and thus screw design can be used to mitigate gels from the final film product.

This chapter will describe the single-screw extrusion process typically used for film processes, common screw designs, troubleshooting operations, and common gel defects that originate from the extruder. In-depth operation and fundamentals of the process are beyond the scope of this writing. The reader can learn more about the fundamentals of single-screw extrusion and troubleshooting in reference 1.

■ 1.1 The Extrusion Process

All single-screw extruders have several common characteristics. The main sections of the extruder include the barrel, a screw that fits inside the barrel, a motor drive system for rotating the screw, and a control system for the barrel heaters and motor speed. A schematic for an extruder is shown in Fig. 1.1. Many innovations on construction of these components have been developed by machine suppliers over the years. A hopper is attached to the barrel at the entrance end of the screw, and the resin is typically fed by gravity (flood fed) into the feed section of the screw. The resin is typically purchased in pellet form (most polyolefins for example), but powders are common (PVDC resin). Recycle film from edge trim is often chopped and metered into the hopper. The extruder screw must first convey the pellets away from the feed opening, melt the resin, and then pump and pressurize it for a downstream filming process. This type of machine is referred to as a plasticating, single-screw extruder. The barrel is usually heated with a minimum of three temperature zones. These different temperature zones are consistent with the three functions of the screw: solids conveying, melting, and pumping or metering of the resin.

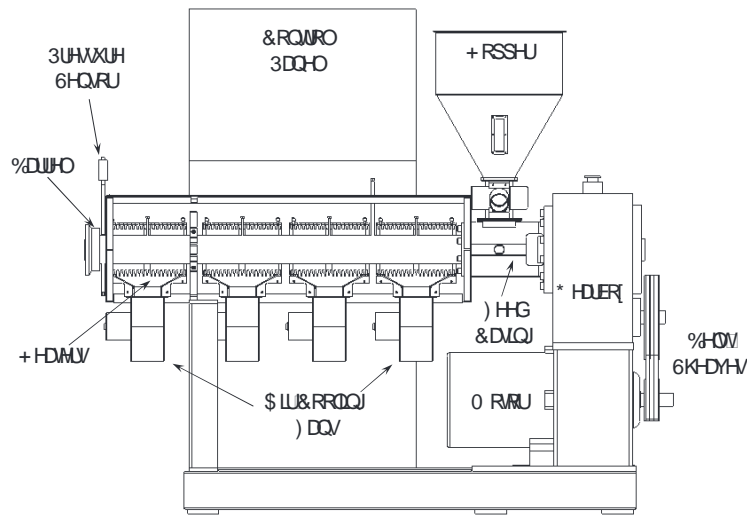


Figure 1.1 Schematic of a typical plasticating, single-screw extruder with a smooth-bore feed section. The extruder is equipped with four barrel heating and cooling zones and a combination belt sheave gearbox speed reduction drivetrain (courtesy of William Kramer of American Kuhne)

The single-screw plasticating process starts with the mixing of the feedstock materials. Typically, several different feedstocks are added to the hopper such as fresh resin pellets, recycle material, additives, and a color concentrate. Often these components need to be blended prior to adding them to the hopper. Next, the feedstock flows via gravity from the hopper through the feed throat of the feed casing and into the solids conveying section of the screw. Typically this feed casing is cooled using water. The feed section of the screw is typically designed with a constant depth and is about 4 to 8 barrel diameters in axial length. Directly after the solids conveying section is a section where the channel depth tapers to a shallow-depth metering section. The tapered depth section is commonly referred to as the transition or melting section. In general, the metering section is also a constant depth, but many variations exist where the channels oscillate in depth. The metering section pumps and pressurizes the material for the downstream unit operations including static mixers, screen filtering devices, and dies. The total length of the extruder screw and barrel is typically measured in barrel diameters or as a length-to-diameter (L/D) ratio. Section lengths are often specified in barrel diameters or simply diameters.

A conventional single-flighted screw is shown in Fig. 1.2. This screw has a single helix wound around the screw root or core. Multiple-flighted screws with two or more helices started on the core are very common on high-performance screws. Screws with barrier flighted melting sections are very common in film processes because they provide high rates with lower discharge temperatures. Barrier melting sections have a secondary barrier flight that is located a fraction of a turn downstream from the primary flight, creating two flow channels in the transition section:

a solids melting channel and a melt conveying channel. Barrier flighted sections will be discussed in more detail later. Many high-performance screws [1] have two or more flights in the metering section of the screw. The screw is rotated by the shank using either specially designed splines or by keys with rectangular cross sections. The mathematical zero position of the screw is set at the pocket where the screw helix starts. Most extruder manufacturers rotate the screw in a counterclockwise direction for viewers positioned on the shank and looking towards the tip. This rotation convention, however, is not standard.

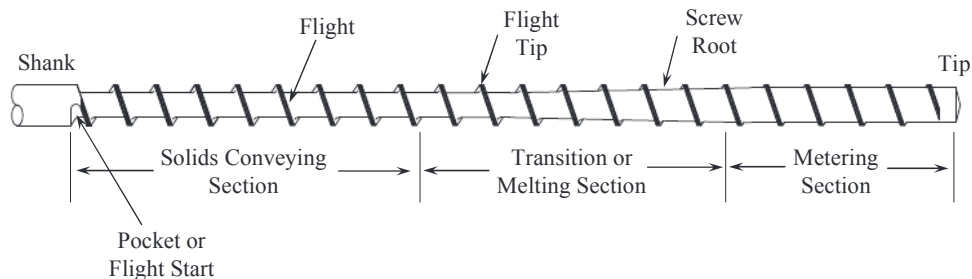


Figure 1.2 Schematic of a typical single-flight screw (courtesy of Jeff A. Myers of Robert Barr, Inc.)

The flight is a helical structure that is machined into the screw and extends from the flight tip to the screw core or root. The flight has a width at the flight tip called the flight land. The small clearance between the flight land and the barrel wall minimizes the flow of resin over the land. The polymer that does flow between the clearances supports the screw and centers it in the barrel. The radial distance between the flight tip and the screw root is referred to as the local flight height or channel depth. The feed section usually has the largest channel depth and provides the largest cross-sectional volume in the screw. The deep channel conveys the relatively low bulk density feedstock pellets into the machine via the motion of the helix. The feedstock is conveyed forward into the transition section or melting section of the screw. The transition section increases in root diameter in the downstream direction, and thus the channel depth is decreasing. Here, the feedstock is subjected to higher pressures and temperatures, causing the feedstock to compact and melt. As the material compacts, its bulk density can increase by a factor of nearly two or more. As the feedstock compacts, the entrained air between the pellets is forced backwards and out through the hopper. For example, a pellet feedstock such as low density polyethylene (LDPE) resin can have a bulk density at ambient conditions of 0.58 g/cm^3 , while as a fully compacted solid bed in the transition section the density will approach 0.92 g/cm^3 before melting starts. Thus for every unit volume of resin that enters the extruder, about 0.4 unit volumes of air must be expelled through the voids in the solid bed and then discharged through the hopper. The transition section is where most of the polymer is converted from a solid to a fluid. The fluid is then conveyed to the metering section where the molten resin is pumped to the

discharge opening of the extruder. In general, the metering section of a conventional screw has a constant root diameter, and it has a much smaller channel depth than the feed section. The ratio of the channel depth in the feed section to the channel depth in the metering section is often referred to as the compression ratio of the screw.

The transition section shown in Fig. 1.2 is a conventional single-flighted design. These designs are still used for film operations, but barrier flighted melting sections are much more common. Barrier flighted melting sections will typically provide higher rates, lower discharge temperatures, a more stable discharge pressure, and extrudates that have fewer gels due to poor mixing. Barrier flighted melting sections are constructed by positioning a second flight (or barrier flight) in the transition section such that the solids are maintained on the trailing side and the molten resin on the pushing side. A schematic of a barrier flighted screw with an Egan (or spiral Maddock-style) mixer is shown by Fig. 1.3. A schematic of a cross section of a barrier melting section is shown in Fig. 1.4. The resin that is melted near the barrel wall is conveyed across the barrier flight and collected in the melt conveying channel. The key design parameters include the position of the barrier flight, the depths of the channels, and the undercut clearance on the barrier flight. The undercut clearance is measured by positioning a segment of straight bar stock across the two main flights and then measuring the gap between the bar and the barrier flight land. For most designs, the barrier flight undercut is constant for the entire length of the section. As a very general rule, the undercut is typically about 0.01 times the diameter of the screw. Undercuts that are smaller than this rule, however, are often used. The position of the barrier flight sets the width of both channels. Many styles of barrier melting screws are commercially available, and many different variations of the channel widths and depths are used commercially.

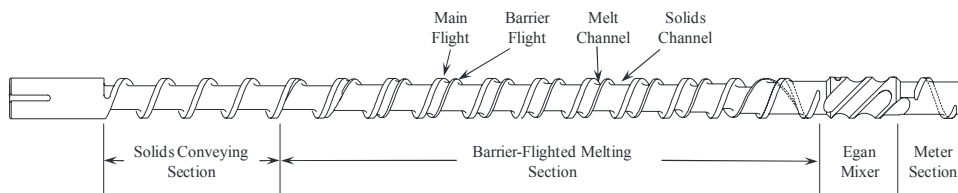


Figure 1.3 Schematic of a Steward barrier flighted screw with a downstream dispersive (Egan) mixer (courtesy of William Kramer of American Kuhne)

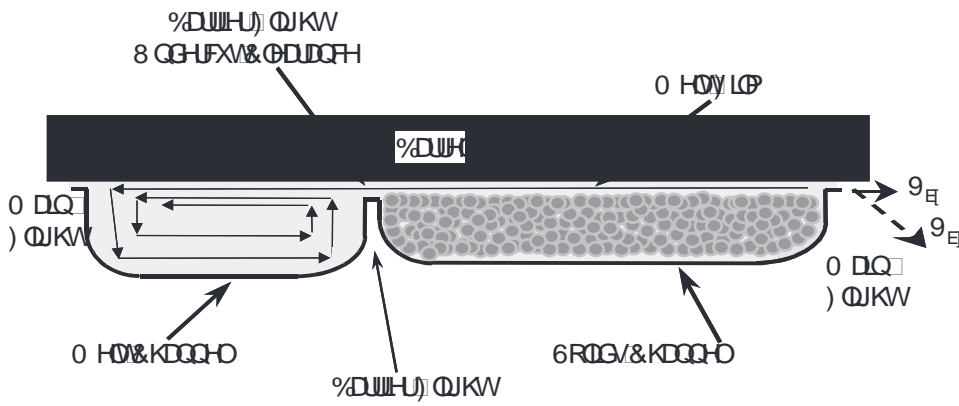


Figure 1.4 Cross-sectional view of a barrier melting section

Solid polymer fragments often exit the solids channel of barrier sections, or they can be discharged from conventional melting sections, especially at high rates and screw speeds. These solids need to be trapped and dispersed before the extrudate is shaped into film. Maddock-style mixers are typical dispersive mixers that are used for this application, but other mixers or high-performance sections are used. An Egan mixer (spiral Maddock-style mixer) is shown in Fig. 1.3 while other Maddock mixer styles are shown in Fig. 1.5.

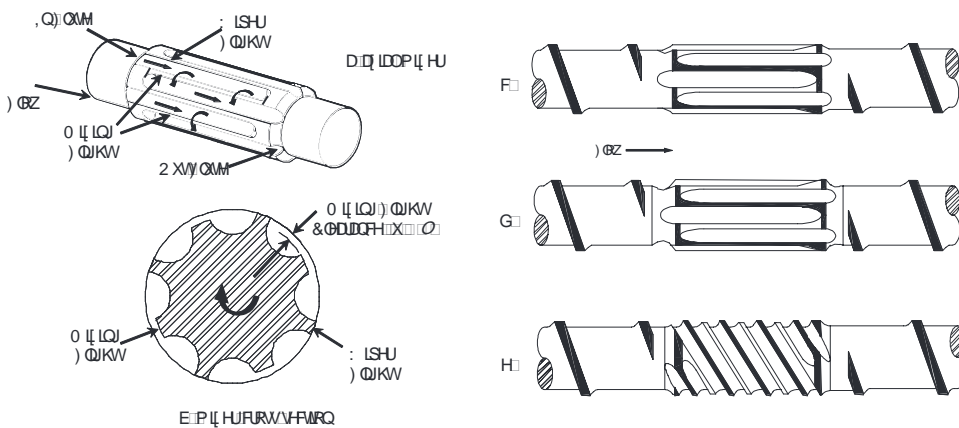


Figure 1.5 Schematic for Maddock-style mixers. (a) A mixer with the flutes aligned in the axial direction; (b) a cross-sectional view perpendicular to the screw axis showing the clearance for the mixing flight; (c) a mixer with the flutes aligned in the axial direction; (d) an axial mixer with pressure relief zones at the entry and exits; (e) an Egan mixer with spiral flutes (courtesy of Jeff A. Myers of Robert Barr, Inc.)

Maddock-style mixers [2] are very commonly used due to their low cost to manufacture and their ability to disperse solid fragments, trap and melt polymer solids, and mitigate color and compositional gradients. Many styles are on the market under two basic types: (1) flutes parallel to the screw axis, and (2) flutes in a spiral pattern

in the same direction as the flights. Schematics for these devices are shown by Fig. 1.5. For small-diameter screws, the mixer is generally constructed with four inflow flutes (or channels) and four outflow flutes. Larger diameter screws will have more paired flutes due to the larger available area at the screw circumference. For a Maddock mixer with the flutes parallel to the axis of the screw, molten polymer flows into the inflow flutes via a pressure gradient and then either continues to flow downstream in the flute or is passed through a small clearance between the mixing flight and the barrel wall. This small clearance is responsible for providing the dispersive mixing characteristics of the device. Screw manufacturers typically specify the mixer flight height position relative to the main flight as an undercut. The undercut u for a 63.5 mm diameter screw is typically about 0.5 to 1.2 mm, although for some applications and designs the clearance can be smaller. For this size screw with an undercut of 0.50 mm and a flight clearance of 0.07 mm, the clearance between the mixing flight and the barrel wall is 0.57 mm. The material that flowed across the mixing flight is accumulated in the outflow flute and is then flowed via pressure to the discharge end of the mixer. The wiper flight shown in Fig. 1.5 is set at the same height as the flight in the metering section. For mixers with the flutes in a spiral pattern, some of the forwarding flow in the flutes is due to the rotational movement of the flute relative to the barrel wall. Performance and simulation details can be found in the references [3, 4].

The specification of the undercut on the mixing flight for Maddock-style mixers is critical to its performance. As previously stated, all material must flow through the clearance provided by the sum of the undercut and flight clearance. If the clearance is too large, some medium- and small-size solid polymer fragments will not be trapped and melted by the device. If the clearance is too small, then a high-pressure gradient can occur, and there exists the possibility of increasing the temperature of the resin beyond its thermal capabilities, that is, causing degradation. The shear stress that the material experiences for flow across the mixing flight of the mixer can be estimated using Eqs. 1.1 and 1.2. The shear stress level is responsible for breaking up agglomerates and dispersing solid polymer fragments. A higher shear stress level will improve the ability of the mixer to disperse smaller size fragments. This shear stress calculation is based on screw rotation physics and is as follows:

$$\dot{\gamma}_M = \frac{\pi(D_b - 2u - 2\lambda)N}{(u + \lambda)} \quad (1.1)$$

$$\tau_M = \eta\dot{\gamma}_M \quad (1.2)$$

where $\dot{\gamma}_M$ is the average shear rate for flow over the mixing flight in 1/s, u is the undercut clearance on the mixing flight, λ is the mechanical clearance of the flights, N is the screw rotation rate in revolutions/s, η is the shear viscosity at the temperature of the mixing process and at shear rate $\dot{\gamma}_M$, and τ_M is the shear stress that the

material will experience for flow over the mixing flight. The stress level for flow across the mixing flight is typically between 50 and 200 kPa.

Several other design factors are important for the correct operation of Maddock-style mixers. These include the positioning of the mixer downstream from the melting section, the distance between where the meter flight ends and the mixer starts, and the elimination of resin stagnation regions. The mixer must be positioned on the screw downstream far enough such that only low levels of solid polymer fragments exist. If the level of solids is too high in the stream, then the fragments may be melted and dispersed at a rate slower than the rate of the entering solids, causing the mixer to become plugged with solids and reducing the rate of the machine. As shown in Fig. 1.5, the mixer should be positioned about 0.3 to 0.5 diameters away from the end of the upstream metering section flight. This creates an annular gap where the material is allowed to flow evenly into all inflow flutes of the mixer. The annular gap is often undercut as shown by Fig. 1.5(d). If the flights extend close to the mixer entry, then it is possible that the inflow flute near the trailing side of the flight will not operate completely full of resin and thus may cause the resin to stagnate and degrade. Moreover, flute depths should be streamlined and shallower at the entry end of the outflow flute and the exit end of the inflow flute. A common design error is to make these regions too deep, creating stagnation regions and causing resin degradation.

As an example of process design, linear low density polyethylene (LLDPE) resin is commonly used for blown film, cast film, and extrusion coating processes. Even though the resin grades are similar (melt indices or MIs do vary) for these three processes, the extrusion equipment is significantly different due to the requirements of the die and downstream equipment. The blown film process requires an extrudate that is relatively low in temperature and typically in the range of 200 to 220°C. In order to plasticate and produce an extrudate in this temperature range the metering channel is relatively deep and the screw would be designed to rotate at speeds less than 100 rpm. For a 150 mm diameter screw with a square-pitch lead length ($L = D_b$), the metering channel depth (H) would be between 9 and 12 mm. The cast film process requires an extrudate that is slightly higher in temperature and typically in the range of 240 to 260°C. The same 150 mm diameter screw would have a metering channel depth of 6 to 9 mm and the screw would rotate at higher speeds. The extrusion coating process requires an extrudate that is very high in temperature and often approaching 310°C. Here the metering channel depth would typically be about 3 to 4 mm for the 150 mm diameter screw, and the screw would be designed to rotate at very high speeds up to 230 rpm. These examples clearly show that the extrudate temperatures are set in part by the geometry of the metering section channel, the conditions of the process, and the MI of the resin.

The specific rate is often a good measure of the relative discharge temperature for a process. The specific rate is simply the rate divided by screw speed. For the exam-

ples above, the specific rate for the metering channels would be the highest for the blown film screw with the deep metering channel and the lowest for the extrusion coating process screw with the very shallow channel. Thus as a general guideline, the extrudate temperature decreases when the specific rate of the screw increases at constant barrel diameter. The specific rates for the screws in these examples increased because the channel depth increased. The specific rate could also be changed by adjusting the lead length. The calculated specific rotation rate can be also used as a similar guideline for discharge temperature. This guideline, however, can be violated if the channel is extremely deep and a large positive pressure gradient exists [1].

■ 1.2 Rate Calculation

For smooth-bore extruders, the rate of the extruder is controlled by the metering section of the screw. The expected rate for the process can be calculated based on the geometry of the metering channel, screw speed, pressure gradient, and the melt density and shear viscosity for the resin. The basic screw geometry for a single-flighted ($p = 1$) metering channel is shown in Fig. 1.6.

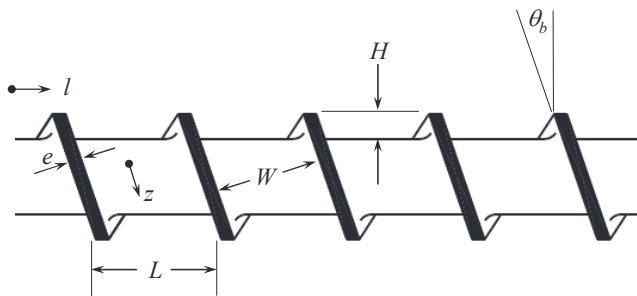


Figure 1.6 Geometric parameters for a single-flighted screw in the wound state

Two driving forces for flow exist in the metering section of the screw. The first flow is due just to the rotation of the screw and is referred to as the rotational flow component. The second component of flow is due to pressure gradients that exist in the z direction, and it is referred to as pressure flow. The sum of the two flows must be equal to the overall flow rate. The overall flow rate, Q , the rotational flow, Q_d , and the pressure flow, Q_p , for a constant depth metering channel are related as shown in Eq. 1.3. The subscript d is maintained in the nomenclature for historical consistency even though the term is for screw rotational flow rather than the historical drag flow concept. The method described here was developed by Rowell and Finlayson [5, 6] and later modified by Tadmor and Klein [7].

$$Q = Q_d - Q_p \quad (1.3)$$

The volumetric rotational flow term (Q_d) depends on the several geometric parameters and rotation speed. Since most extruder rates are measured in mass per unit time, the term Q_{md} is defined as the mass rotational flow:

$$Q_d = \frac{\rho V_{bz} W H F_d}{2} \quad (1.4)$$

$$Q_{md} = \frac{\rho \rho_m V_{bz} W H F_d}{2} \quad (1.5)$$

where ρ_m is the melt density at the average fluid temperature of the resin, V_{bz} is the z component of the screw velocity at the flight tip, W is the average width of the channel, p is the number of flight starts, H is the depth of the channel, and F_d is the shape factor for plane couette flow. The analysis using plane couette flow does not take into account the effect of the flights (channel helix) on the flow rate. For an infinitely wide channel, no flights, F_d would be equal to 1. As the channel width approaches the height, F_d is about 0.5. It is important to include the shape factors when evaluating commercial screw channels. This becomes extremely important for deep channels where H/W does not approach zero. An additional correction factor can be used to improve the calculation of the rotational flow term [1]. The shape factors are calculated as follows:

$$F_d = \frac{16W}{\pi^3 H} \sum_{i=1,3,5,\dots}^{\infty} \frac{1}{i^3} \tanh\left(\frac{i\pi H}{2W}\right) \quad (1.6)$$

The velocity of the flight tip V_{bz} is calculated as follows:

$$V_{bz} = \pi N D_b \cos \theta_b \quad (1.7)$$

$$\tan \theta_b = \frac{L}{\pi D_b} \quad \text{thus} \quad \theta_b = \arctan \frac{L}{\pi D_b} \quad (1.8)$$

where N is the screw rotation rate in revolutions per second, D_b is the diameter of the inside barrel wall, L is the lead length, and θ_b is the helix angle at the barrel wall.

Because of the helical nature of the screw, the width of the channel is narrower at the core of the screw as compared to that at the barrel wall. The calculation of the rotational flow rate, however, requires the average width of the channel. The average width of the channel is calculated as follows:

$$W_b = \frac{L}{p} \cos \theta_b - e \quad (1.9)$$

$$W_c = \frac{L}{p} \cos \theta_c - e \quad (1.10)$$

$$\tan \theta_c = \frac{L}{\pi(D_b - 2H)} \quad \text{thus} \quad \theta_c = \arctan \frac{L}{\pi D_c} \quad (1.11)$$

$$W = \frac{W_b + W_c}{2} \quad (1.12)$$

where e is the width of the flight perpendicular to the edge, D_c is the diameter at the screw core, W_c is the channel width at the screw core, and θ_c is the helix angle at the screw core.

The volumetric pressure flow term, Q_p , and the mass flow pressure flow term, Q_{mp} , are computed as follows:

$$Q_p = \frac{pWH^3F_p}{12\eta} \left[\frac{\partial P}{\partial z} \right] \quad (1.13)$$

$$Q_{mp} = \frac{p\rho_mWH^3F_p}{12\eta} \left[\frac{\partial P}{\partial z} \right] \quad (1.14)$$

$$F_p = 1 - \frac{192H}{\pi^3W} \sum_{i=1,3,5,\dots}^{\infty} \frac{1}{i^5} \tanh \left(\frac{i\pi W}{2H} \right) \quad (1.15)$$

where F_p is the shape factor for pressure flow, $\partial P/\partial z$ is the pressure gradient in the channel in the z direction, and η is the shear viscosity of the molten polymer at the average channel temperature and at an average shear rate, $\dot{\gamma}$:

$$\dot{\gamma} = \frac{\pi D_c N}{H} \quad (1.16)$$

The shear rate in the channel contains contributions from the rotational motion of the screw and the pressure-driven flow. The calculation of the shear rate, $\dot{\gamma}$, using Eq. 1.16, is based on the rotational component only and ignores the smaller contribution due to pressure flow.

The relationship between the pressure gradient in the z direction to the axial direction, l , is as follows:

$$\frac{\partial P}{\partial z} = \frac{\partial P}{\partial l} \sin \theta_b \quad (1.17)$$

The pressure gradient is generally unknown, but the maximum that it can be for a single-stage extruder screw is simply the discharge pressure, P_{dis} , divided by the helical length of the metering section. This maximum gradient assumes that the pressure at the start of the metering section is zero. For a properly designed pro-

cess, the actual gradient will be less than this maximum, and the pressure at the start of the metering section will not be zero.

$$\frac{\partial P}{\partial z} = \frac{P_{dis} \sin \theta_b}{l_m} \quad (1.18)$$

where l_m is the axial length of the metering section.

The total mass flow rate, Q_m , is calculated by combining the flow components as provided in Eq. 1.19 for the total mass flow rate. An additional correction factor can be used to improve the calculation of the rotational flow term [1].

$$Q_m = \frac{p\rho_m V_{bz} WHF_d}{2} - \frac{p\rho_m WH^3 F_p}{12\eta} \left[\frac{\partial P}{\partial z} \right] \quad (1.19)$$

Estimation of the rate and pressure gradient using Eq. 1.19 should be performed for each troubleshooting operation. Examples for its use are available elsewhere [1].

For grooved-bore extruders, the design of the feed section including the grooves and screw section control the rate of the process. This calculation of the rate is considerably more complex and is out of the scope of this chapter. Grooved bore extruders are discussed in detail elsewhere [8–10].

■ 1.3 Gels

A common contaminant in polyolefin film products is gels. The term gel is commonly used to refer to any small defect that distorts a film product, creating an optical distortion. There are many types of gels [11, 12], and the most common include (1) gels that are crosslinked via an oxidative process, (2) highly entangled resin gels (undispersed but not crosslinked), (3) unmelted resin, and (4) a different type of resin or contaminant such as wood, cloth fibers, or dirt. A crosslinked resin gel is typically formed during an oxidation process, resulting in the crosslinking of the resin chains and the generation of color bodies. These gels will not melt fully during analysis using a hot-stage microscope. Highly entangled gels are typically high molecular weight polymer chains that are highly entangled and thus difficult to disperse during the extrusion process. When analyzed using a hot-stage microscope, this gel type will melt as the stage temperature is increased. When the stage temperature is then decreased, the gel will crystallize before the surrounding material, creating the appearance of a gel. Since these gels are not oxidized they are not associated with color. They are commonly referred to as undispersed or unmixed gels. Unmelted resin exiting with the discharge can sometimes occur, especially at high extrusion rates. These gels will melt during heating with a hot-stage microscope,

and typically they will not re-form during the cooling phase. Numerous sophisticated methods are available for analyzing gels, including epi-fluorescence microscopy, polarized light microscopy, and electron microscopy with X-ray analysis.

Gels can be generated from many different sources and include (1) the resin manufacturer, (2) the converting process, (3) pellet blending of resins with significantly different shear viscosities, (4) pellet blending of different resin types, and (5) direct contamination. Modern resin manufacturing processes exclude oxygen from the system and are very streamlined such that process areas with long residence times do not exist. As such crosslinked and oxidative gels are likely not generated by the manufacturer. Improperly designed extrusion equipment and processes, however, are common, leading to the oxidative degradation of resins and crosslinked gels. Several case studies in the next sections show how poorly designed processing equipment can lead to crosslinked and unmixed gel contamination of products.

Established protocols for gel analysis in polymer films are well documented in the literature [11–14]. Typically a film with defects is visually inspected using a low power dissecting microscope. The gels can be classified based on size, color, and shape and isolated using a razor blade or scissors. Cross sections of the gels ranging from 5 μm to 10 μm thick are collected at temperatures below the glass transition (T_g) temperature of the film using a cryogenic microtome, about -80°C to -120°C . For optical examination, a thin section containing the gels is placed on a glass microscope slide with a drop of silicone oil and covered with a glass cover slip. Additional sections are collected for examination via hot-stage microscopy and for compositional identification if needed.

After collecting the sections, the remaining polished block-face containing the remainder of the gel is retained. In many instances, gels arise from inorganic contaminants such as the metals from pellet handling equipment, extruders, or components from masterbatches. Examination of these inorganic components are best performed with the block-face sample using a scanning electron microscope (SEM) equipped with an energy dispersive X-ray detector (EDX) [15, 16]. In some cases, additives or inorganic residues are present in low concentrations within the gels. A method to enrich the concentration of these materials is to expose the block-face containing the gel to oxygen plasma. Etching will preferentially remove the polymer at a much faster rate than the inorganic materials, enriching these components for elemental analyses. It must be noted that prior to SEM and EDX analyses, a thin conductive coating like carbon is typically evaporated onto the sample to render it conductive under the electron beam.

The most common type of gel is caused by oxidative processes that crosslink the PE chains. The best way to identify this gel type is by observation with polarized light and ultraviolet (UV) light sources. Transmitted polarized light microscopy represents an effective technique [17] that can be used to investigate structures in crys-

talline films. For example, black speck gels were contaminating a multilayer film product. The gels were relatively brittle when cut for analysis. The source was unknown. The detail of the gel is clearly visible using transmitted polarized light, as shown in Fig. 1.7(a). Close examination of this gel using epi-fluorescence with an ultraviolet light source caused an intense fluorescence emission, as shown in Fig. 1.7(b). This type of emission suggests thermal oxidation and crosslinking of the polymer. Microinfrared analysis of the gel indicated that it contained oxidized PE and maleic anhydride [1, 12]. This material likely formed on the metal surfaces of the extruder and then flaked off during a minor process instability. The material then flowed downstream and contaminated the film as a gel.

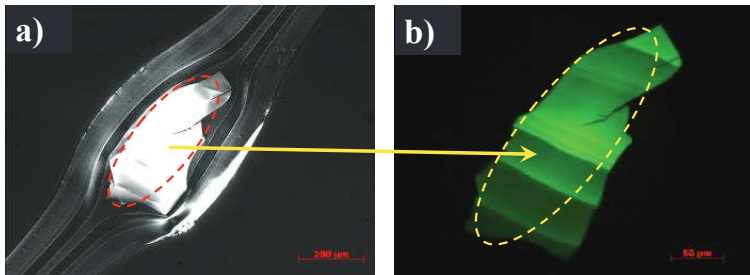


Figure 1.7 Transmitted polarized light images of a thermally oxidized and crosslinked gel in a multilayer film: (a) photograph in polarized light, and (b) the gel fluorescing under UV light [12]. Photographs were provided by E. Garcia-Meitin of The Dow Chemical Company

Crosslinked gels are oxidized gels, but the level of oxidation is not enough to cause them to fluoresce under UV light. The gels may have a level of crystallinity and thus be birefringent under polarized light. For example, the slightly birefringent gel shown in Fig. 1.8(a) was studied using a temperature-programmable, hot-stage, polarizing light microscope [16]. The optical melting temperature (T_m) of the gel was measured at 128°C and consistent with the PE used to make the product, as shown in Fig. 1.8(b). To determine if the gel was unmixed (highly entangled but not crosslinked), the gel was held above the melting temperature (135°C) and then stressed. A dental tool was used to stress the top of the glass slip cover. Crosslinked gels will appear birefringent, as shown in Fig. 1.8(c), in response to the anisotropy of stress distribution in the gel to polarized light. The gel dimensions and shape remained after cooling, verifying crosslinking, as shown in Fig. 1.8(d). If the gel was highly entangled and not crosslinked (unmixed gel), the gel would have disappeared after stress and cooling were applied.

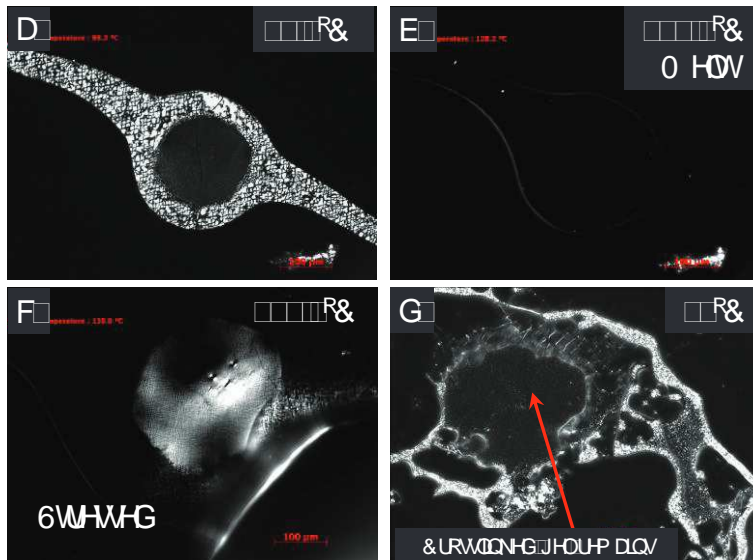


Figure 1.8 Hot-stage microscopy of a crosslinked gel in a crystalline monolayer film: (a) below the melting temperature, (b) optical melting point at 128°C, (c) appearance of birefringence after stressing at 135°C, and (d) intact crosslinked gel after cooling to 30°C [12]. Photographs were provided by E. Garcia-Meitin of The Dow Chemical Company

The origin of defects causing discoloration in polyolefin pellets can be identified using light and electron microscopy. For example, PE pellets from an in-plant recycle repelletizing process contained pellets that were off-color and had black specks, as shown in Fig. 1.9(a). One of these defects was isolated using the cross-sectioning technique, as shown in Fig. 1.9(b). The cross section revealed an intense reddish particle that caused the discoloration of the pellet. SEM and EDX microanalyses were used to determine that the defects contained primarily iron and oxygen, and it likely was iron oxide. A backscatter electron image (BEI) of the pellet block-face sample showed the defect causing the discoloration, and the elemental spectrum was shown to be iron oxide [1]. Metal-based defects can originate from process equipment, railcars used for shipment, pellet transfer lines, and poor housekeeping. The origin of the iron oxide was likely from a storage bin.

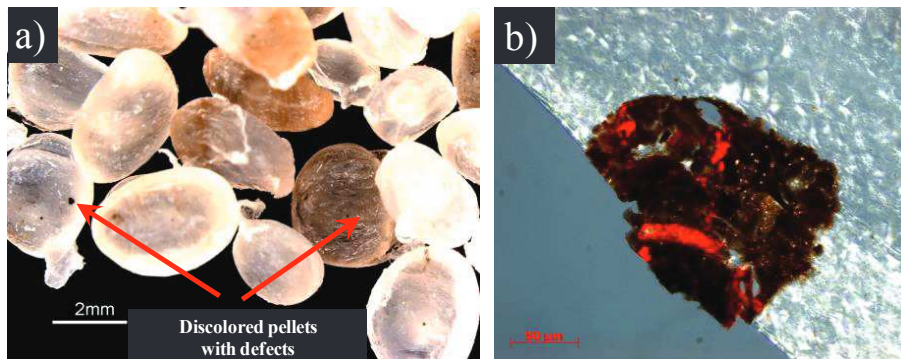


Figure 1.9 Photographs of foreign contamination in pellets of a repelletized reclaim stream: (a) photomicrograph of discolored PE pellets containing dark defects, and (b) transmitted polarized light micrograph of a pellet cross section containing a defect [12]. Photographs were provided by E. Garcia-Meitin of The Dow Chemical Company

In another example, a multilayer film product was experiencing occasional gels. The gels were isolated and the cross sections were collected as shown in Fig. 1.10(a). These gels contained highly birefringent particles that resided in the core layer. The outer film layers appeared amorphous, and the core layer was slightly birefringent. The optical melting temperature of the core layer was determined to be 123°C while the birefringent gels melted at 265°C. The melting temperature of 123°C was consistent with the polyethylene (PE) resin used to produce the core layer. The higher melting temperature of the material and microinfrared analyses of the defects indicate that they were foreign contaminants, and they were identified as a polyester resin. The polyester resin was used in another process in the converting plant, and it inadvertently contaminated the PE feedstock.

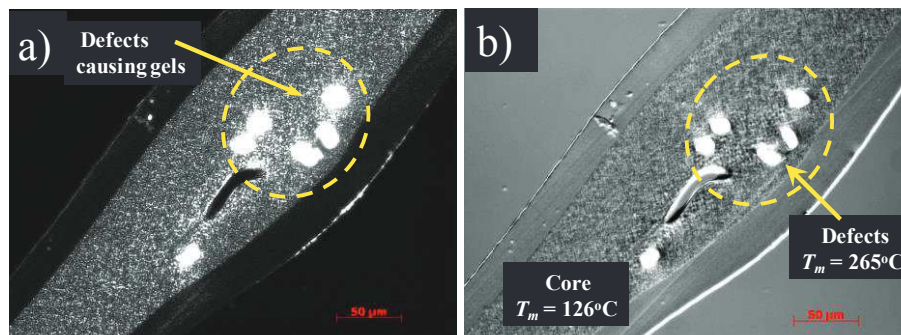


Figure 1.10 Photographs of gels in the core layer of a three-layer film: (a) transmitted polarized light, and (b) hot-stage microscopy was used to determine the melting temperatures of the core resin and defects [12]. Photographs were provided by E. Garcia-Meitin of The Dow Chemical Company

Another common contaminant that produces gels is fiber, as shown in Fig. 1.11. In many cases, these contaminants are cotton fibers from clothing and gloves or cellulosic fibers from packaging materials. Fourier transform infrared (FTIR) spectroscopy is one of the best techniques for determining the chemical functionality of organic-based defects in PE films.



Figure 1.11 Transmitted bright-field image of a PE film containing a fibrous gel [12]

Once the contaminant is identified, the troubleshooter must determine how the material entered the feedstock stream. Process controls must be identified and implemented to mitigate the contaminant source.

■ 1.4 Troubleshooting Extrusion Processes

Eventually every single-screw extrusion process will experience periods when the machine is operating at a performance level that is less than designed. During these periods, the cost of manufacturing will increase due to the production of off-specification products, loss of production rates, high levels of recycle, higher labor costs, and lower daily production of prime product. In extreme cases, the problem can be so severe that the line must be shut down. Obviously the plant needs to restore the operation of the line to the original performance level as soon as possible to maximize profitability. Many things can cause an extruder to malfunction, including mechanical and electrical failures, installation of new equipment, process changes, and resin changes. A complete process for troubleshooting an extrusion process can be obtained in reference 1. Several of the most common problems associated with film production processes are presented in this section.

1.4.1 Improper Shutdown of Processing Equipment

Shutting down an extrusion line occurs for many reasons, including planned shutdowns for maintenance, shift changes, changing filtering screens, product changes, and many unplanned events. A shutdown period is defined here as a period when the screw is not rotating and thus resin is not discharging from the line. If the shutdown period is relatively short such that very little resin degradation can occur, then the extruder barrel temperatures can be maintained at the operating set point temperatures. If the length of the shutdown is long relative to the time required to create a significant level of degradation products, then the extruder should be either purged with a more thermally stable resin or the barrel set point temperatures should be decreased to considerably lower temperatures. Purging the resin with an inert gas to exclude oxygen is also effective at mitigating gels [1]. An extruder that is maintained at process conditions long enough to create degradation products can be very difficult to bring back online running prime product. In this case, the surfaces of the screw and all metal surfaces in contact with molten resin may become coated with degradation products, as shown in Fig. 1.12. The time to purge them out can be extremely long and very expensive. For example, LLDPE resins can form crosslinked gels and black specks after 30 minutes of being off-line at process temperatures. If the shutdown is under 30 minutes, the barrel can be held at the process temperature. If the shutdown is longer but the line will be brought back online soon, the screw could be rotated at a low speed of 5 rpm to keep resin flowing, mitigating the formation of degradation products. For longer shutdown periods, the extruder should be purged using an LDPE resin and then cooled to ambient temperature.



Figure 1.12 Photograph of a screw that had numerous shutdowns where the extruder was maintained at operating temperatures for an extended time. The extruder was purged prior to removing the screw, yet dark degraded resin covers most areas of the screw [1]

Antioxidant chemicals are typically added at levels that stabilize the resin during normal melt processing. They are not meant to protect the resin from degradation during an extended shutdown period. Antioxidants are slowly consumed during the extrusion process, and thus they can be fully consumed during an extended shut-

down period. When they are fully consumed, the resin system is not protected from degradation, and thus degradation reactions will occur at accelerated rates.

1.4.2 Gel Showers in a Cast Film Process

Crosslinked gels can form in stagnant regions of screw channels, transfer lines, and dies. The time required for these gels to form range from about 30 minutes for linear low density polyethylene (LLDPE) resin up to 12 days for low density polyethylene (LDPE) resin. Stagnant regions can occur at entries and exits of mixers [1] and barrier sections, and they can occur when the metering channel of smooth-bore extruders is not controlling the rate. In these cases, a section upstream of the metering section is rate limiting, causing portions of the metering section to operate partially filled [18, 19]. When these channels operate partially filled, the main flow is on the pushing side of the channel while the trailing side operates void at first. After a period of time, clean resin gets into the void regions and rotates with the screw for long durations. Eventually the resin will degrade, forming crosslinked gels. Slight process upsets can dislodge this material, allowing the material to flow downstream, creating a gel shower in the film.

A film plant was extruding an LDPE resin into a specialty product using a cast film process [18, 19]. Due to high demand, a new 88.9 mm diameter, 33 L/D extruder was installed in the plant. Soon after start-up the product was acceptable and high quality. After 12 days, the line began to experience intermittent discharges of crosslinked material (gel showers) and carbon specks. Photographs of these gels are shown in Fig. 1.13. In some cases, the gel showers were observed two to three times per day and would last from 1 to 5 minutes. The gels were clearly crosslinked and were brown in color. The extrudate temperature was higher than expected for the process. The intermittent gels resulted in production downtime due to purging and in numerous customer complaints. A high and costly level of quality control was required to remove the gel-contaminated product from the prime product. Due to the high amount of downtime and the high levels of quality control needed, the operation of the new line was considerably more expensive than planned.

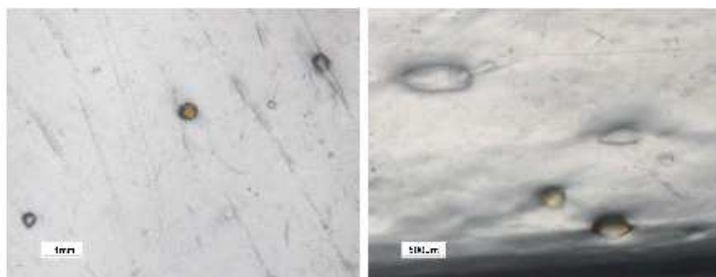


Figure 1.13 Photographs of crosslinked gels in an LDPE film

It was hypothesized that the extruder was operating partially full due to the low specific rate during operation. To determine if partially filled channels were the root cause of the reduced rate, high discharge temperature, and degraded material, screw rotation was stopped and the screw was removed while hot from the extruder. Examination of the polymer on the screw indicated that in the meter section about half of the channel width on the trailing sides of the flights for all but the last diameter were filled with a dark-colored, partially carbonized LDPE resin, indicating that these regions were stagnant. The reduced flow rate caused these regions to be partially filled, creating void regions on the trailing side of the channel. Some of the resin adhered to the trailing side of the screw in the void regions and stayed there for extended time periods, as shown in Figure 1.14. The resin adhering in the void regions eventually degraded into the dark-colored, crosslinked material. Small process variations dislodged some of this material and caused the intermittent gel showers that contaminated the product. Moreover, compacted solids were found wedged in the channel at the entrance to the barrier section. The wedged material was caused by the relatively large width of the entering solid bed being forced into the continually decreasing width of the solids channel of the barrier section.

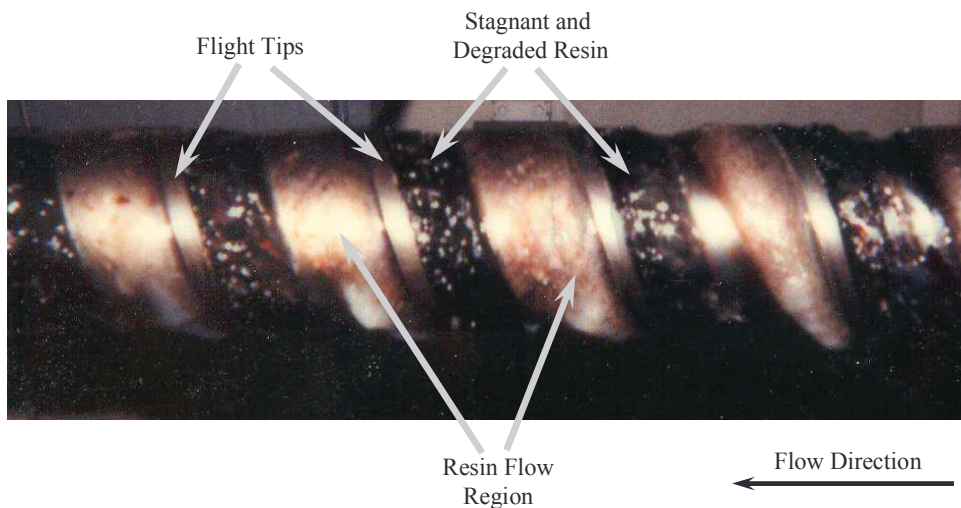


Figure 1.14 Photograph of a removed screw showing the resin flow and degraded resin due to stagnant regions [1]

The technical solution to eliminate this problem was a simple modification to the entry of the barrier melting section. For this modification [18], some of the metal in the melt conveying channel was removed along with a portion of the barrier flight, allowing some solid material to enter the melt channel and reducing the restriction at the entry. By reducing the restriction, the rate-limiting step of the process changed from the entry region of the barrier section to the metering section. After the modification was made, the gels were eliminated from the process.

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