

DuPont™ Surlyn® Extrusion Guide

Technical Information

DuPont™ Surlyn®



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I. Introduction

Extrusions of DuPont™ Surlyn® have earned industry acceptance, in many performance-demanding applications, by offering these primary performance benefits:

- Outstanding toughness, especially at low temperature
- Clarity including weather resistant formulations
- Lightweight
- Favorable value-in-use and economics

Surlyn® resins are relatively simple to extrude. They process very similar to polyethylene and Elvax® ethylene vinyl acetate resins. Melt strength is outstanding and thermal stability is good. Processing characteristics which tend to be unique to Surlyn® are covered in other sections of this manual.

II. Safety

Safety hazards, common to all thermoplastic extrusion operations, apply to Surlyn® and require standard, industry accepted, safety practice. Primary hazards relate to: high temperatures; high pressures; potential fume generation (especially over 250°C or 482°F) and spills. Molten Surlyn® can easily stick to skin and this should be recognized as a specific hazard.

DuPont places a very high priority on safety. We believe that the personal safety of each employee is of great importance. We operate on the philosophy that, with proper attention, all personal injuries can and should be prevented. The following protective measures are worthy of review and consideration:

- Use gloves and other protective clothing, as necessary, when handling hot polymer or machine parts
- Wear approved safety glasses • Use adequate ventilation
- Use accepted engineering designs and process controls
- Promptly cleanup any resin pellet spills (especially on the floor)

Surlyn® Material Safety Data Sheets are available at **www.dupont.com** or from your local DuPont representative.

III. Surlyn® Resin Selection

Surlyn® resins are available in a variety of grades. Selection of a grade for a particular application is typically based on consideration of the following properties:

- Melt Index (generally 5 or less for extrusion)
- Stiffness: available from flexural modulus of 94 to 376 MPa; 14,000 to 55,000 psi – in natural, unmodified grades

- Clarity: transparency, gloss and haze
- Low temperature toughness
- Impact strength
- Adhesion to other materials (zinc cation preferred- usually 9950)
- Abrasion resistance
- Cation type (zinc preferred for UV stabilized formulations)

IV. Surlyn® Chemistry

Surlyn® resins are based on copolymers of ethylene and methacrylic acid (E/MAA) which have been partially reacted with metallic salts to form ionic crosslinks between the acid groups within a chain, or between neighboring chains. (Ionically crosslinked structures). Surlyn® is DuPont's registered trademark for its brand of Surlyn® resins.

Many unique and attractive properties of Surlyn® resins result specifically from the ionically crosslinked and hydrogen bonded structure, and include: low crystallinity; low temperature impact and flex toughness; abrasion and solvent resistance; transparency with low haze at thickness up to 6.4 mm (0.25 in.) - high melt strength; and adhesion to epoxy finishes, metals and many other materials.

V. Extrusion Processes

Industrial extrusion processes for Surlyn® resins are all similar with respect to selection, handling and formulating of the resin. In addition, the extruder design and operation for Surlyn® is basically common to the variety of extruded product configurations. Process differences begin with the extrusion die and continue through the cooling, haul-off, and product packing stages of the operation.

Extruded industrial products of Surlyn® resins include:

- Profiles and shapes
- Tubing and pipe
- Sheet (generally defined as 0.25 mm (10 mils) thickness and greater)
- Coated wire and cable
- Blow molded articles
- Rod
- Coextrusions of each item above

This manual will cover, primarily, resin preparation and extruder considerations. Experience has demonstrated that these subjects are of highest priority to our customers and prospects. Information is included with respect to cooling, haul-off, and product packing. However, it is beyond the scope of this manual to cover all details for

these process steps for all of the potential extruded products.

An extrusion operation is conveniently divided into six steps. Identification and recognition of these individual steps is extremely beneficial. Clear understanding of these steps will assist in efficient problem identification and solution. The basic steps in the extrusion process are:

6 BASIC EXTRUSION STEPS:

1. Preparation of Surlyn® resins
2. Extruder
3. Adaptor and die
4. Forming and cooling
5. Haul-off
6. Product packing (typically rolls, coils or cut to length)

VI. Pre-Extrusion Checklist

During preparation for trials or production with DuPont™ Surlyn® resins, several questions and concerns are typically of interest. These items are important for review during planning stages prior to extrusion. They will assure that the extrusion "set up" will provide the best opportunity for trouble-free operation.

Following is an important, pre-extrusion checklist:

IMPORTANT, PRE-EXTRUSION CHECKLIST:

1. Surlyn® resin from sealed, undamaged containers
2. Clean extruder preferred; extruder which still contains a polyolefin inventory (PE, E/MAA, EEA, PP, etc.), acceptable, if necessary.
3. Polyolefin-type metering screw or equivalent in performance (confirm and document screw dimensions)
4. Metering screw compression ratio: 4/1 preferred; 3.5/1 acceptable (ratio of feed to metering channel depths).
5. Internal screw cooling - first 4-5 turns of feed section
6. Hopper feed throat jacket-water cooled
7. Rear barrel heat zone set low: 150°C (302°F) or less, unless drive power requirement is excessive.
8. Temperature profile for 200°C (392°F) melt temperature for 0.7-2.5 melt index grades; or 175°C (347°F) melt temperature for 5-14 melt flow index grades, unless experience has confirmed other temperatures acceptable.
9. Cooling tools, forming tools, chill rolls and water quench, all as cold as possible

VII. Preparation of Surlyn® Resins

RAW MATERIALS

Natural Surlyn® Resins

DuPont™ Surlyn® resins are supplied in moistureproof containers – either 22.7 kg (50 lb.) bags or 500 kg (1,100 lb.) boxes. Many products are extruded from natural resin Doc. Ref. SUR020429_1, v.2, December 2003
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as supplied. However, care should be taken to minimize exposure of resin to the atmosphere, since Surlyn® resins are hygroscopic (will absorb moisture). A good rule of thumb – conservative, but protective – for nominal 200°C (392°F) melt temperature extrusion is: open only sufficient inventory for 2 hours operation; during very hot and humid seasons, one hour of open inventory is preferred.

Any material in opened or damaged containers should be assumed to be high in moisture and may require drying.

Unused resin should be resealed in its original package or another moistureproof container. Improperly opened, a bag or box liner can be heat sealed to keep the resin dry during storage..

Pre-compounded Surlyn® Resins

Many finished products will require, or will benefit by, addition of ingredients to Surlyn® to provide, for example, weathering (UV) resistance, colors, foamed products, flame retardant products, glass reinforcement, or combinations of these examples.

The use of fully compounded product may be desirable depending on the additive system and the requirement for optimum mixing. For example, UV resistant formulations require a high degree of mixing (dispersion) to assure optimum end-use performance. Pre-compounded formulations can be prepared by commercial compounders or in-house, if the required compounding equipment is available.

Formulations via Concentrates

The types of formulations referred to above under "Pre-compounded Surlyn® Resins" can also be prepared by use of a concentrate by the extruder. Concentrates are usually prepared by commercial compounders to assure optimum dispersion.

Typically, a concentrate will contain ten times the additive level desired in the "extended" or finished product. In this instance, nine parts of natural resin would be thoroughly mixed with one part of concentrate to produce ten parts of a resin pellet blend, to be supplied to the extrusion machine. This is commonly referred to as a 10/ 1 letdown ratio. Depending upon the formulation, letdown ratios range from 2/1 to 30/ 1.

Ideally, the base or carrier resin used to prepare the concentrate will be a natural Surlyn® or similar ethylene copolymer, but of an appropriate melt index to compensate for changes in melt viscosity, depending on the additives. For example, if high loadings of filler are required, then a higher melt index base would be recommended; if the additive is a liquid (plasticizer), then a lower melt index base would be recommended. Following these recommendations will assure that the final melt extension of the concentrate and natural resin will take place at nearly equal melt viscosities, thus achieving optimum melt shear mixing.

The concentrate carrier resin must be melt-compatible with Surlyn®. Commercial compounders are generally

familiar with these requirements. It should be noted that polyethylene or polypropylene homopolymer-based concentrates are not melt-compatible with DuPont™

The use of mixing type screws may be desirable when extending concentrates in a production extruder. This will depend upon the formulation and end-use performance requirements.

Dry Blending

Surlyn® resins are recognized as outstanding binders. They readily accept most additives, frequently at higher than anticipated loading levels for polyolefin resins. Therefore, it is not unusual for formulations to be prepared by dry blending at the extruder. This may offer economic incentive; however, the potential for cross-product contamination is great, and the dry blending technique is judged unacceptable by many extruders.

Dry blend systems may be adequate for many applications. However, they will not have the dispersion quality of precompounded or concentrate systems. Consideration should be given to mixing type screws for production with dry blend systems. Dusting of dry blended additives can be minimized by addition of 0.1 weight % of mineral oil. Thorough dry mixing is very important.

Regrind

Surlyn® resins have good temperature stability, especially when extruded in the range of 150-230°C (302-446°F). Consequently, use of good quality regrind is acceptable. Regrind can be used at levels of up to 15-20%, provided that the regrind is:

- Clean and free of any contaminants
- Dry
- Not degraded by excessive time/temperature exposure (for natural resins, no signs of yellowness; except for a natural resin containing, for example, an antioxidant with a yellow tint).

Regrind can be cut with standard granulators, provided that the knives are sharp. Granulator settings for polyethylene with 8 mm (5/16 in.) diameter screens are acceptable.

FORMULATIONS

Ultraviolet/Weather/Sunlight Resistant

A beneficial feature of Surlyn® resins is that they can be stabilized to provide ultraviolet resistance (sunlight or weathering resistance), with little or no sacrifice of the inherent clarity of the resin.

The preferred UV stabilizer system includes: a zinc cation Surlyn®; a UV absorber; an energy quencher; an antioxidant; and optionally, a transparent blue or violet pigment. The preferred UV absorber imparts a slight yellow cast to the product. This can be masked by addition of a small amount of transparent blue or violet pigment. In

any case, the blue, violet or yellow tints cannot be detected at nominal part thickness of 0.5 mm (20 mils), unless compared to a control. As thickness increases, the tints will become progressively more apparent.

The predicted service life of the preferred clear UV stabilizer system is in the range of 3-5 years depending upon the severity of exposure. This is a remarkable improvement as compared to unstabilized polyolefins or to some other stabilized polymers.

The preferred UV absorber and energy quencher both tend to volatilize at temperatures typical for compounding or extrusion. Therefore, these ingredients are added at quantities which will assure adequate protection in the finished product after processing volatilization losses (typically 33% loss or 50% excess in original material balance formula). Losses due to volatilization can be minimized by compounding and extruding at the lowest feasible temperatures.

Experience has shown that the clear UV formulations, either pre-compounded or concentrates, are more prone to extruder screw bridging than natural Surlyn®s.

The tendency to bridge is minimized, if not eliminated, by specifying pellet size of the stabilized product to be nominally 3.2 mm (1/8 in.) diameter by 3.2 mm (1/8 in.) long (or equivalent). Smaller pellets are likely to cause bridging in UV stabilized formulations.

Optimum UV and weathering resistance is achieved by incorporation of carbon black and antioxidant in Surlyn®. The carbon grade must be selected for UV protection and not simply as a pigment or colorant. In addition, the degree of carbon dispersion relates directly to service life. The carbon must be well dispersed. Surlyn® products stabilized with carbon and antioxidant have been in continuous weathering service for over 10 years.

The preferred quantity of carbon depends on the Surlyn® cation type and the degree of dispersion. Generally, zinc types require at least 2.6 weight %; sodium types, 5.0%. Additional formulations and performance data are available from DuPont upon request.

Colors –Opaque and Transparent

Surlyn® resins readily accept colorants to provide parts with high quality aesthetic appeal. Either transparent or opaque pigments can be used. The inherent clarity of Surlyn® provides the opportunity to select a wide array of transparent colors and tints in addition to the more traditional opaque colors.

Pigments are generally preferred, rather than dyes. Pigments, typically, have better thermal stability at extrusion temperatures and better lightfastness. Improved dye systems are available and may be adequate for some colors.

Foam

Foamed products offer economic incentives and opportunity to design selective properties, such as

resiliency or weight reduction, into finished products. It should be noted that many strength properties are a direct function of density. However, because of the remarkable toughness of DuPont™ Surlyn®, adequate toughness remains after selected density reduction by foaming.

Surlyn® resins can be foamed by either gas injection - e.g., Freon®, fluorocarbons – or by chemical blowing agents (CBA).

The gas injection method allows achievement of lower densities, but also requires equipment specifically designed for high pressure injection. If this equipment is not available, CBA systems will frequently accommodate most standard extruders.

A typical CBA formulation would include: a chemical blowing agent with an activation temperature in the range of 150-200°C (302-392°F); an activator which will reduce the temperature at which the CBA will release gas; a nucleator which will provide tiny sites for gas release, assuring uniform cell structure; and optionally, mineral oil, if the formulation is prepared by dry blending.

The surface of foamed extruded products may be disrupted as a result of the blowing mechanism. If the surface is undesirable, a coextruded solid skin over the foam can be considered.

Surlyn® resin grades with high melt viscosity (low melt flow rate) should be selected for foam formulations. In addition, they should be extruded at relatively low temperatures (125-150°C; 257-302°F), to provide high melt viscosity. Experience has demonstrated that good candidates are Surlyn® 9720 or 9721. High melt viscosity is mandatory to provide resistance to the evolved gas and promote small, uniform cell structure.

All products cannot be produced in foam. Limitations relate to size, configuration and desired density. For example, low density, flat sheet cannot be produced using a flat die. Exit the die, expansion occurs in the machine and in transverse directions and in thickness. The 3-dimensional expansion will result in a distorted, "wavy" sheet at low densities. Flat sheeting at low density can be made using a circular die. The "tube" is slit, opened, and wound flat.

Foamed products, by their nature, are insulators, due to the trapped gas in their structure. For this reason they are more difficult to cool than solid extrusions. Inadequate cooling will result in distortion and cell collapse.

Flame Retardant

The flammability properties of Surlyn® can be improved by incorporation of flame retardant additives. Relatively high loadings are required. Therefore, precompounded or concentrate formulations are preferred. The high loadings also reduce the melt flow index and may require selection of higher melt flow index resins to compensate and maintain adequate flow for extrusion. Typical flame retardant formulations include a brominated organic compound and antimony oxide.

Glass Reinforced

Surlyn® can be modified with glass fiber. This will provide a remarkable increase in flexural modulus (stiffness) and will generally not interfere with other properties. Glass fiber concentrates are available commercially.

Compounders are familiar with fiber sizing (surface treatment) requirements to assure acceptable adhesion within Surlyn® resins.

MOISTURE AND DRYING

Sources of Moisture

Surlyn® resins are hygroscopic. They will absorb moisture when exposed to the atmosphere. The rate of moisture absorption is dependent upon many factors including: Surlyn® grade; cation type; ambient temperature; relative humidity; and air flow around the pellets.

Another source of moisture is atmospheric condensation on cold resin. Condensation of moisture on the resin in cold weather can be avoided by keeping about a one-shift supply of resin in the operating area. This allows the resin to warm above the condensation temperature (i.e., dew point), before the container is opened.

Moisture Content vs. Maximum Extrusion Temperature

The amount of moisture, which can be tolerated in Surlyn®, depends upon the melt temperature for the extrusion. For example, at a melt temperature of 170°C (338°F), any moisture content less than 2600 parts per million (ppm), or 0.26 %, will permit extrusion without moisture defects. At a melt temperature of 250°C (482°F), any moisture content less than 1250 ppm (0.126%) is acceptable. The relationship of moisture content vs. maximum melt temperature – without moisture defects – is shown graphically in Figure 1, for melt temperature from 150°C (302°F) to 250°C (482°F).

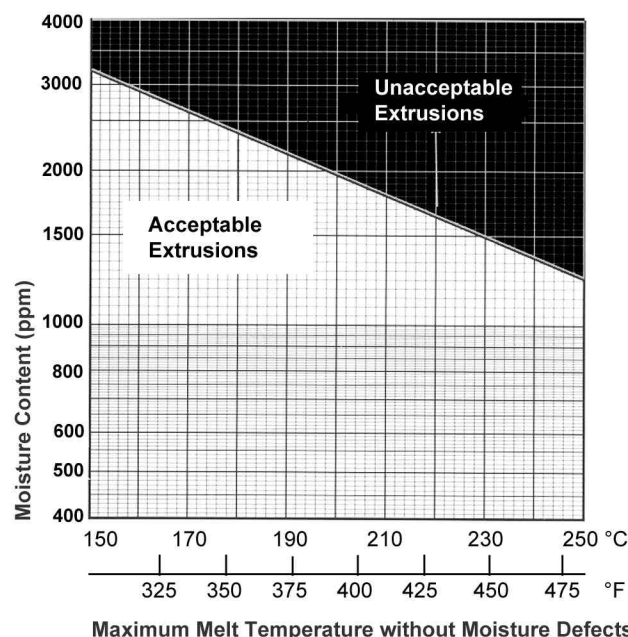
Surlyn® products are typically packaged with moisture content of less than 800 ppm. Therefore, Surlyn® from freshly opened, undamaged packages should not result in any moisture related problems. At typical melt temperature of 200°C (392°F), there is a significant tolerance for moisture pickup, as shown in Figure 1.

Defects will appear on the surface of the extrudate when moisture content is marginal. The defects will be smeared, elongated surface bubbles. At very high moisture content, defects will appear as bubbles in the extrudate cross-section. However, bubbles may also be a result of a starve feeding condition caused, for example, by extruder screw bridging.

Drying of Surlyn®

Normally, DuPont™ Surlyn® resins do not require drying. However, drying may be required as a result of the following conditions: inadvertent exposure of resin to the atmosphere for several hours; containers ruptured as a result of plant handling; and use of reground.

Figure 1 — Moisture Content vs Maximum Extrusion Temperature for Surlyn® Resins



The preferred method of drying is use of a dehumidified or vacuum tray-type oven. The oven temperature must be controlled accurately to avoid any cyclic increases in temperature. Most ionomers should be dried at the maximum temperature of 65°C (149°F) to avoid potential "caking" of pellets. Softer products like DuPont™ Surlyn® 9020 and 8020 should be dried at a lower temperature of 60°C (140°F). Generally, eight hours of drying time is adequate. Resin depth in the trays should be 50 mm (2 in.) or less.

The relationship of moisture contents vs. dehumidified drying times is shown in Figure 2. The family of plots represent starting moisture contents of 5000, 4000, 3000, and 2000 ppm and respective drying times for each.

Figure 2 — Moisture Content vs Dehumidified Drying Time, Surlyn®

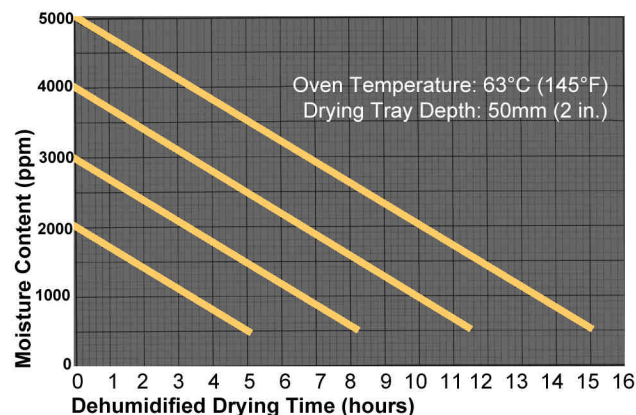
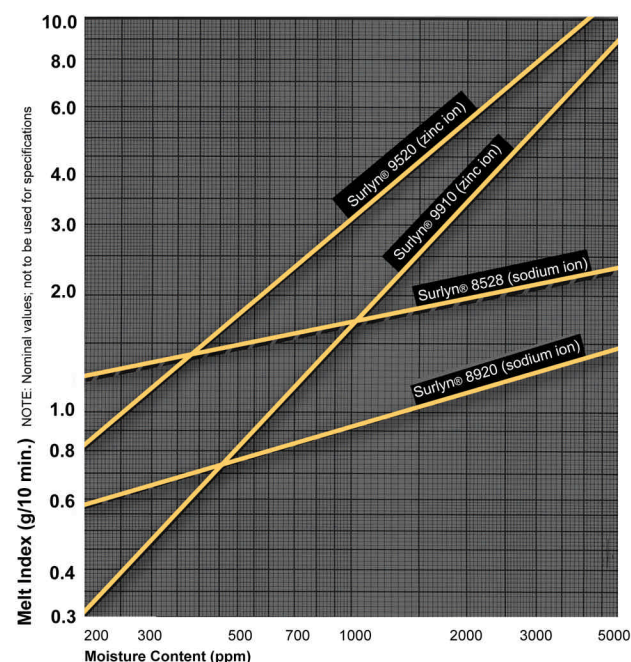


Figure 3 — Moisture Content vs Melt Index, Surlyn®



Drying ovens without dehumidifier or vacuum features should never be used. Hot air ovens will typically add, rather than remove, moisture.

Hopper dryers are not recommended. At the required maximum drying temperature of 65°C (149°F), the weight of the resin in the hopper will cause agglomeration of pellets, resulting in bridging in the hopper throat or feed zone. If drying temperature is reduced, then required drying time will be substantially increased.

Moisture Content vs. Melt Flow Index

Moisture has a plasticizing effect on Surlyn®. The melt flow index or viscosity is a function of the Surlyn® moisture content. Increased moisture will cause an increase in melt flow index (a decrease in viscosity). The relationship of melt flow index and moisture content is shown in Figure 3, for several grades of Surlyn®. These data show that zinc cation resins are much more sensitive than sodium cation resins to change in melt flow index with increasing moisture content.

VIII. Extrusion Equipment

HANDLING OF SURLYN® RESINS

Typical resin handling equipment is used for Surlyn® resins, including pneumatic hopper loaders, provided that resin exposure to the atmosphere is minimized, as discussed above.

Machine hoppers should be covered to prevent contamination. In addition, a cover will minimize air exposure and air circulation around the pellets, thus preventing unnecessary moisture absorption.

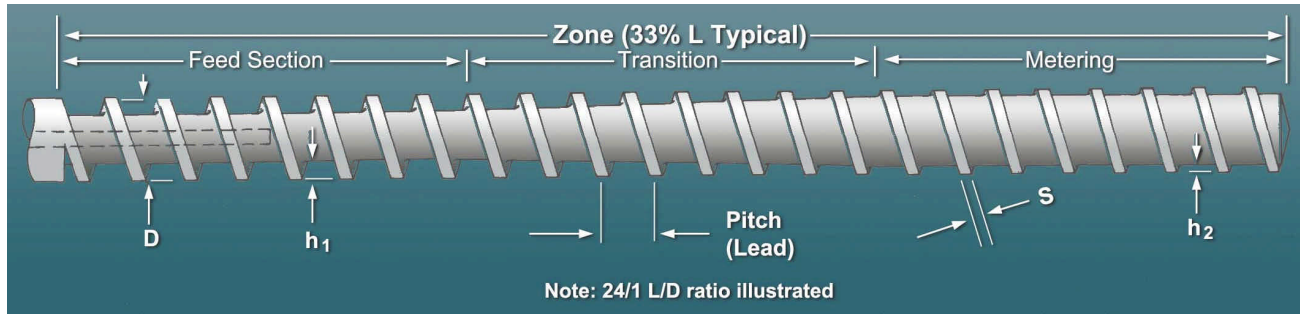
Occasionally, bin storage or hopper inventory requirements will result in excessive moisture absorption. The use of a dry nitrogen purge will eliminate exposure to moisture under these conditions. If nitrogen is used for this purpose, it must be specified to be dry. A purge stream of dry nitrogen is introduced at the base of the covered bin or hopper. Nitrogen, being slightly lighter than air, will rise and displace the air containing moisture.

EXTRUDER

Typical Screw Design

Metering type screws, common to polyolefins, generally perform satisfactorily with DuPont™ Surlyn®. Typical metering screw design and dimensions are shown in Figure 4.

Figure 4 — Typical Metering Screw Design Dimensions



Screw Diameter, mm (in.) (ref. figure, "D")	Feed Depth, mm (in.) (ref. figure, "h1")	Metering Depth, mm (in.) (ref. figure, "h2")	Flight Width, mm (in.) (ref. Figure 2, "S")	Radial Clearance
38 (1.5)	9.7 (0.380)	2.4 (0.095)	3.8 (0.15)	0.1 (0.004)
50 (2.0)	11.0 (0.432)	2.7 (0.108)	5.0 (0.20)	0.1 (0.004)
64 (2.5)	11.2 (0.440)	2.8 (0.110)	6.4 (0.25)	0.1 (0.004)
89 (3.5)	11.9 (0.468)	3.0 (0.117)	8.9 (0.35)	0.1 (0.005)
114 (4.5)	13.2 (0.520)	3.3 (0.130)	11.4 (0.45)	0.1 (0.005)
152 (6.0)	17.0 (0.670)	4.3 (0.168)	15.2 (0.60)	0.2 (0.006)

The minimum preferred length to diameter (L/D) ratio is 20/1. Ratios less than 20/1 will likely result in variations in output, delivery pressure, and melt temperature. L/D ratios as high as 32/1 are used commercially. Ratios of 24/1 and 28/1 are, perhaps, most common.

A screw compression ratio of 4/1 is preferred. This is the ratio of feed channel depth to metering channel depth. Compression ratios as low as 3.5/1 are satisfactory, especially in the larger diameter screws. If the compression ratio is too low, extruder screw bridging is likely.

A minimum feed channel depth of 9.5 mm (0.375 in.) is preferred. Smaller feed channel depths may likely interfere with uniform feeding and conveying of nominal 3.2 mm (1/8 in.) pellets and result in bridging and non-uniform extruder output.

The combination – of preferred compression ratio, minimum feed channel depth, and requirement for internal screw cooling – places significant limitations on the ability to design optimum performance screws at diameters less than 50 mm (2.0 in.). The design balance is very critical, in order to maintain adequate strength in the feed zone, due to minimum steel thickness remaining after cutting the feed channel and cooling bore.

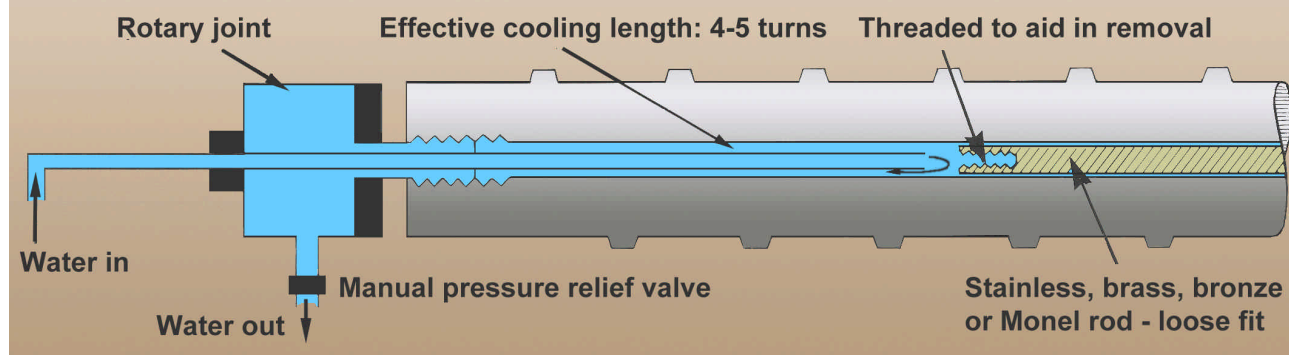
The nominal distribution of screw zone lengths (feed, transition and metering), is dependent upon the L/D ratio. For example:

L/D Ratio	Typical Zone Length, %		
	Feed	Transition	Metering
20/1	25	50	25
24/1 – 32/1	33	33	33

The feed zone length should never be less than 25% to minimize the tendency for bridging. Gradual transition in the range of 33-50% will improve uniformity of output, as compared to short or "sudden" transitions.

Given combinations of metering depth and length can result in an adiabatic melt temperature. This is a condition where the resin is heated internally by friction (shear), and no external heat from the barrel heaters is required. This adiabatic condition can result from a shallow channel depth over a long length. Reduction of extruder barrel temperatures will not reduce melt temperature when this condition exists. Lower melt temperatures can be achieved by reducing output or modifying the screw for a deeper metering depth and/or shorter metering length.

A variety of screw designs and configurations are available to meet specific requirements such as: (a) very high output; (b) precise control of melt temperature and

Figure 5 — Flow Restrictor Plug for Screw Cooling

pressure; and (c) mixing of ingredients. These high performance designs should be considered for industrial extrusion operations which may be limited by the more general purpose metering type screw. (For example, heavy gauge sheeting at high output; or optimum mixing).

Screws designed for PVC are prevalent and, typically, have a constant pitch and uniformly increasing root diameter from the rear to the front. This type screw may cause variable output (surging) with Surlyn® resins, especially as output is increased.

Vented (vacuum) type, 2-stage, screws can be used for DuPont™ Elvax® resins. However, their performance is critically dependent upon given resin viscosity and throughput rate. This type design should be considered only for operations where the same basic resin and extruder rate are both maintained for long production runs.

Screw Cooling

Internal cooling of the extruder screw is preferred with DuPont™ Surlyn® resins. (See Figure 5.) The internal cooling should cover the first 4-5 turns of the feed zone of the screw. Cooling this zone will greatly reduce the tendency for bridging and unacceptable degrees of starve feeding.

It is ideal if the screw is bored only 4-5 turns for coolant circulation. If a given screw has been bored beyond the feed zone, then it will be necessary to restrict the effective bored length by insertion of a loose fitting plug as shown in Figure 5. Internal cooling of the metering zone should be avoided. Such cooling will alter the performance characteristics of a properly designed screw.

CAUTION: Screw cooling components (rotary joints, seals, connections, etc.), must be designed to withstand potential generation of steam pressure. A manual pressure relief valve should be activated prior to mechanical disassembly.

Instrumentation

Instruments to monitor melt temperature and pressure are extremely important. Without these instruments only "guesses" can be made regarding the extruder performance. In addition, duplication of conditions from run to run may be impossible.

Melt temperature can conveniently be measured in the vicinity of the breakerplate (either upstream or downstream). An exposed junction (for rapid response) melt thermocouple with 6.4 or 12.7 mm (1/4 or 1/2 in.) probe is preferred to avoid close proximity to the flow channel wall and to gain immersion into the melt stream. Probe lengths over 12.7 mm (1/2 in.) will frequently "bend over" against the flow channel wall due to the pressure of the polymer flow.

Melt pressure is best measured between the end of the screw and the breakerplate. (Pressure readings may show "blips" at this location due to the passing of the screw flight with each revolution.) Pressure can also be measured downstream of the breakerplate although this location will not effectively allow monitoring of screenpack condition. Either a pressure transducer or Bourdon type gauge can be used with the tip flush with the inside of the flow channel. The Bourdon type gauge requires a silicone "grease leg" to transmit pressure to the gauge and is subject to leaving a slight contamination on the surface of the extrusion. Therefore, direct reading transducers are preferred.

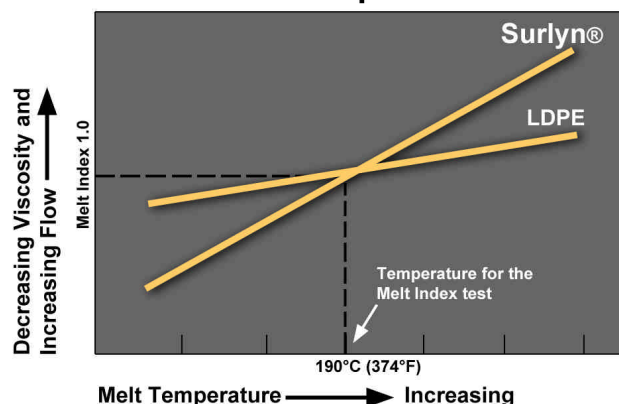
Standard instrument wells are used for both melt thermocouples and pressure gauges. Details are available on request.

General

Extrusion of Surlyn® requires a water cooled hopper feed throat. Virtually all modern extruders include this feature, necessary to prevent pellet agglomeration from heat in the feed throat. However, frequently the cooling channel is fouled and dirty, and requires cleaning to provide efficient cooling.

Extruder drive horsepower requirement for Surlyn® resins is a function of melt temperature. As compared to low density polyethylene (LDPE) of equivalent melt index, Surlyn® may require less horsepower at melt temperatures above 190°C (374°F), and will require more horsepower at lower temperatures. The horsepower increase could approach 30-50% depending upon melt temperature. These differences relate to the slope of the viscosity vs. melt temperature curves for Surlyn® and LDPE and are shown graphically in Figure 6.

**Figure 6 —
Melt Flow vs Melt Temperature**



Another consideration regarding power requirement relates to the progressive stages of resin temperature, as it is first melted, and then increased in temperature in the feed and transition zones of the screw. During these stages resin temperature is low and viscosity is high, as shown in Figure 6. The high viscosity, or resistance to flow of DuPont™ Surlyn® resins during the melting/heating process, requires more horsepower than is needed for LDPE.

The power requirement for extrusion of Surlyn® has not been a demonstrated problem, probably because most commercial extruders have adequate drive power.

The extruder barrel should have at least four independently controlled heat zones (each 25% of the length). This will assure adequate control capability for optimum production. Many machines are equipped for barrel cooling. This feature may be of benefit to help prevent bridging in the first heat zone.

Standard extrusion operation should include a breakerplate and screen pack. The breakerplate will interrupt the spiral flow from the screw and the screen pack can prevent inadvertent debris from damaging die parts. The screen pack is not intended to develop back pressure; this is a function of a properly designed screw. A typical screen pack is 2-80 mesh and 1-40 mesh screens (40 mounted against the breakerplate). Streamlining of the breakerplate assembly is covered in the following section, "Adaptor and Die."

Since Surlyn® resins with absorbed moisture can create an acidic vapor at processing temperatures, extruder, adaptor and die components which come in contact with molten resin should be constructed from corrosion resistant alloys or surfaced with durable chrome plating. Mild steel components may be attacked under certain conditions of high temperature and time when in contact with acidic water vapor.

ADAPTOR AND DIE

Streamlining

Streamlining of the polymer flow paths in the adaptor and die assembly is extremely important. Resin flow in these components is laminar and not "plug flow" This means that flow velocity is highest in the center of the flow channel and, at least in theory, is at zero velocity on the walls of the flow channel. Therefore, resin near the walls has an appreciably longer holdup time than resin toward the center of the channel.

While Surlyn® resins are reasonably heat stable, they are, like any polymer, subject to degradation when exposed to high temperatures for a long time—commonly referred to as time/temperature exposure. The temperature is dictated by the extrusion process. The time factor, by definition, is increased for resin near the channel wall in systems with good streamlining design. Poor streamlining or, in effect, "unnatural flow paths" can result in areas of total stagnation and no flow. Resin will degrade and slough off into the extruded product, causing defects. Examples of "right" and "wrong" designs are illustrated in Figure 7.

Heating

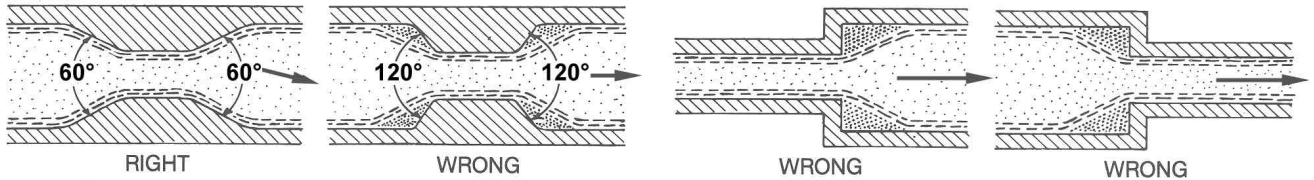
The adaptor and die assembly should be totally heated to maintain a uniform melt temperature. Generally, this assembly is not intended to either remove or add heat to the melt; but rather to maintain the melt temperature, pre-established by the screw. All possible areas of the adaptor and die should be covered by heater bands or equipped with internal heaters. An exception would be over the area of a melt flow seal, which could possibly leak. In this case a heater band could conceal the leak until significant cleanup would be required.

Some designs do not permit effective placement of heaters—for example, massive head clamps or swing gates. In these cases, the non-heated areas should be insulated. This will allow adjacent heated zones to more effectively heat these areas and also will minimize heat loss from the non-heated area.

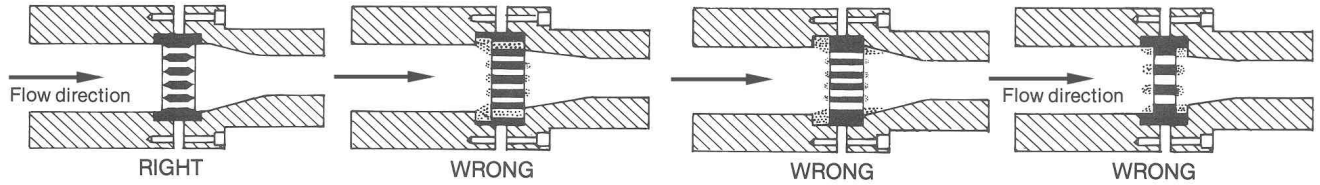
Each adaptor and die heat zone should have about the same geometry throughout the zone. An undesirable situation would be combining a massive die zone with a small diameter neck piece (common to sheeting dies), and controlling both by a thermocouple in the massive zone. The neck piece can be subject to severe overheating. Conversely, if the controlling thermocouple is in the neck piece, the massive die zone would not come up to heat.

Figure 7 — Adaptor and Die Streamlining

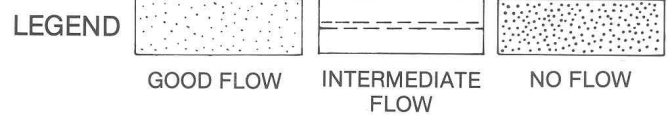
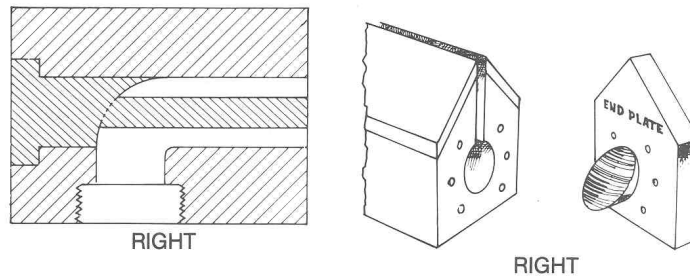
1. CHANGE IN FLOW CHANNEL DIAMETER



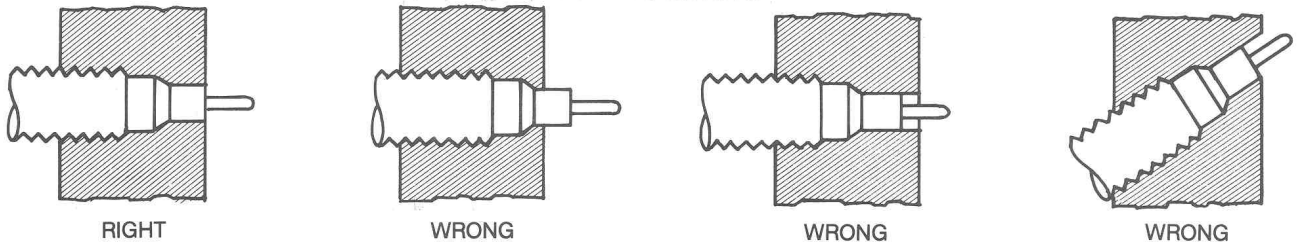
2. BREAKER PLATE DESIGN AND MOUNTING



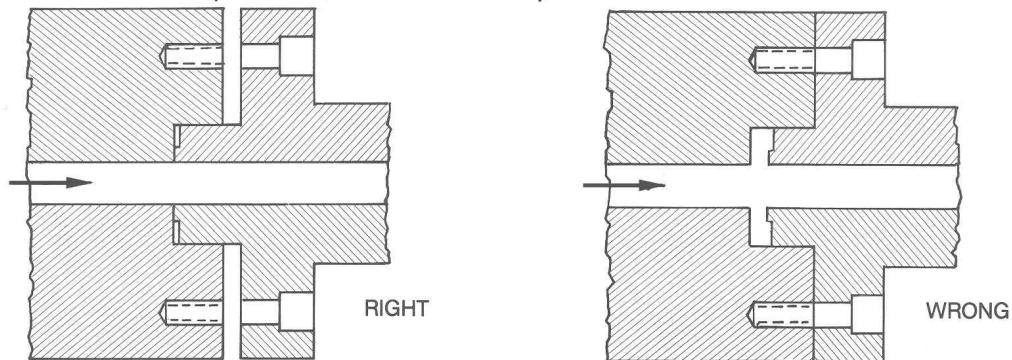
3. CHANGE IN FLOW DIRECTION



4. MELT THERMOCOUPLE OR PRESSURE GAUGE MOUNTING



5. MATING COMPONENTS (METAL TO METAL SEALS)



"Streamlining of Extrusion Dies," P.N. Richardson, SPE Journal, Vol. 14, Number 8. Reprinted with permission.

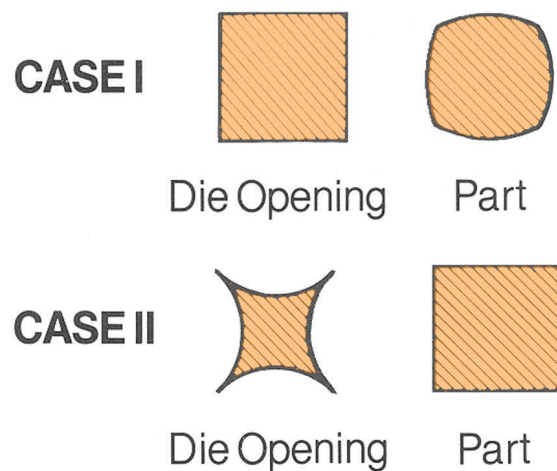
Die Sizing

The area of the die face opening is typically larger than the area of the finished product. The comparison of these cross-sectional areas is defined as drawdown. For example, for tubular products the drawdown ratio is:

$$\text{Cross-sectional Area Drawdown Ratio} = \frac{(\text{Die diameter})^2 - (\text{Mandrel diameter})^2}{(\text{Tube OD})^2 - (\text{Tube ID})^2}$$

Drawdown ratios for DuPont™ Surlyn® are typically in the range of 1.5/1 to 6/1. Drawdown is desirable because it provides tension in the melt and makes the melt easier to handle. Simple, symmetrical extrusions such as tubing, pipe, and sheeting rarely require drawdown over 3/1. More complex parts may have differential drawdown ratios throughout their cross-section.

Figure 8 — Profile Die vs Part Configuration



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The melt strength of Surlyn® is very high and it will tolerate very high drawdown ratios without tearing or breaking. However, as drawdown ratios increase, the balance of machine and transverse direction properties can be affected. Therefore, it is best to minimize drawdown ratio unless product testing shows no detrimental loss of properties.

Die sizing for profile extrusions is predominately an "art" and not a science. It is heavily dependent upon experience and is not easily reduced to guidelines, due to the almost infinite variety of pro-files which can be extruded. It is not unusual to modify profile dies several times before an acceptable part is produced. Therefore, it may be helpful to make trials with any similar dies available before fabricating a die for a new part.

Profile dies are typically flat plates (thickness of 3.2-12.7 mm; 1/8-1/2 in.), mounted on the die body. Practicality dictates this technique even though it is not optimum with respect to streamlining and resin holdup. The flow channel feeding the flat plate die is frequently circular. The diameter of the flow channel face should be approximately equal to the major dimensions of the die opening. If the major and minor dimensions of the die opening are remarkably different-then entry contouring is required. Contouring of the flow channel entry to the flat plate die is preferred in all cases.

A simple example of die vs. part configuration is shown in Figure 8. The part configuration is significantly different than the die opening. Additional differences would result from changes in land length, die temperature variation, melt viscosity, melt swell, etc.

Typical Extrusion Dies

A variety of typical extrusion die features and assemblies are illustrated in Figure 9. Also included is typical die component nomenclature.

FORMING AND COOLING

Forming Methods

There are several basic forming techniques which are used in combination with the initial cooling of extruded products. Each has many variations as they have been adapted to meet particular requirements. The forming techniques include:

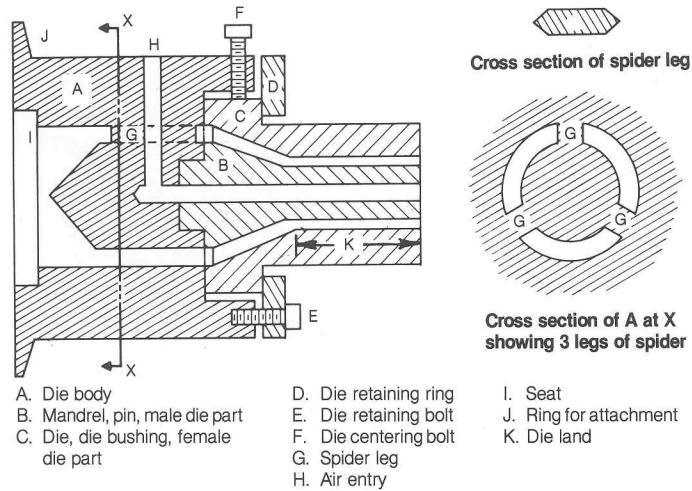
- Free extrusion
- Sizing ring
- Sizing plate or shoe
- Vacuum sizing
- Internal forming mandrel
- Chill roll
- 3-roll finisher

Free extrusion is perhaps the most simple method. The extrusion is formed by entry into a water quench tank with a guide plate to control water flow and rollers to keep the product submerged. This method is generally used for small diameter tubing and rod, and other uncomplicated cross-sections. The inside of tubing must be vented to the atmosphere or provided with low positive pressure to prevent collapsing.

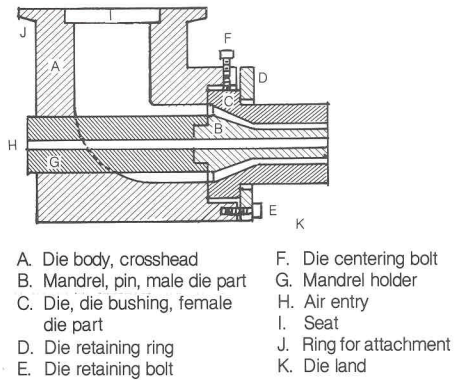
Sizing ring forming applies to circular products (tubing and pipe) and **sizing plate** or **shoe forming** (profiles or shapes) is similar, except that the products are not circular and, in most cases, are non-symmetrical. These extrusions are formed by drawdown against the ring or plate at the entry to the water quench tank or by introduction of low pressure air, if the section is hollow. Most operations require multiple sizing plates, progressively smaller in size and located downstream, as required, until final form stability is achieved.

Figure 9 — Typical Extrusion Die Features and Assemblies

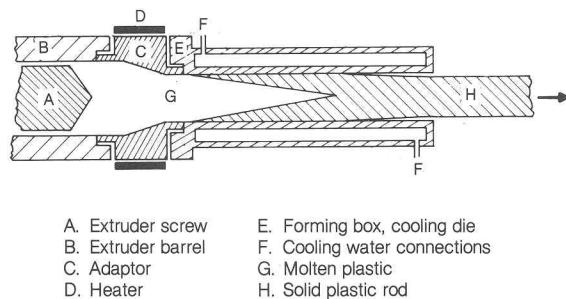
PIPE OR TUBING DIE FOR INLINE EXTRUSION



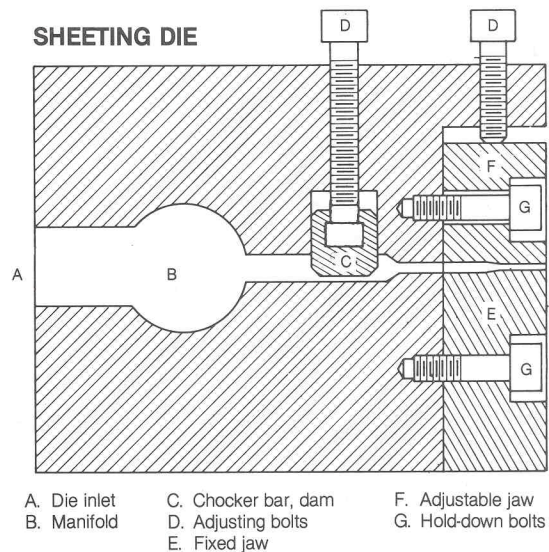
PIPE OR TUBING DIE FOR CROSSHEAD EXTRUSION



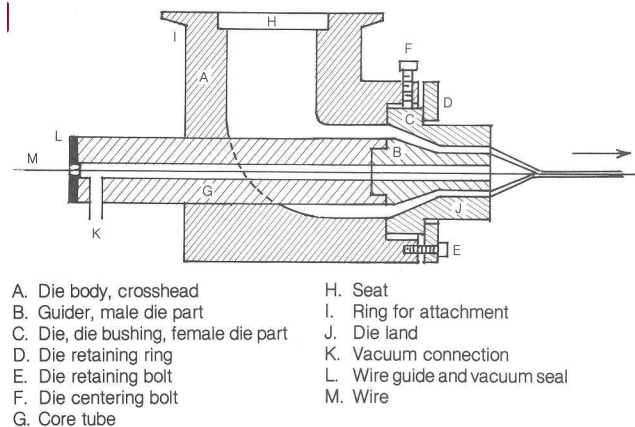
ROD DIE AND COOLING



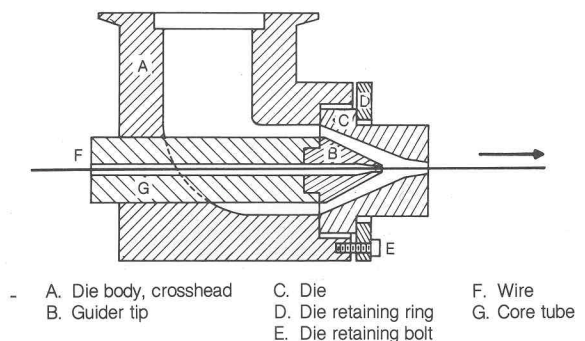
SHEETING DIE



WIRE COATING TUBING DIE



WIRE COATING PRESSURE DIE



"Introduction to Extrusion," P.N. Richardson, SPE Processing Series. Reprinted with permission.

Hollow products will accommodate the **vacuum sizing** technique (sometimes called differential pressure sizing). It does not require positive internal air pressure. The initial section of the water quench tank is sealed to permit a vacuum to be applied. As the product enters this vacuum section it passes through a series of sizing plates, usually

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placed very close together. With vacuum applied, and the inside of the section vented to the atmosphere, the differential pressure "pushes" the extrusion against the sizing plates for forming during cooling. Seals must be provided at the entry and exit of the vacuum tank in order to maintain uniform vacuum.

The **internal forming mandrel** method is used for hollow products, traditionally pipe and tubing. However, it may be considered for non-circular, hollow sections. The method involves the attachment of an extended mandrel with capability for internal water coolant circulation, in-line with the hot mandrel (pin or male die part) in the die assembly. As the product is drawn over the internal forming mandrel it is cooled and shaped. Additional cooling is provided by a water quench tank. The downstream end of the product must be kept open to the atmosphere, otherwise the product will collapse. The cold mandrel is normally tapered to prevent seizure. A cross head or offset die is preferred to allow coolant circulation from the rear of the die assembly to the cold mandrel. (Coolant entry in an inline die must be through a spider leg and is very likely to upset the temperature uniformity in the die and result in weak weld lines).

Some relatively flat, but simply formed, profiles can be extruded onto an internally cooled **chill roll** to accomplish initial or complete cooling. Use of a chill roll alone depends on thin sections and rates may be limited. A chill roll can be followed by a water quench tank. Within limits the chill roll finish can be modified to achieve various finishes on the extruded product.

Sheeting is typically produced using a **3-roll finisher** stack. Sizing is accomplished by controlling the initial nip opening. Cooling is achieved on both sides by an "S" wrap around the rolls. However, the center roll must remove adequate heat to allow the sheet to release from the roll. Therefore, roll diameters may be 61 cm (24 in.), or larger, and should be chilled to a temperature at or near the dew point.

Forming/Cooling Tools

The above forming methods involve a wide variety of specific forming and cooling tools (plates, rings, shoes, rolls, etc.). A general and important rule in the extrusion of DuPont™ Surlyn® resins is that: **All these forming and cooling tools should be kept as cold as possible.** The molten resins are prone to stick to metal parts. The most effective means of eliminating sticking is good cooling. Most often this is accomplished by minimizing metal mass to be chilled; and designing and operating for good circulation of cold water on the tooling, particularly in the area adjacent to the hot polymer contact.

Forming and cooling tools are typically fabricated of brass or aluminum. A roughening of the surfaces which contact the polymer will help reduce sticking. This can be accomplished by sandblasting or sanding with emory cloth at a right angle to the product travel. The entry side of cooling devices should have a radius.

Another technique helpful in eliminating sticking is to lubricate the melt, with careful control, just prior to contact with the forming device. Lubricants can be water or a water soluble oil. Some consideration has been given to the use of porous bronze components to allow a controlled "weeping" of lubricant.

It is common practice to direct "jets" of cooling air onto selected portions of many extrusions between the die and the forming/cooling tools. Care should be taken to prevent these air jets from cooling the die. This technique is useful to help cool thin fins and give them more strength before entering the forming tool.

Depending upon part configuration, pairs of rollers can be placed in the early stages of the quench tank to modify surface finish. One roller would be used for support; the other to "emboss" the desired finish.

Water Cooling

Surlyn® resins have melting and freezing temperatures toward the lower end of the scale for polyolefins. Therefore, they must be cooled to lower temperatures before form stability is achieved. This can result in potentially longer cooling facilities. However, very few water quench tanks are operated with good efficiency in cooling. If simple, high efficiency cooling techniques are used, Surlyn® resins can be adequately cooled in most existing installations.

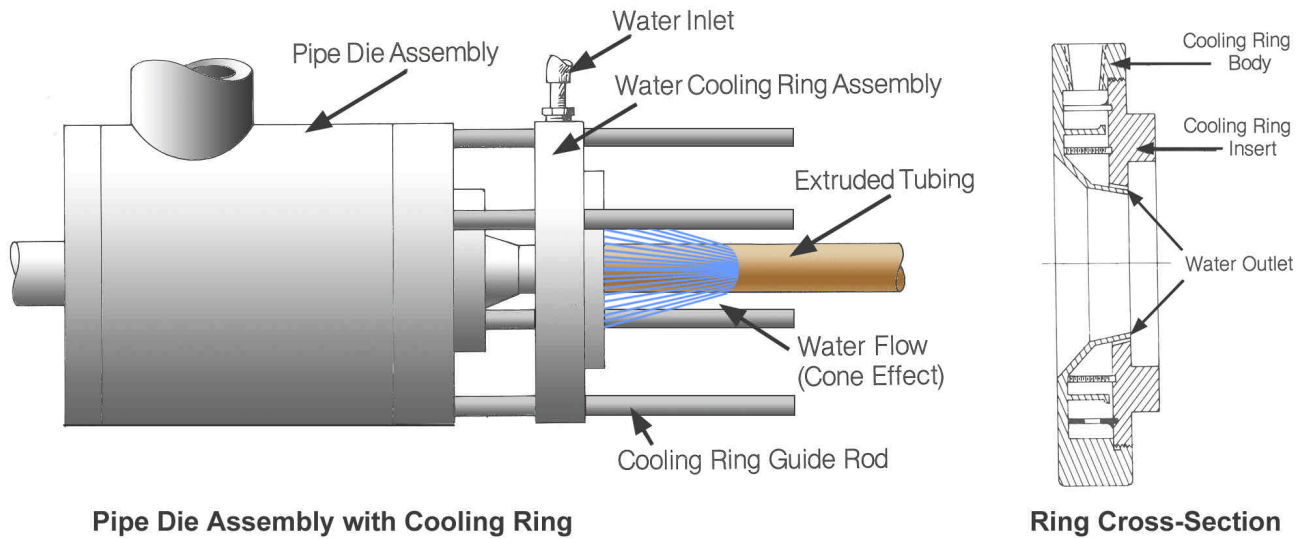
Typical quench tanks have only one or two water inlets and drains. This arrangement is most likely very inefficient. Frequently, the water flow path does not intersect the product to be cooled as the water flows from inlet to drain. In practice, the only water capable of cooling the product efficiently is the water "skin" directly in contact with the product. Therefore, **it is critical that quench water be circulated directly on and around the product to be cooled.** This can be accomplished by selective positioning of inlets and drains, or by introduction of water through submerged copper tubes aimed directly at the extrusion throughout the length of the tank. Mechanical stirring devices can also be considered (air operation preferred).

DuPont has developed a device called a water cooling ring. It was initially intended for cooling pipe but can also be used for many other sections. The cooling ring delivers a "cone" of high pressure water onto the extruded product. When the "cone" strikes the product a "sheet" of water flows downstream, as far as 1-2 m (39-78 in.), before any water falls away. This represents highly efficient use of cooling water. The cooling rings can be installed over splash pans or in sections of the quench tank which have been baffled to remove normal water inventory. The rings can also be used in series.

The water cooling ring is illustrated in Figure 7. It is shown installed on a pipe die with internal forming mandrel. Also shown is a cross-section of the cooling ring assembly. The ring can be used for initial cooling with the internal forming mandrel method. In most other methods it would be used for efficient cooling after forming is complete.

A modified version of the ring combines the functions of sizing and cooling. It can be used for circular products and would replace the initial sizing plate. It is called a water sizing ring.

Figure 10 — Water Cooling Ring



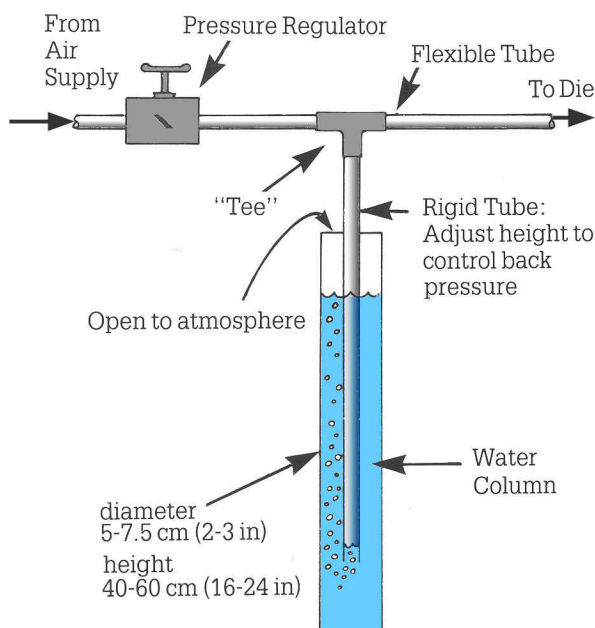
Pipe Die Assembly with Cooling Ring

Ring Cross-Section

Internal Air Pressure

Several of the forming methods for hollow products require introduction of low pressure air inside the product. This air must be delivered at a very uniform rate in order to produce parts without dimensional variation. Frequently standard pressure regulators result in unacceptable variations in air delivery. A simple manostat, or "bubbler," can be assembled from storeroom items and will permit uniform air delivery. The manostat assembly and operation is illustrated in Figure 11.

Figure 11 — Manostat ("Bubbler") Assembly and Operation



PRODUCT PACKING

Cutting/Trimming

DuPont™ Surlyn® resins may be difficult to cut or trim at the end of the extrusion line due to their inherent toughness. It is mandatory that all cutting tools be sharp. In addition, if the frequency of cutting is high, the cutting tools may get hot causing the polymer to stick. If so, it may be necessary to either cool or lubricate the cutters.

Packing

Parts made of Surlyn® resins are softer and more flexible immediately after extrusion than they will be about a day later. Aging causes an increase in stiffness, due to crystalline growth, following the heat treatment and cooling involved in extrusion. Care should be taken to preserve the part shape until completely cooled and form stability has been achieved. Any bends or dents introduced during this period may be difficult to remove. If the utility of the part is destroyed by such distortions, additional care must be taken to handle and store the parts.

IX. Extrusion Operation

Melt Temperature

Melt temperature for industrial extrusions of Surlyn® are, generally, in the overall range of 150-230°C (302-446°F). The melt temperature depends upon the melt flow index of the Surlyn® grade and the part configuration. Melt temperatures are most typically in the range of 175-200°C

The more commonly used lower melt flow index ionomers (0.7-2.5 MFI) generally perform best at the higher melt temperatures of 175-200°C (347-392°F). The high melt flow index ionomers (5-14 MFI) require lower melt

temperatures to achieve adequate viscosity for ease of handling the melt.

While industrial extrusion operations utilize melt temperatures in the above ranges, it should be noted that DuPont™ Surlyn® resins have good temperature stability at considerably higher temperatures. For example, some packaging market products utilize melt temperatures in the range of 260-320°C (500-608°F), without thermal stability problems.

It is extremely important to recognize the benefit of operating at the lowest practical melt temperature. Industrial extrusion operations are usually rate limited by cooling capacity. Therefore, all other things being equal, lower melt temperatures will require less heat removal (i.e., cooling capacity), and result in higher production rates.

The achievement of desired melt temperature, assuming adequate screw design, is dependent upon the extruder/adaptor/die temperature profile. Typical temperature profiles are listed in Figure 12, for a variety of common melt temperatures. Note that the melt temperature should be established in the extruder where mixing is possible. The head, adaptor, and die zones should only maintain

the pre-established melt temperature. If heat is added or removed in the head, adaptor, or die zones, it will not be uniform throughout the melt, and there is no opportunity to mix melt layers of different temperatures in these sections of the machine.

The change in flow (viscosity) as temperature is changed is greater for Surlyn® than for low density polyethylene.

This relationship is shown graphically in Figure 6, for resins of equal melt flow index. This inherent relationship can be used to advantage, for example:

1. Changes in flow (viscosity) for Surlyn® can be achieved by changing melt temperature in relatively small increments -10°C (18°F). Similar viscosity changes for polyethylene may require a melt temperature change of 20°C (36°F) or more. Therefore, Surlyn® will allow viscosity control without dramatic change in temperatures.
2. During resin change purging procedures it is desirable for the incoming resin to be more viscous than the machine inventory resin. If Surlyn® is being used to purge polyethylene from the machine (both equal melt flow index), then melt temperatures below 190°C (374°F) will promote more effective purging.

Figure 12 — Typical Extruder Temperature Profiles,

Barrel zones							
Rear*	Rear Center	Center	Front Center	Front	Head and Adaptor	Die	Desired Nominal Melt Temperature
150 (300°F)	150 (300°F)	150 (300°F)	150 (300°F)	150 (300°F)	150 (300°F)	150 (300°F)	150 (300°F)
150 (300°F)	175 (350°F)	175 (350°F)	175 (350°F)	175 (350°F)	175 (350°F)	175 (350°F)	175 (350°F)
150 (300°F)	175 (350°F)	200 (400°F)	200 (400°F)	200 (400°F)	200 (400°F)	200 (400°F)	200 (400°F)
150 (300°F)	190 (400°F)	230 (450°F)	230 (450°F)	230 (450°F)	230 (450°F)	230 (450°F)	230 (450°F)

*Lower than shown if drive horsepower permits; as low as 120°C (248 F)

Bridging

A bridge is defined as a flow interruption in the channel of the extruder screw, usually in the feed zone. It can be a partial or total flow interruption. The cause of a bridge is pellets sticking to the root of the feed channel, frequently followed by a buildup of pellets sticking to one another until the flow channel is totally restricted. A bridging condition will result in: (a) entrapped air and bubbles in the melt; (b) reduction of head pressure and drive motor amperage due to the starve feeding of the screw metering zone; (c) variations in motor amperage, head pressure, and die output; and (d) in severe cases, complete loss of die output, with no head pressure and minimum amperage.

Surlyn® resins are sensitive to bridging because they have relatively low melting points and an affinity for metal. They are similar to some other polyolefins, but about 10-15°C (18-27°F) lower in melting point than low density polyethylene.

Potential bridging can be eliminated by use of the preferred screw designs discussed in Section VIII, and the following operating procedures:

1. Maintain the rear barrel heat zone at 150°C (302°F) or less, if the extruder drive power is adequate.
2. Use internal screw cooling for the first 4-5 turns of the feed zone.
3. Use water cooling for the hopper feed throat jacket.

These procedures, with good screw design, should eliminate virtually all bridging. In the event that a bridge does develop, a number of operational techniques can be used for removal:

1. Rapidly increase the screw speed to maximum and maintain for 1-3 minutes. The increased delivery of pellets and higher pressure of the pellets as they are conveyed forward may dislodge the bridge. Return to a moderate screw speed for 1-3 minutes and repeat this procedure as necessary.
2. Reduce the resin inventory to the feed throat over the screw. Force feed a rod of polyethylene into the screw channel, allowing large chunks to be bitten off and conveyed forward. CAUTION.- Do not place hands or fingers in hopper feed throat. These larger pieces will melt more slowly and may mechanically dislodge the

bridge. A purge to polyethylene pellets is optional before reintroducing DuPont™ Surlyn® to the hopper.

3. If a rod of polyethylene is not available, then a purge to polyethylene may be helpful. The alternate high and moderate screw speed in item No. 1, above, should be used during the purge and subsequent operation with polyethylene.
4. Should all these techniques fail to dislodge the bridge, then screw removal and cleaning will be required. Therefore, design and operation to avoid bridging is very worthwhile to avoid a tear-down procedure.

Cooling

Cooling of extruded products deserves special consideration because this process step, most frequently, is the limiting factor in production rate. Attention is redirected to Section VIII. Extrusion Equipment – "Forming and Cooling."

Certain product defects can develop during cooling. They include:

- Raised, circular defects can be formed by water droplets splashing on the product in the air gap. The water droplet chills the surface it strikes and prevents the small area from further drawdown, leaving a raised defect.
- Depressed, circular defects can be formed by air bubbles attaching themselves to the extruded product as it travels through the quench tank. The air bubble "insulates" the product surface and slows the local cooling rate.
- The slow cooling permits more shrinkage and a subsequent depression (sink) in the surface.
- "Stick release" patterns (fine lines across the sheet) can be formed on sheeting as a result of sticking to the chill roll.

General

Specific product quality requirements

can be influenced by adjustment of operating conditions. For example:

- Surface gloss is predominantly influenced by melt temperature. Higher temperatures provide more gloss. Conversely, a styling interest in a "dry look" or dull finish can be achieved by using lower melt temperatures and lower melt flow grades of Surlyn®. Gloss is also influenced by length of air gap. Longer air gaps will improve gloss.
- Many die configurations require recombining of melt streams; for example, dies with spiders or crossheads with mandrels. The area of recombination is referred to as a weld line. Adequate temperature must be maintained in this area to assure that the weld line is as strong as the adjacent material in the finished product. A common cause of poor weld line strength

can be contamination with previous polymers that have not been fully purged from the flow paths. Poor streamlining will accentuate this problem.

Record Keeping

The maintenance of a production log or data sheet is extremely important and is frequently overlooked. Such records document production and allow retrieval of data when problems are encountered. Examples of the type of information which should be routinely recorded are given in Figure 13.

(DuPont permits copying and reuse of this Production Record.)

Figure 13 — Typical Production Record	
Job Name or Number	
Date	
Names of Operators	
Surlyn® Resin Grade and Lot Concentrates, additives, etc.	
Extruder Screw Designation	
Die-Part No. and Size	
Screens	
Cooling and Haul-off Equipment	
Extruder conditions	
Temperatures	
Barrel, Adaptor, Die (Set/Actual)	
Cooling	
Screw, Barrel, Hopper Feed Throat	
Screw Speed	
Motor Power	
Melt Temperature Melt Pressure	
Rate, kg (lbs)/hour	
Water or Roll Temperatures	
Haul-off Rate	
Product	
Thickness	
Width	
Length	
Appearance	
From "Introduction to Extrusion," P.N. Richardson, SPE Processing series. Used with permission.	

Startup Procedures

Prior to startup, the information in Sections II, Safety, and VI, Pre-extrusion Checklist, should be specifically reviewed.

Ideally, startup will be with an entirely clean extruder, adaptor, and die assembly, although this is not necessarily a requirement. Most often experience and individual plant policy dictate whether machines are cleaned or purged prior to startup with a new resin. The following startup procedure assumes: (a) startup from room temperature; and (b) startup with a clean and empty barrel and screw

(or the screw has been run "dry" from a previous shutdown with DuPont™ Surlyn® or polyethylene resin).

1. Machine heats should be turned on one to two hours prior to introduction of resin. This will assure a total heat-up and prevent excessive pressures and drive power during startup. Less time may be acceptable with some machines if experience has shown that excessive pressure and power consumption does not result. Cooling water to the screw and hopper feed throat jacket should be on during this period.
2. Machine temperatures should be set to provide a melt temperature of 200°C (392°F), for ionomers with melt flow index of 2.5 or less; or 175°C (347°F), for ionomers with melt flow index of 5 or more. This assumes that experience has not demonstrated other acceptable temperatures. It is likely that, once the operation is established, temperatures can be lowered. However, for safety reasons (primarily excessive head pressure or torque), it is preferred that initial temperatures be higher than necessary rather than lower.
3. Surlyn® should be introduced to the screw at full feed (not starve feed), and the screw speed should be moderate (30-40 % of maximum rpm). Full feed and moderate rpm will help establish good conveying without bridging. Once the extruder output is established the screw speed can be reduced. If production is off-line or down, it is best to maintain the screw speed at 5-10 rpm, unless experience has shown that a stopped screw will not cause a bridge.
4. Situations requiring resin change purging are covered below.

SHUTDOWN PROCEDURES

These shutdown procedures apply to shutdown with a given Surlyn® resin, and subsequent startup (overnight or over a weekend) with the same or similar melt flow index Surlyn®. It is also assumed that preferred screw designs and operating conditions have been used.

1. The melt temperature at shutdown should be 175°C (347°F), or less. If the operating melt temperature is higher, then screw speed should be set at 5-10 rpm, with the temperature settings reduced or temperature controllers turned off, if carefully monitored. When the melt temperature reaches 175°C (347°F), polymer feed to the screw is stopped and the screw is run dry. When polymer delivery at the die ceases, the screw is shut off. Screw cooling and hopper feed throat jacket cooling water should be left on until the machine has reached room temperature. The machine is then ready for startup as outlined above.
2. An alternate shutdown procedure is to purge the Surlyn® from the machine with polyethylene. Normal shutdown procedures for polyethylene are then followed.

Purge Procedures

Purging from other polyolefins to Surlyn® resin or vice versa, is relatively simple if the viscosity of the new resin is higher (lower flow), or if the viscosity of the two resins is similar. If the viscosity of the new resin is lower (higher flow), then the following steps may be helpful. (Note: Generally, high viscosity is low melt flow; low viscosity is high melt flow):

1. Use an intermediate polyolefin purge resin which has a viscosity intermediate between the initial and final resins.
2. Allow the machine temperature to decrease during entry of the new lower viscosity resin. This will cause it to gain viscosity and displace the hotter inventory in the machine.

The effectiveness of purging can be improved by varying the screw speed during the purge procedure. Increase the screw speed to maximum for one to three minutes; then decrease to 5-10 rpm for four to five minutes; and repeat. This procedure will upset the laminar flow patterns in the adaptor and die and result in faster, more effective purging.

Do not purge directly from heat-sensitive materials to Surlyn®. Use pre-established methods to remove heat sensitive materials based on individual plant experience. Purging of non-polyolefin and non-heat sensitive materials is usually accomplished by use of an intermediate purge resin such as polyethylene.

Barrel and Screw Cleaning

On occasion it is necessary, or helpful, to thoroughly clean the extruder barrel and screw. A procedure for cleaning with acrylic purge compound has been developed at DuPont's Technical Services Laboratory. The procedure is intended for the barrel and screw, and must be used only after the breakerplate, adaptor and die assemblies have been removed. After using this procedure, removal of the screw from the barrel is accomplished with ease. Contact DuPont for more information.

X. Coextrusion and Coating of "Bright"

The design and function of many industrial products can be enhanced by coextrusion. In addition, incorporation of various "bright" components can provide appealing decorative effects. An example of these features is illustrated in Figure 14. This product is coextruded to provide a colored Surlyn® surface adjacent to the "bright" and a clear UV stabilized Surlyn® coating over the aluminum foil "bright" These concepts offer unlimited design opportunities for a wide range of industrial products.

Typical "bright" materials are aluminum foil, stainless steel foil, or metallized flexible films, and/or laminations. Good levels of Surlyn® adhesion to metal surfaces are achieved

without priming. Product testing is suggested to confirm maintenance of adhesion through the expected product life.

The "bright" material can be passed through a crosshead die and coated or encapsulated; or it can be rolled into the surface of a DuPont™ Surlyn® extrusion, exit the die. Cleaning or degreasing of the "bright" may be necessary if the surface has residual process rolling oils or other contaminants. Such contaminants may interfere with adhesion.

The surface of "bright" material must be free of dust and dirt specks. When coated or encapsulated with Surlyn®, the dust can act as a nucleators causing moisture release and formation of tiny bubbles at resin moisture contents which otherwise would be entirely acceptable. Any dust specks may cause visual defects under clear coatings.

Coating or encapsulation of "bright" materials can be accomplished using a crosshead (or offset) die body with a pressure type or tubing type tooling assembly. These dies are illustrated in Figure 9 as "Wire Coating Pressure Die" and "Wire Coating Tubing Die."

In the pressure type die, the Surlyn® meets the "bright" inside the die and is pressured into place. The guider tip assembly should be movable, so that the distance from the end of the tip to the die face can be adjusted. Such adjustment allows placement of the guider tip, under particular operating conditions, so that polymer will flow to the die face and not "back flow" into the core tube. If polymer enters the core tube it will degrade and contaminate the "bright" material.

In the tubing type die, the Surlyn® is "drawn" onto the "bright" exit the die. Application of a vacuum through the core tube is sometimes helpful to improve process control and adhesion.

Coextrusion die designs involve a multitude of configurations to meet specific requirements. There is no one specific optimum design. Systems are in use based on principles of: (a) introducing melt streams to a combining adaptor and allowing laminar flow behavior to maintain layer segregation; (b) combining melt streams in the die land area; and (c) combining individual extrudates, exit the die. It is generally necessary to select resins and melt temperatures to achieve nearly equal viscosity at the point of combining the melts.

XI. Troubleshooting Guide

Figure 15 lists some of the problems that may occur in the extrusion operation. For each problem possible causes and remedies are suggested.

XII. FDA Status

Surlyn® packaging resins are available that comply with US FDA Regulations for use in food contact applications.

For more information contact your DuPont sales office or email us at: packaging@dupont.com.

Figure 14 — Typical Coextruded Automotive "Bright" Trim

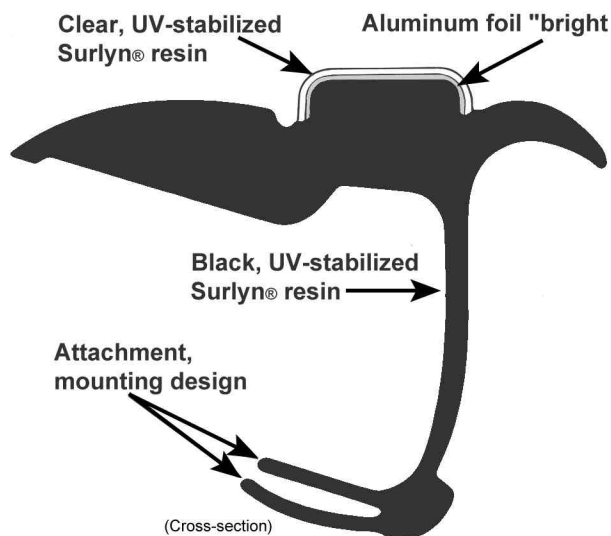


Figure 15 — Troubleshooting Guide

PROBLEM	POSSIBLE CAUSE	REMEDY
I. No extruder output	A. Hopper empty	1. Fill hopper
	B. Hopper feed throat bridge	1. Cool hopper feed throat jacket. 2. Clean feed throat coolant channel 3. Do not use hopper dryer.
	C. Complete screw bridge	1. Cool rear barrel heat zone to 150°C (302°F), or less. 2. Internal screw cooling-first 4-5 turns of feed section. 3. Cool hopper feed throat jacket. 4. Select metering type screw with compression ratio of 3.5:1 or more and recommended feed zone depth. 5. Calibrate rear barrel heat zone controller.
II. Excessive extruder drive power requirement	A. Rear barrel heat zone too low	1. Increase rear barrel temperature.
	B. Barrel heat zone over screw transition zone too low	1. Increase temperature of barrel heat zone over screw transition zone.
	C. Melt temperature too low	1. Increase barrel temperatures. 2. Increase screw rpm to increase shear working.
III. Surging	A. Screw design	1. Select screw with: <ul style="list-style-type: none"> • metering zone • compression ratio 3.5/1 or more • more shallow metering depth • feed zone at least 25% of length • gradual transition
	B. Partial screw bridge	1. See Section I-C-1 through 5.
	C. Extruder drive variation	1. Check drive performance.
	D. Stick-Slip in forming tools	1. Chill forming tools. 2. Lubricate forming tools. 3. Reduce mass of forming tool to be cooled. 4. Sandblast or sand polymer contact area. 5. Contour entry into tool. 6. Provide uniform internal air pressure.
	E. Variation in haul-off unit	1. Set belts for positive grip. 2. Check unit-mechanical/electrical.
IV. Bubbles in melt	A. Partial screw bridging – starve feed (entrapped air)	1. See Section I-C-1 through 5.
	B. Excessive moisture	1. Use resin from sealed, undamaged containers. 2. Condition resin container in operating area before opening. 3. Reduce melt temperature. 4. Keep hopper covered. 5. Use nitrogen purge in hopper. 6. Dry natural resin, concentrate, or regrind, as necessary, in dehumidified tray oven.
V. Surface defects	A. Water splashing in air gap	1. Eliminate splash or shield splash
	B. Air bubbles in quench water	1. Improve quench water circulation.
	C. Stick-Slip in forming tools	1. See Section III-D-1 through 6.

VI. Smeared, elongated surface bubbles (rough surface)	A. Excessive moisture	1. See Section IV-B-1 through 6.
VII. Dull surface	A. Resin melt index too low	1. Select resin with higher melt index
	B. Melt temperature too low	1. Increase melt temperature.
	C. Short air gap	1. Increase air gap.
VIII. Poor caliper control	A. Surging	1. See Section III
IX. Distortion	A. Inadequate cooling	1. Use colder quench water.
		2. Circulate quench water efficiently.
		3. Consider use of water cooling ring.
		4. Reduce melt temperature.
X. Poor weld line strength	A. Poor purge from previous resin	1. Use variable speed purge procedure.
		2. Streamline to allow effective purging.
	B. Melt temperature too low	1. Increase melt temperature.
	C. Poor in-line die spider design	1. Streamline spider design.
XI. Excessive shrinkage	D. Concentrate base resin not compatible with Surlyn® resins.	1. Select concentrate base resin of Surlyn® or similar ethylene copolymer resins.
	A. Excessive cross-sectional area drawdown	1. Decrease drawdown.
	B. Melt temperature too low	1. Increase melt temperature.
	C. Resin melt index too low	1. Select resin with higher melt index.
XII. Degradation	A. Excessive resin holdup	1. Increase extruder output.
		2. Streamline flow paths in adaptor and die.
	B. Poor shutdown procedure	1. Reduce temperatures before shutdown.
		2. Purge to clear degradation.
XIII. Carbon specks	C. Excessive temperature	1. Check temperature control systems.
	D. Poor purge from previous heat sensitive resin	1. See Section X-A-1 and 2.
	A. Excessive resin holdup	1. Streamline flow paths in adaptor and die.
	B. Dirty equipment	1. Extruder/adaptor/die cleaning.
XIII. Poor adhesion to "bright"		2. Avoid shutdown with high temperature.
	C. Excessive temperature	1. Check temperature control systems.
	A. Resin selection	1. Select Surlyn® resin with better adhesion potential.
	B. "Bright" surface contaminated	1. Degrease or clean surface.
XV. Bubbles over "Bright"	C. Primer selection, if used	1. Select primer with better adhesion potential.
	D. Low melt temperature	1. Increase melt temperature.
	E. Low "Bright" temperature	1. Preheat "Bright" if possible.
	F. No vacuum if tubing type die	1. Use vacuum with tubing type die.
XV. Bubbles over "Bright"	A. Dust/dirt on "Bright"	1. Clean "Bright"

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