

# DuPont™ Hytrel® Extrusion Manual

## THERMOPLASTIC POLYESTER ELASTOMER



### Introduction

The purpose of this manual is to present the latest information on the extrusion of Hytrel® thermoplastic polyester elastomer resins. It represents DuPont's continuing effort to keep users of Hytrel® up-to-date on extrusion data, processes and techniques.

The material contained in this manual is based on DuPont's technical experience in the laboratory and practical knowledge in the field.

### Hytrel® is a registered trademark of DuPont for its family of thermoplastic polyester elastomers.

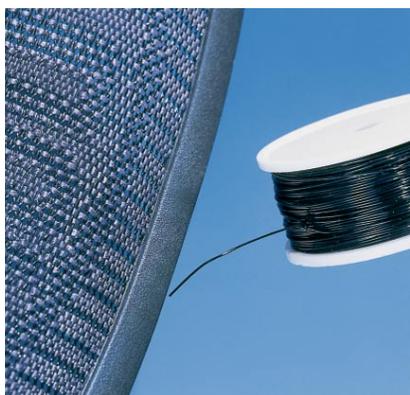
Hytrel® combines many of the most desirable characteristics of high performance elastomers and flexible plastics.

It features:

- exceptional toughness and resilience
- high resistance to creep, impact and flex fatigue
- flexibility at low temperatures
- good retention of properties at elevated temperatures
- resistance to many hydrocarbon based chemicals, oils and solvents



*The miracles of science™*



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## Hytrel® Thermoplastic Polyester Elastomer

Hytrel® thermoplastic polyester elastomers are block copolymers, consisting of a hard (crystalline) segment of polybutylene terephthalate and a soft (amorphous) segment based on long-chain glycols. Properties are determined by the ratio of hard to soft segments and by the make-up of the segments. Most grades of Hytrel® do not contain or require additives to enhance their properties, except for specific applications. In such cases, specially compounded grades, as well as masterbatches (concentrates) are available.

The grades of Hytrel® are grouped into four categories:

1. Standard General Purpose grades, which exhibit versatile processing characteristics, are generally lower in cost, and are suitable for many extrusion applications;
2. High Performance grades, which can provide an extra margin of mechanical performance for the more demanding applications;
3. Specialty grades provide special properties or processing characteristics;
4. Masterbatches or concentrates contain relatively high concentrations of specific property-enhancing additives for blending with other grades of Hytrel®. A masterbatch cannot be used by itself, it should be used in specified let down ratios.

Hytrel® thermoplastic polyester elastomers are supplied as cylindrical pellets, having a bulk density of about 700 kg/m<sup>3</sup>. They are packaged in 25 kg multiwall paper bags with a moisture barrier inner wall. Palletized units contain 40 bags, or 1000 kg net weight, wrapped in film on disposable wooden pallets. Most grades of Hytrel® are also available in bulk packages with a moisture resistant liner. For details on bulk quantities, contact your regional DuPont representative.

## Extrusion Applications and Grade Selection

The excellent properties and processing characteristics of Hytrel® qualify it for many demanding applications. Such properties as mechanical strength, durability, dynamic flex performance, fluid and chemical resistance, and wide service temperature range, have brought benefits in many different extruded products, including:

- hose and tubing
- belts
- extruded profiles
- hose mandrels
- rope and cable covers
- sheeting and films
- wire and cable

Many Hytrel® grades are suitable for extrusion processes. Selection of the most appropriate grade for a particular application, in terms of properties and serviceability, should be made by referring to the general Hytrel® product literature, as well as “Engineering Polymers for Extrusion Applications,” available from your DuPont Engineering Polymers representative or our website, [plastics.dupont.com](http://plastics.dupont.com) or [hytrel.com](http://hytrel.com).

Processing considerations, as far as they relate to extrusion, should also take into account the following points:

- The softer grades are more suitable for free extrusion of solid profiles. These are the Hytrel® types having Shore D hardness up to and including 55D, in particular those with low melt flow rates (MFR).
- Complex profile extrusion using vacuum calibration dies is generally not possible with Hytrel®, although it may be possible to achieve acceptable results in certain “hollow” profiles with the harder grades or those with low MFR.
- The hardest grades are best for vacuum calibration of tubing. Those grades of shore D hardness 45D and above give excellent results in this process.
- Intermediate properties may be obtained by mixing Hytrel® types (pellet blending) before extrusion, provided that appropriate processing conditions are used. However, it is wise to consult your DuPont representative for specific recommendations.

## Melt Properties

The ability to process Hytrel® by a specific extrusion technique depends largely on the characteristics of the melt, which are determined by the grade of Hytrel® selected, and by the processing conditions within the extruder.

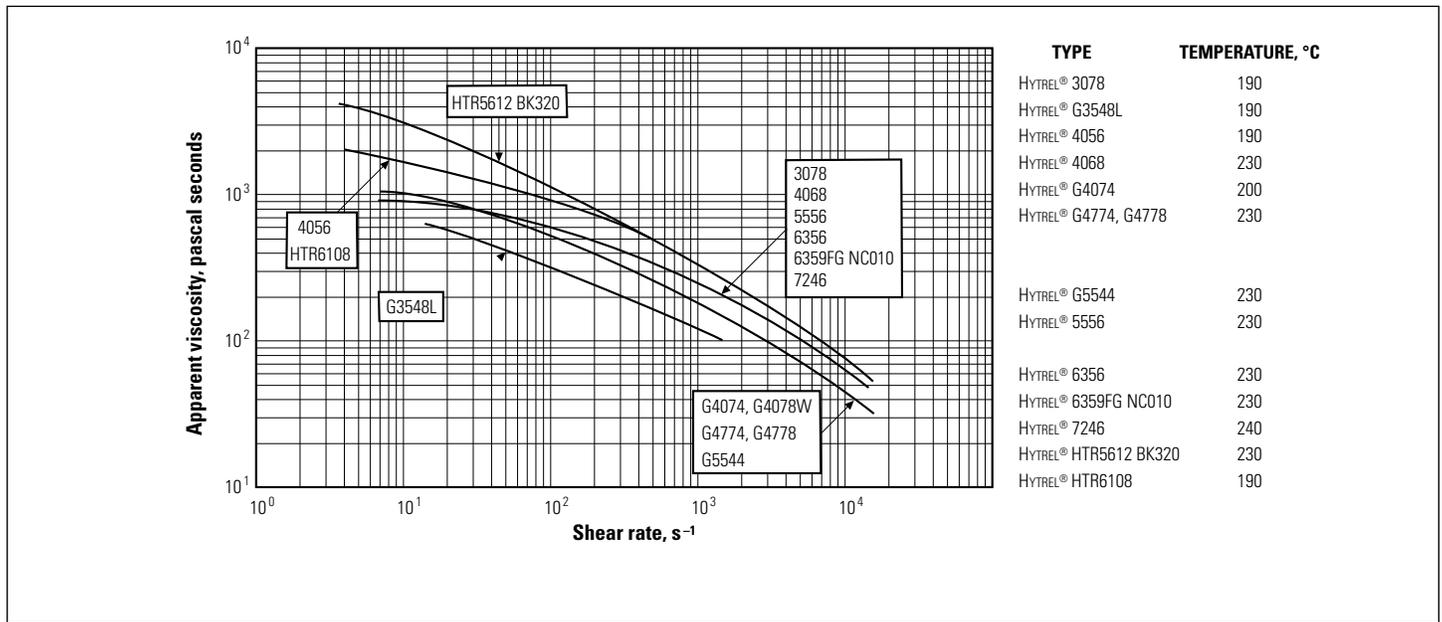
Some general melt properties should be considered in extrusion of Hytrel®:

- All grades have a relatively sharp crystalline melting point which increases (and becomes sharper) with increasing hardness and crystallinity.
- Melt viscosity is strongly dependent upon melt temperature, and this dependency increases with increasing hardness.
- Crystallization rate generally increases with increasing hardness; therefore, the ability to supercool the melt without onset of crystallization decreases with increasing hardness.

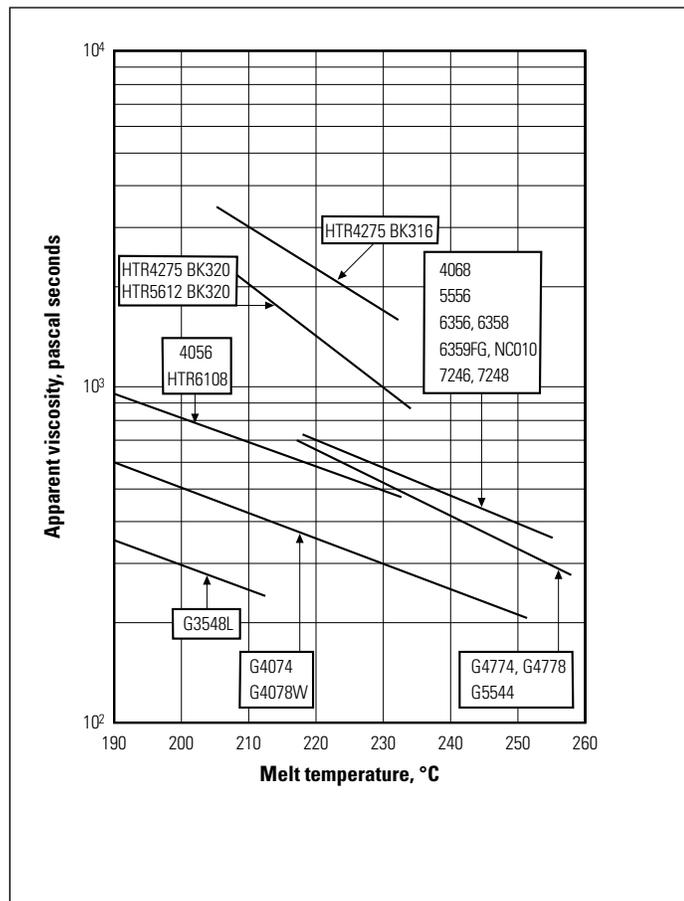
Melt viscosity as a function of shear rate and temperature for Hytrel® extrusion grades is shown in **Figures 1** and **2**.

Hytrel® polymers have relatively flat viscosity vs. shear rate curves, especially at the low shear rates which are typical for extrusion. This means, for example, that high shear screw and die designs will not reduce the melt viscosity of Hytrel® as much as with some other polymers, but may cause undesirable local temperature increase (see section on “Screw Design” on page 3).

**Figure 1. Melt viscosity at processing temperature**



**Figure 2. Apparent melt viscosity vs. temperature at a shear rate of 100 s⁻¹**



From **Figure 2** it can be seen that a significant change in melt viscosity can result from a relatively small change in melt temperature. The extrusion melt temperature can therefore be decreased (within limits) to provide greater melt strength for improved stability of the extrudate. It also means that good control of melt temperature is an important factor in successful extrusion of Hytrel® resins.

## Basic Extrusion Equipment

### General design of extruder

Experience has shown that the best results with Hytrel® are obtained with a single screw extruder design. Twin screw extruders tend to generate excessive shear, and are not recommended. Vented machines are not necessary, since there are no significant volatiles present in Hytrel®.

The use of melt pumps (gear pumps) is possible with Hytrel®, and these are discussed later (see page 5).

The emphasis should be on the uniformity and quality of the melt produced. A constant delivery of homogeneous melt of uniform temperature, with the ability to maintain the desired melt temperature over a range of screw speeds, should be the objective for good extrusion.

The extruder drive should provide good speed control and adjustment over a wide speed range. The drive should have automatic current limitation to prevent screw breakage as a result of excessive torque.

## Safety devices

As well as automatic current limitation in the extruder drive, the extruder should be equipped with the following protective devices in order to prevent personal injury or damage to the extruder:

- A pressure transducer installed in the flow channel immediately after the screw and before the breaker plate/screen pack. The pressure indicator should have an alarm setting for high pressure and ideally also a cut-out setting designed to stop the extruder screw before any damage can occur.
- As a further safety option, a high pressure rupture disc should be installed in the same area as the pressure transducer, designed to rupture at a pressure above the alarm/cut-out settings on the pressure indicator.
- An ammeter, installed to show the drive motor current, can be a useful indicator to help monitor start-up and running torque on the screw. Abnormally high motor current normally means insufficient temperature in parts of the extruder, and low or fluctuating current may indicate feeding problems or even wrong screw design for the material.

## Materials of construction

Hytrel® thermoplastic polyester elastomer in the molten state is non-corrosive to metals. Screws should have hardened (nitrided) surfaces but need not be made from corrosion-resistant alloys.

## Material hopper and feed throat

Overhead or tangential-type feed throats as normally provided on single screw extruders work well with Hytrel®. Water cooling of the throat is recommended to prevent excessive heating of the resin as it enters the screw, which could result in “bridging” of the material at this point, and also to serve as protection for the drive bearings.

## Dryer

As explained later (see Extruder Operation, p. 6), Hytrel® must be dry when processed — we recommend a moisture content of 0.08% or less to ensure that no significant hydrolytic degradation of the polymer or loss of viscosity occurs during extrusion. This moisture level can be achieved by drying the polymer. A suitably sized dehumidifying hopper can be fitted to the extruder, or alternatively a small sealed hopper may be used, which is then fed directly and continuously from a separate dehumidifying drying unit. If no drying is employed at all, it is especially important to ensure that Hytrel® is not exposed to atmosphere for more than a few minutes once the bags have been opened (see Extruder Operation).

## Extruder barrel

Extruder barrels which are suitable for use with common thermoplastics such as nylon, PVC or polyolefins are usually suitable for Hytrel®.

Length-to-diameter ratios of 24:1 or higher, provide the best melt quality for good extrusion.

Small clearances between the screw flights and the barrel wall are important to prevent backflow of molten resin and possible surging in extruder output. It is suggested that clearances of 0.08 to 0.10 mm are maintained for extruders up to 50 mm screw diameter, or 0.10 to 0.16 mm for larger extruders. These clearances should be checked from time to time, and refurbishment of the screw or barrel carried out when necessary.

Modern extruders are sometimes provided with an intensively cooled barrel feed zone containing axial or radial “grooves” to encourage positive feeding of granules. Some of these grooved barrel designs have been found to cause excessive shear with Hytrel® during the melting/compression stage, with consequent rapid temperature build-up and high motor drive current. Other problems may occur with the softer Hytrel® grades due to premature melting/sticking of the granules in the barrel grooves. Such barrels may therefore be unsuitable for processing Hytrel®.

It is recommended that the barrel is equipped with at least four heat control zones (3 for small extruders) and the temperature of each zone controlled by a separate thermocouple and proportional control instrument. Efficient cooling should also be provided by air blowers or water, independently controlled for each zone.

## Screw design

The most important element of an extruder is the screw. Its design can mean the difference between successful and troublesome processing of any thermoplastic, and Hytrel® is no exception.

For most Hytrel® extrusion purposes good results are obtained with simple 3-zone screws, having approximately equal length feed, transition (compression) and metering sections. The length-to-diameter (L/D) ratio should be ideally 24:1 or higher for good uniformity of extrudate (i.e., minimum temperature and pressure variations). Compression ratios should be between 2.5 and 3.5:1, as determined by the depth of feed zone channel divided by the depth of metering zone channel (“apparent compression ratio”) for constant pitch screws. The depth of channel in both feed and metering sections is important: if the feed channel is too deep and not sufficiently long, particularly with large diameter screws, it can cause poor feeding and irregular output with some harder Hytrel® types.

If the metering channel is too deep, it can result in non-uniform temperature distribution through the melt, while a metering channel which is too shallow can result in overheating of the melt due to excessive shear.

The screw should have a rounded or conical tip to avoid “dead spots” in front of the screw where degradation of polymer could occur.

A typical screw of the 3-zone, gradual transition type suitable for processing Hytrel® is shown in **Figure 3**.

The use of complex designs incorporating high shear zones, intense mixing devices, decompression zones, etc are not recommended for Hytrel®. They can cause excessive local heat build-up due to intensive shearing action, and usually cause difficulty in achieving the desired melt temperature, as well as possible high motor drive torque.

However, certain designs of “barrier” screw have been found to be very successful with Hytrel®, particularly in achieving constant melt characteristics for critical extrusion operations (such as high speed tubing extrusion). Such screws are designed to separate the solid and molten phases of the material while providing relatively gentle and controlled shear over the “compression” section of the screw.

Internal screw cooling is not normally desirable for Hytrel®, although limited cooling of the screw in the feed zone has been found to be successful in eliminating problems of irregular feeding (fall-off in output) in larger extruders.

For further details of screw design for Hytrel®, please contact your DuPont representative.

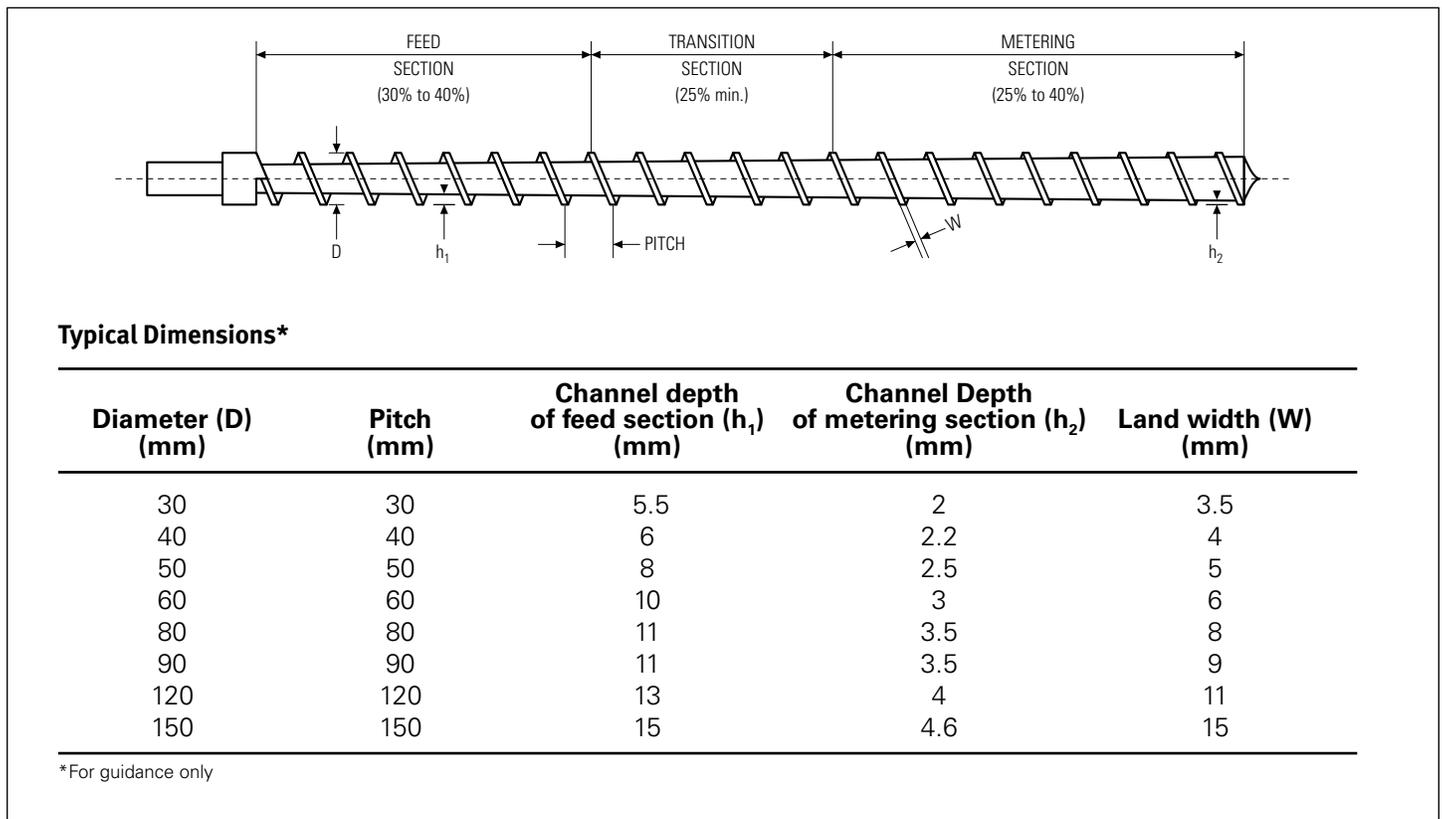
## Screens and breaker plate

A breaker plate of streamlined design (e.g., counterbored on both sides) is usually clamped between the end of the extruder barrel and the head. It acts to convert the material from the screw to a linear flow, and also develops some back-pressure on the screw as well as supporting the screen pack made from several layers of fine wire mesh. A screen pack is used for two purposes: to remove any impurities or unmelted material from the melt stream; and, to ensure sufficient back-pressure at the end of the screw to help create a homogeneous melt and constant output pressure.

The screen pack should consist of two 80 mesh or finer screens, supported by a 40 mesh screen on the downstream side, next to the breaker plate. For critical operations, where ultimate melt cleanliness is required, finer screens (120 or 150 mesh) can be used.

Regular replacement of screens will be necessary to avoid excessive pressure drops developing across the screen pack, and consequent loss of flow. Where fine mesh screens are used, this replacement may be necessary every 12–24 hours of running, or whenever an extended shutdown occurs, or when there is a change of polymer type being extruded.

**Figure 3. Gradual transition screw**



Good external heating is essential in the breaker plate/ clamp area of the extruder. Sufficient heating capacity should be provided to rapidly raise the temperature in this region during start-up to the normal processing setting, in order to ensure that any residual polymer is thoroughly melted. Because the breaker plate (head clamp) area is usually such that a large amount of heat is lost to the surrounding air, the heater design in this zone is critical.

## **Adaptor, head and die**

Both the adaptor and the head must be of a streamlined design. Flow channels should not contain sudden changes in cross section, surface interruptions (caused, for example, by mismatched assembly joints or damage), or other effects which might cause "dead spots." Such small areas of flow stagnation can give rise to localized polymer degradation and subsequent release of degraded resin into the melt stream. Similar problems can occur if flow velocity is reduced due to large cross sections in some flow channels.

Adequately sized heaters must be provided for the adaptor since it is generally a heavy piece of metal. It is especially important to control the temperature of the adaptor and head separately, since they usually differ greatly in size and energy requirements.

The die, where it extends beyond the extruder head, should also have its own thermocouple and temperature controller.

For head and die designs for specific extrusion processes, such as tubing extrusion, please refer to the appropriate process in the Extrusion Processes section of this manual.

## **Melt pumps**

Where used, melt pumps (gear pumps) can be successful in providing a very constant output rate in processes such as high speed tubing extrusion. They are usually installed before the extruder head and fitted with pressure transducers both upstream and downstream, so that this pressure differential can be used to control the extruder screw speed. Care should be taken to ensure that the design of the melt pump does not cause excessive shear heat to be generated (for example, a small pump running at too high speed), otherwise the downstream melt temperature may become excessive.

## **Instrumentation**

The function of an extruder is to pump molten thermoplastic at a constant rate and temperature. Sophisticated instrumentation is a prerequisite for quality production. To gauge extruder performance it is important to determine the pressure and temperature of the melt, as well as to provide adequate methods of control.

## **Pressure indicators**

Melt pressure should be monitored during extrusion, particularly at start-up. Recording and monitoring of melt pressure during start-up will indicate if there is proper flow of the material or if a feed problem or freeze-off situation exists. During production, pressure changes will also indicate output and viscosity changes of the molten plastic.

For accurate measurement and rapid response, a diaphragm-type transducer with electronic indicator is recommended. The most common location for the transducer is immediately before the screen pack, since this is where high pressure is most likely to be generated. However, it may also be advantageous to install a second transducer in the extruder head area to indicate output pressure closer to the die.

Long-term or short-term pressure fluctuations may influence the quality and uniformity of the product, and for this reason it may be beneficial to continuously monitor melt pressure by linking the transducer output to a data logging device, or chart recorder.

## **Temperature controllers**

Relatively small temperature fluctuations in the extruder, particularly in the front end of the barrel, head, or die, can greatly influence extrudate quality and output rate because the melt viscosity of Hytrel® is strongly temperature dependent. Therefore, the type of temperature control device used is of considerable importance.

To maintain optimum temperature control and a thermally homogeneous melt, the controller should be of the proportioning or P.I.D. type. An on-off controller is not recommended for use with Hytrel®. A temperature fluctuation of more than  $\pm 2^{\circ}\text{C}$  should be cause for concern; temperature variations of this magnitude can produce excessive viscosity fluctuations in the melt and unacceptable dimensional variability in critical extrusion processes. If wide fluctuations in temperature readings are experienced, it is possible that the heater capacity (wattage) is incorrect for the particular zone to which it is fitted, or that the controller is badly tuned.

Temperature feedback to the controllers is provided by suitably positioned thermocouples in each temperature zone of the barrel, adaptor, head and die. It is important that these thermocouples are positioned close enough to the melt stream to accurately register the temperature of the metal immediately surrounding the melt.

## **Melt thermocouples**

Thermocouples that indicate the actual melt temperature of the extrudate ("mass temperature") are useful when extruding Hytrel®. For rapid response they should be located either in the adaptor or, preferably in the die, as close to the melt stream as possible.

The use of a hand-held needle pyrometer to check the actual melt temperature before start-up should be encouraged. Measurements should be made at normal running screw speed, after purging for sufficient time to allow temperatures to stabilize.

Infra-red (non-contact) thermometers may also be used to check melt temperature when running, but should not be relied upon unless they have been previously checked against a thermocouple (needle pyrometer) in the melt stream, since their effectiveness can be influenced by factors such as color and reflectiveness of the extrudate.

## Extruder Operation

### General resin handling

Hytrel® is normally supplied in 25 kg moisture resistant, sealed bags. For bulk quantities, contact your regional DuPont representative.

In extrusion processing operations, the highest possible levels of cleanliness should be maintained in the preparation, processing, and reworking of the material, in order to prevent dust particles or other forms of contamination from entering the extruder.

Automatic granule conveying systems, sealed hoppers, careful opening of bags, and proper handling of regrind material will contribute greatly to the overall cleanliness and quality of the finished product.

### Safety precautions

All safety practices normally followed in the handling and processing of thermoplastic polymers should be followed for Hytrel® polyester elastomer. The polymer is not hazardous under normal shipping and storage conditions.

During processing, if recommended temperatures and hold-up times are exceeded, Hytrel® may degrade and decompose with evolution of gaseous products. Usually, under normal process temperature and throughput conditions, the risk of decomposition of these resins is minimal.

Depending on time and temperature conditions, the fumes evolved by excessive thermal degradation of Hytrel® may cause toxic vapors to be generated (principally tetrahydrofuran). Please refer to bulletin "Safety and Handling Precautions for Hytrel®" for more details.

As with all thermoplastics, thermal burns from contact with molten polymer are a potential hazard.

Compounding ingredients or additives may present hazards in handling and use. Before proceeding with any compounding or processing work, consult and follow label directions and handling precautions from suppliers of all ingredients.

## Moisture pick-up and drying

Hytrel® granules are supplied in moisture resistant packaging with a moisture level of less than 0.1%. However, when exposed to air, the granules pick up moisture, which may result in bubbles or streaks appearing in the extrudate.

Even when only a relatively low level of moisture is present (not enough to be visible in the melt), there is likely to be a loss of viscosity and possible loss of properties in the finished product. This is because at temperatures above the melting point, excessive moisture causes a degree of hydrolytic degradation of the polymer.

Generally, no significant degradation of the polymer or loss of viscosity in extrusion occurs if the moisture content is kept below 0.08%. This is the maximum moisture level recommended for extrusion of all grades of Hytrel®. When dry polymer is subjected to 50% relative humidity, the level of 0.08% moisture may be reached in exposed material in about 30 minutes, depending on the grade, while at 100% relative humidity it will occur even faster.

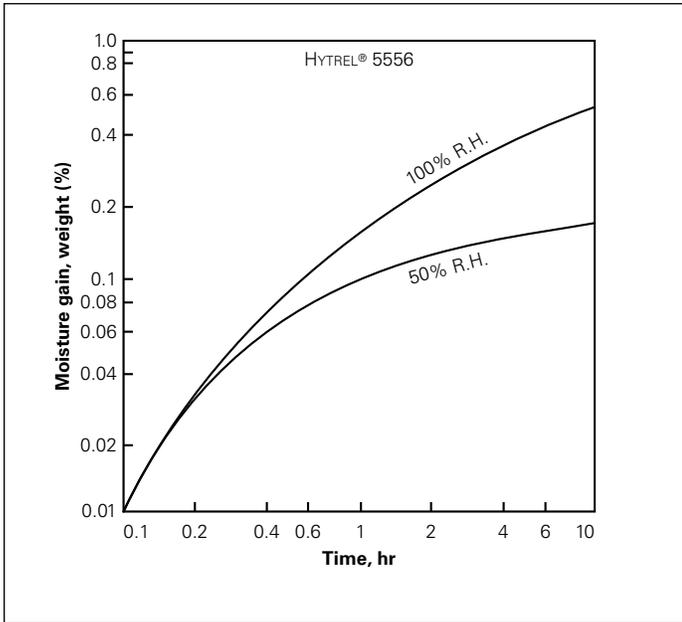
The rate of moisture absorption of a typical Hytrel® grade is shown in **Figure 4**, while the equilibrium moisture level for several Hytrel® grades is given in **Table 1**.

**Table 1**

Type of Hytrel®	Equilibrium moisture level (% after immersion, 24 hr)
Hytrel® G3548L	5.0
Hytrel® 4056	0.6
Hytrel® 4068	0.7
Hytrel® G4074	2.1
Hytrel® G4078W	2.1
Hytrel® HTR4275 BK316	0.5
Hytrel® G4774	2.5
Hytrel® G5544	1.6
Hytrel® 5556	0.6
Hytrel® HTR5612 BK320, HTR4275 BK320	0.5
Hytrel® HTR6108	0.2
Hytrel® 6356, 6358	0.5
Hytrel® 6359FG NC010	0.3
Hytrel® 7246, 7248	0.3
Hytrel® 8238	0.3

It follows from the above that Hytrel® granules which have been exposed to atmospheric humidity for any significant time must be re-dried before use.

**Figure 4. Moisture absorption at ambient temperature**



## Drying

As explained above, Hytrel® thermoplastic polyester elastomer must be dry before processing. When used immediately from sealed undamaged bags, it may be possible that the moisture level is below the recommended limit of 0.08%, however in order to guarantee the elimination of potential problems due to moisture, it is strongly recommended that all resin is routinely dried. In circumstances where bags are damaged or material from opened bags or regrind is used, the resin must be dried.

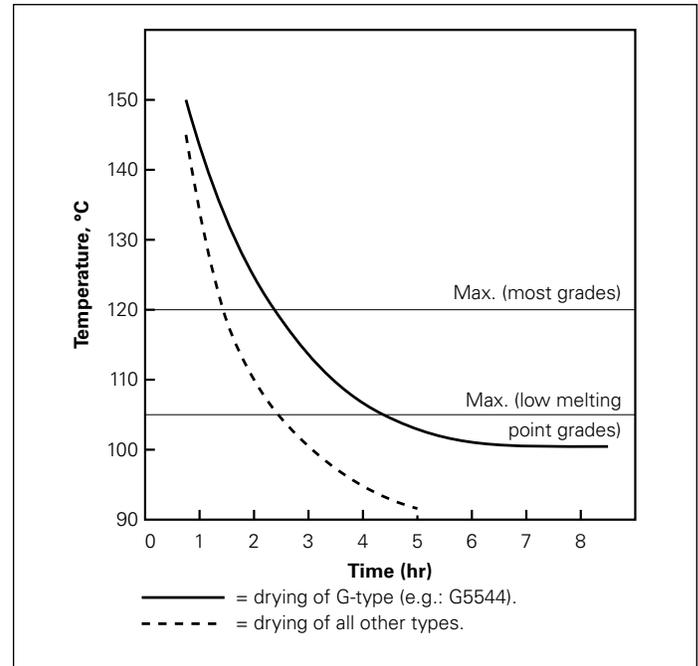
In many cases, hot air ovens may not be adequate. It is strongly recommended that dehumidifying (desiccant) dryers are employed, in order to ensure effective drying under all ambient humidity conditions.

Particularly in the case of critical extrusion operations, such as vacuum calibration of tubes to small tolerances, it has been found that extruder output may fluctuate slightly with changing moisture levels and temperature of the granules in the hopper. For this reason, again, the use of a dehumidifying drier under conditions of fixed temperature and granule residence time is always recommended.

The dryer should either be directly mounted on the machine hopper (hopper dryer) or, if it is a separate unit, should incorporate automatic material transfer to a sealed feed hopper.

Drying time and temperature will depend on the initial moisture level in the material, as well as the type of dryer or oven used. However, general guidelines for drying Hytrel®, which are based on laboratory and industrial experience, are shown in **Figure 5**.

**Figure 5. Recommended guidelines for drying Hytrel®: drying time vs. temperature**



## Start-up, shut-down and purging procedures

### Start-up

Start-up technique is important, as it involves the safety of both operating personnel and the equipment.

Start-up techniques vary depending on whether or not the machine is clean.

#### A. Clean machine

To start up a clean, empty extruder, set the temperature controllers for the die, head, adaptor and barrel zones at the recommended operating temperatures for the particular resin grade being used (refer to processing data in **Table 2**).

At this time the operation of the heaters and controllers should also be checked. When all zones reach their operating temperatures, they should be allowed to “soak” for 30–60 minutes (depending on the size of the extruder) before feeding resin to the screw. Turn on the feed throat cooling water.

When all heating zones have been at their set temperatures for 30–60 minutes, turn on the screw at slow speed (5 to 10 rev/min) and start feeding resin through the hopper. When the melt appears at the die it should become smooth and glossy after a few minutes, and both the melt temperature and head pressure should level out.

It is desirable to use a hand-held needle pyrometer to monitor melt temperature at start-up. The extruder pressure and motor amperage should be checked and monitored.

When the melt quality appears to be uniform and there is no sign of any lumps or bubbles, increase screw speed to normal running speed to check final melt quality and temperature before starting production.

If a melt pump is being used, then the start-up procedure may be slightly different to the above — please refer to the equipment supplier's guidelines.

### **B. Full/partly full machine**

Sometimes the extruder has been shut-down after extruding another polymer, and may be partly filled with that material. In this case, the melting point of the residual polymer should be considered during start-up.

Be careful when starting a full machine to prevent bridging in the feed section, localized overheating (which may result in polymer degradation), and cold spots (plugs of unmelted resin occurring primarily in the adaptor or head zones).

Set all controllers (with the exception of the first barrel zone, which can be set to its normal running temperature) to a value which is 10–20°C above the nominal melting point of the grade to be processed (refer to **Table 2**). If the residual material in the machine has a melting point which is above that of the new resin, then the controllers should be set higher than the melting point of the old material.

When the controllers have reached these temperatures and have been held for 30–60 minutes, slowly increase the screw speed to about 10 rev/min until molten polymer flows from the die. It is important at this time to check for any excessive die pressure or drive motor current which may indicate a plug of unmelted resin. Start feeding fresh resin through the hopper, again checking for excessive pressures or motor current. Run the extruder slowly while purging with fresh resin for up to 30 minutes, or until a flow of clean and homogeneous molten polymer is obtained.

During the purging process, the screw speed should occasionally be increased to normal operating speeds, or above, for short periods. This will help to release any old degraded or unmelted resin from the internal surfaces of the extruder.

When a smooth flow of clean molten polymer with no lumps of old or degraded material has been obtained, all temperatures should be re-set to normal running settings, and slow purging continued until the temperatures have re-stabilized.

At this point, melt temperature should be checked using a needle pyrometer, with the screw speed at the desired running speed.

### **Shut-down and purging procedures**

For brief stoppages of 30 minutes or less, no action is required, other than a short purge with new resin after start-up.

If the extruder is to be shut down for longer periods, empty the barrel and turn off the heaters. It can be advantageous to purge a small quantity of a low melting point Hytrel® grade (such as 4056) through the extruder immediately before shutting down. This will help minimise purging time on start-up. If the extruder is so equipped, barrel cooling may be used to cool the residual melt rapidly and prevent any polymer degradation. During the next start-up, any material contained in the machine should be expelled and not used. Venting of gases which may be generated should be considered (see Safety Precautions).

Purging with polyethylene or other polymers is not normally advised, except just prior to strip-down of the equipment for cleaning (see Cleaning). This is because it can take a long period after start-up to completely eliminate traces of polyethylene from the Hytrel®.

Any purging should be done with temperatures set to a value which is above the melting point of the resin being purged.

Special purging compounds (e.g., cast acrylic resins) may be used to purge the extruder. Since these resins are often cross-linked, or behave in such a way that they do not melt, but only soften, it is normally necessary to remove the head by separating at the breaker plate before purging. If this is not done, unsafe drive torque and pressures may result which may damage the machine or injure the operators.

### **Equipment cleaning**

Occasional dismantling and cleaning of the extruder screw, adaptor, head and die components is recommended. The optimum frequency for these strip-downs will depend on the number of start-up/shut-down operations and the number of different resins which have been used in the extruder.

Cleanout procedure consists of purging the extruder with high/medium density polyethylene or polypropylene and then removing the die, adaptor, and head from the extruder. With the head removed, the screw and barrel may then be cleaned by running the machine while feeding a suitable cleaning compound (e.g., cast acrylic type) through the machine. The adaptor, head and die should be completely dismantled for cleaning.

Complete removal of the screw for thorough cleaning is essential from time-to-time, since it is the only way to ensure that hard particles of degraded polymer and other residues are properly removed from the screw and barrel surfaces.

The larger quantities of resin can then be removed from the screw and other components by scraping while still hot. This may be followed by wire brushing or the use of special scouring pads. If need be, the screw should be polished to remove carbonized residue from the root of the screw. A rotating wire brush attached to an extension rod on a suitable power tool should be used to clean along the full length of the extruder barrel.

Stubborn residue may be removed from the head and die parts by burning with a propane torch; this method, however is not generally encouraged, since flammable and toxic gases may be formed. A much superior method is to immerse the parts in a hot fluidized sand bath, or by using cleaning ovens which are specially designed for the purpose. With suitable fume extraction, these methods are fast and thorough, and parts are left ready for re-installation.

## Adding concentrates and pigments

### Concentrates

DuPont offers several concentrates for use with Hytrel®; these are:

- Hytrel® 40CB, 41CB — Carbon black concentrates
- Hytrel® 20UV — Ultraviolet light stabilizer
- Hytrel® 30HS — Heat stabilizer
- Hytrel® 51FR, 52FR — Flame retardant concentrates

In all cases the concentrates are designed to be tumble blended with any grade of Hytrel® before extrusion, to enhance specific properties. Further information and recommended let-down ratios for blending of the concentrates is given in the relevant individual product data sheets.

With the exception of Hytrel® 52FR which has a melting point of 205°C, all of the concentrates are dispersions based on a low melting point Hytrel®. Even though they are normally used in low concentrations as indicated above, three of the concentrates — Hytrel® 30HS, 51FR and 52FR — have some hazards or off-gasses associated with their active ingredients. Therefore these products require some special precautions when handling and processing, and the specific material literature should be carefully studied.

### Pigments

Most Hytrel® grades are available in custom colors, based on a minimum order quantity. However, for many extrusion applications, it may be desirable to add pigments or color concentrates directly at the extruder. The most convenient way to do this is by means of a color masterbatch, in granule form. Ideally, these masterbatches should be based on a low melting point grade of Hytrel®, and such masterbatches are available (for example, from Wilson/Poly-One company). Some "universal" masterbatches on the market have been found to give satisfactory results in extru-

sion of Hytrel®, but performance of individual products need to be tested by the user. Let-down ratios of 1–3% are typically used for most color masterbatches.

Masterbatches based on PE, Nylon, PVC or other resins are generally not recommended, although acceptable results may be found with LDPE based masterbatches in non-critical applications. Some powder and liquid pigments have been evaluated with Hytrel® and may give acceptable results, however they are not as convenient to use as masterbatches.

For black pigmentation, especially where good outdoor aging is required, the use of Hytrel® 40CB or 41CB is always preferred.

## Recycling of scrap

The good thermal stability and completely thermoplastic nature of Hytrel® allows use of scrap (regrind) from the extrusion process. Hytrel® can be reground and blended with virgin polymer at a level up to approximately 50%, assuming the polymer has been correctly processed. At all times, care should be exercised to ensure that it has not been degraded and is free from contamination.

Chop scrap into chips approximately the same size as the original pellets. Use a scrap grinder with well adjusted sharp knives, as Hytrel® is slightly rubbery by nature.

Dry all regrind material and blend well with virgin polymer to ensure uniform quality.

Melt Flow Rate (MFR or MFI) checks are a practical way to monitor quality of regrind on representative samples, and is a useful quality control tool for both finished products and regrind material.

Melt Flow Rate, in effect, measures flow of molten polymer through a restricted orifice under standard conditions of material pressure and temperature, and thus has an inverse relationship to viscosity: the higher the MFR, the lower the viscosity. Typical values for various Hytrel® grades are given in **Table 2**. However, moisture content may significantly affect the result so it is essential that the regrind is dry before testing.

The difference in MFR between granules and finished product or regrind is an indication of polymer degradation. As a general rule, scrap material should not be re-used if the melt index check shows a value which is more than about 30% above that for the virgin material. In the case of properly processed Hytrel®, the increase in melt flow rate obtained from regrind should be well below this value.

Further information on melt flow rate measurements can be obtained from your DuPont representative.

## Extrusion Processes

### General

Many different extrusion processes have been successfully used with Hytrel®, and most of these are covered in the following section.

The melting characteristics and temperature guidelines for extrusion of various Hytrel® grades is given in **Table 2** below, however there are also some more general points which apply to all Hytrel® extrusion processes.

As discussed under “Melt properties,” the viscosity of dry Hytrel® extrudate depends very much on the melt temperature. It follows that melt temperatures (that is, actual temperature of the molten polymer) should be just above the nominal melting point to give the highest viscosity in processing. This is normally desirable in most (though not all) extrusion processes. Typically, the actual melt temperature should be 10–15°C above the nominal melting point when measured with a needle pyrometer held in the melt flow, and the extruder running at normal operating speed.

The temperature profile used to achieve this will depend on the individual extruder, and to a large extent on its screw design.

However, it is important that temperatures in the clamp, adaptor, head and die zones, as well as the melt pump (if used) should at all times be set to a value which is at least equal to the nominal melting point of the grade of Hytrel® being processed, otherwise cold slugs and skins of unmelted material may accumulate on the internal metal surfaces, and result in the release of lumps of cold or degraded Hytrel® into the melt flow.

In fact, when starting up a machine which was previously shut down with residual Hytrel® in the head, these temperatures (and also the barrel zones) should be set at least 10°C higher than the melting point (see section 5 “Start-up Procedures”) and only changed to normal running temperatures after purging out all old material from the machine.

Further adjustments of the melt temperature should normally then be made by alterations to the barrel temperatures only. In other words, the desired melt temperature should be achieved at the end of the screw, and the head and die zone settings should not be used to adjust melt temperature!

Because of the viscosity/temperature relationship, it is important that good control of temperature in each zone is maintained. This depends not only on having properly maintained and calibrated instruments, but also on having correctly positioned and working thermocouples and on other good operating practices.

**Table 2**  
**Hytrel® Extrusion Grades – Processing Conditions**

Hytrel® grade	Nominal melting point <sup>1</sup> , °C	Melt flow, index <sup>2</sup> , g/10 min	Typical temperature settings, °C				Adaptor, head and die	Typical melt temperature, °C
			Rear barrel	Center rear barrel	Center front barrel	Front barrel		
4056	150	6 (190°C)	150–160	160–170	160–175	160–175	160–180	165–185
G3548L	155	10 (190°C)	150–160	160–170	160–175	160–175	165–180	170–190
G4078W	170	5 (190°C)	160–170	170–180	170–190	170–190	175–195	185–200
G4074	170	5 (190°C)	160–170	170–180	170–190	170–190	175–195	185–200
4068, 4069	193	9 (220°C)	190–200	200–210	200–215	200–215	205–220	205–230
4556	193	9 (220°C)	190–205	200–210	200–215	200–215	205–220	210–230
G4774	208	11 (230°C)	195–210	205–215	205–225	205–225	210–230	220–240
5556, 5586	203	8 (220°C)	195–210	205–215	205–220	205–220	210–225	215–235
5555HS	203	8 (220°C)	195–210	205–215	205–220	205–220	210–225	215–235
G5544	215	10 (230°C)	200–215	210–220	210–225	210–225	215–230	230–245
6356, 6358, HTR8241, HTR8163	211 211 210	9 (230°C) 4 (230°C) 5 (230°C)	200–215	210–220	210–225	210–225	215–230	225–240
7246, 7248,	218	13 (240°C)	205–220	215–225	215–225	220–235	220–235	230–245
8238	221	12 (240°C)	210–225	220–230	220–230	220–230	225–240	235–250
HTR8303	221	6 (250°C) <sup>3</sup>	215–230	220–230	230–240	240–255	245–250	230–270

<sup>1</sup> Differential Scanning Calorimeter (DSC), peak of endotherm

<sup>2</sup> Nominal value — Test conditions: 2.16 kg load, temperature shown in parenthesis ( )

<sup>3</sup> Nominal value — Test conditions: 10.0 kg load, temperature shown in parenthesis ( )

Attention to detail in other parts of the extrusion process can also help prevent potential problems. Such factors as fluctuating quenching water temperatures and water turbulence, variations in haul-off speed, vibrations in mechanical equipment, and fluctuations in resin temperature in the hopper have all been known to cause problems which may be wrongly attributed to the material or the extruder.

## Profile extrusion

Solid and hollow profiles may be extruded successfully from many Hytrel® grades, depending on the complexity and shape required.

### By free extrusion

Simple shapes, such as solid round or flat strip profiles, are best produced by horizontal free extrusion into a water bath, over a suitable weir. This technique is relatively inexpensive in tooling, and in many cases a simple flat plate die (in aluminium, for example) can be simply fixed to the front of the extruder head, by means of a suitable adaptor if necessary. Although these plate dies can have the disadvantage of encouraging a build-up of stagnant polymer at the rear of the plate, which will eventually degrade and cause problems during long extrusion runs, they are nevertheless useful for prototyping and for shorter production runs (provided they are dismantled and cleaned after each run). Plate dies should have a thickness of 6–12 mm, depending on the profile size, and should be reduced locally to 3–5 mm where the profile may have a section which is relatively thin. The actual dimensions of the die orifice should be roughly 2–3 times the dimensions of the finished profile, in order to provide enough draw-down of the melt during extrusion. This draw-down is necessary to give sufficient tension in the extrudate to prevent sagging and fluctuations in dimensions.

Normally, some modifications to the die orifice are necessary after initial trials, in order to achieve the required distribution of material. With a simple plate die in aluminium, it is relatively easy to hand file or grind the die, for example to encourage material flow to selected parts of the profile cross section.

These techniques are particularly successful with the softer Hytrel® grades, up to, say, 55D in hardness, and with the higher viscosity grades, extruded at lowest possible melt temperature (within the limitations mentioned under “General” on page 10). However, it should be noted that the final profile shape achieved may be affected by changing grades, or by altering extrusion speeds or temperature settings.

Of course more streamlined dies, made from hardened steel, are preferable for longer runs, but the simple plate die can often be used as a basis for the design of the final die, or for prototype parts where the cost of a full production die may not be justified.

While free extrusion is acceptable for simple “solid” shapes, it may not be possible to achieve the desired profiles with this technique where complex shapes or large changes in section are involved (for example, “U” channels or ribbed profiles). In such cases, it may be desirable to arrange suitable guides or supports within the water bath which “hold” the shape of the extruded profile until sufficiently solidified. However, this technique may still not be adequate, particularly with the harder Hytrel® grades where differential shrinkage can cause warping and distortion of certain profile shapes. Vacuum calibration may therefore be required, but — as discussed below — there are limitations with vacuum calibration of profiles, particularly regarding the grades of Hytrel® which are suitable, and the complexity of the shape to be extruded.

### Hose mandrels

Hose mandrels are a specific case of a simple, solid round profile. Such mandrels in Hytrel® are widely used in the manufacturing process for rubber hoses.

The control of diameter and ovality, is particularly important and for this reason the following points have been found to be helpful:

- Melt temperature should be as close as possible to the nominal melting point of the Hytrel® grade in use.
- Use of a melt pump will help ensure constant extruder output, which is important in maintaining dimensions to tight tolerances.
- The profile should enter the water bath smoothly, with no turbulence in the water flow at the point of entry.
- The profile should be supported underwater within the cooling bath by means of a “V” or “U” guide channel.
- The use of a short initial cooling bath will ensure minimum stretching of the profile between extruder die and haul-off (“elastic band” effect). Additional cooling is provided after the first haul-off, before reaching a second haul-off or capstan unit.
- The first haul-off should only apply light pressure in order to avoid deformation of the shape while still warm.
- Harder Hytrel® grades (55D and above) tend to form internal shrinkage voids in larger mandrel diameters (typically >12 mm), but this may be improved or eliminated by using hot water quenching in the first cooling bath (e.g., water at 70°C), or by alternating short sections of water and air cooling.

### Profiles by vacuum calibration (sizing)

This technique, which is well established for profiles in more rigid polymers such as rigid PVC, polycarbonate, etc. has been found to be moderately successful with the harder Hytrel® grades. Unfortunately, the softer types exhibit too much friction against the calibration die surfaces and tend to stick.

Normally, this process can only be used for relatively simple “hollow” profiles, where there is an internal hole or cavity which can be held at ambient pressure during the extrusion process via an airway in the extrusion mandrel (die core).

In this process, a “wet” calibration die system should be used, where the extrudate is lubricated by a film of water supplied through a series of small (0.5–1.0 mm) holes drilled close together around the entrance to the calibration die. Alternatively, a small slot can be used to provide the required film of water at this point. The die entrance should be radiused (3–4 mm) and the whole internal surface finished by sand blasting, in order to give a “matte” surface which helps retain the water lubrication film.

Extruder die design should follow the principles above for free extrusion, although less draw-down can be used (depending on the profile size and shape). The die land length should be relatively short (typically 5–10 mm, depending on the profile thickness) in order to prevent the extrudate being drawn away from the die wall at the die exit point.

## Monofilaments

Monofilament extrusion is straightforward with the medium viscosity Hytrel® extrusion grades.

Equipment normally used with Nylon, PBT, and similar resins may also be used for Hytrel®, and for this reason it is not considered necessary to describe such equipment in detail here. Processing temperatures may be somewhat higher for monofilament extrusion than for most other extrusion processes, with typical melt temperatures being 20–30°C above the nominal melting point of the particular Hytrel® grade in use.

For maximum process stability, the draw-down ratio, as measured by the die diameter relative to the monofilament diameter, should be between 4:1 and 10:1 as the filament leaves the first water quench bath.

The amount of filament orientation, and the temperatures for further drawing and annealing should be determined by trials which involve measurements of the required end-use properties for the monofilament.

**Note:** In both profile and monofilament extrusion, the use of a melt pump can greatly assist dimensional control of the finished profile.

## Rodstock

The production of rodstock and other stock shapes in Hytrel® is achieved by a special extrusion process, which is also used for other polymers. Since the cross-sectional areas of these products are relatively large, this limits the rate of cooling of the section, and extrusion speeds are consequently very slow in this process. It is essential to ensure that the shrinkage during solidification of the material in the center of the shape is being

continuously compensated by an additional flow of material under pressure from the extruder. Furthermore, the slow movement of the Hytrel® shape through the cooling sections of the die must be smoothly controlled, and a constant back pressure on the melt must also be maintained by control of the take-off unit.

Since the equipment for stock shape production tends to be rather specialized, it will not be described further in this manual.

## Melt casting

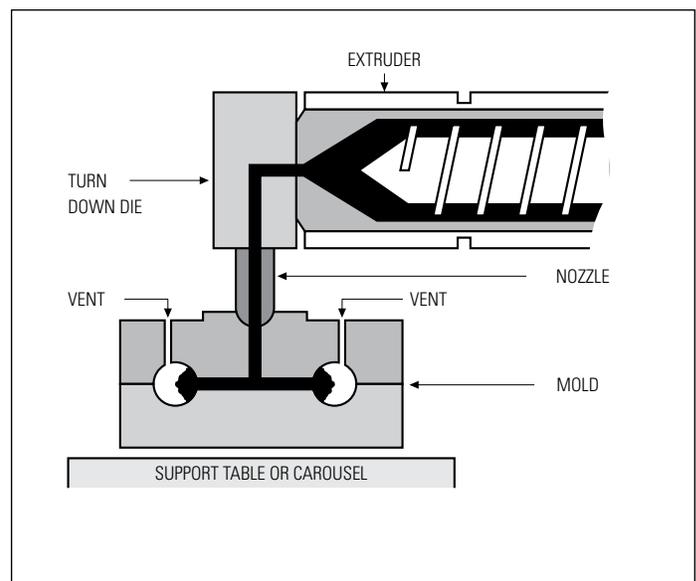
The melt casting process may be used with Hytrel® to produce solid molded shapes using a simple low-pressure mold which is filled from a suitable nozzle fitted to the extruder head.

Although not many resins may be processed by this technique, it has been found that the combination of good thermal stability, relatively low shrinkage, and suitable viscosity characteristics make Hytrel® ideally suited to this process.

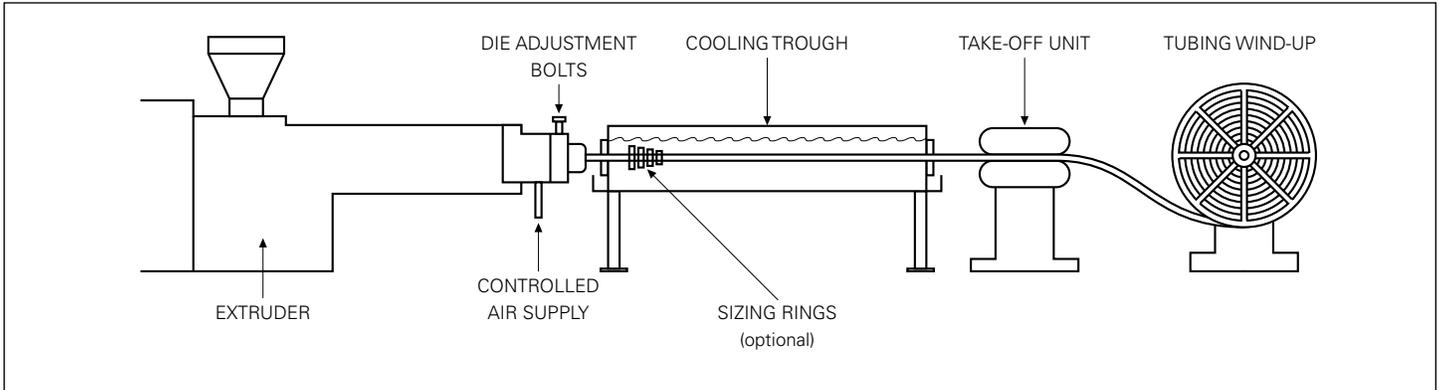
The melt casting technique may be used (a) where large, thick parts would not be economically viable by injection molding due to the excessive cycle times, or (b) where injection mold tooling cost would be too high because of small numbers of parts required — melt cast tooling is relatively inexpensive, or (c) for prototyping parts which might subsequently be made by injection molding.

A typical equipment set-up is shown in **Figure 6**. Note that the mold may be one of several which are filled in turn on a carousel system, while others are undergoing the cooling or demolding stages. Although shown with a vertical feed from the extruder, it is also possible to utilise a horizontal feed channel from the extruder to the mold cavity.

**Figure 6. Use of extruder for melt casting of Hytrel®**



**Figure 7. Free extrusion of tubing**



### Free extrusion of tubing

Small size tubing can be prepared from Hytrel® by free extrusion. This method is generally only successful with softer Hytrel® grades and for tubing up to 6 mm diameter. Larger sizes in grades of 45D hardness or above can be made by the differential pressure sizing method (also called vacuum calibration or vacuum sizing) — see next section.

A typical set-up for free extrusion is shown in **Figure 7**. The process consists of extruding a tube of resin and pulling it through a trough of cold water. A relatively high draw-down ratio should be used (typically, die diameter should be 2–3 times tube diameter) for good process stability. One or more “V” or “U” shape guides may be installed under the water surface, to assist in maintaining tube shape.

The process is inexpensive, and a variety of sizes can be made from one die and mandrel combination by simply varying the screw speed, take-off rate, and internal air pressure (if used).

In order to prevent collapse of the tube during free extrusion, it is sometimes necessary to supply air under very low pressure, through the mandrel (core pin) to act as internal support. This pressure must be very accurately controlled, and for this reason a very sensitive control valve or pressure regulator must be used.

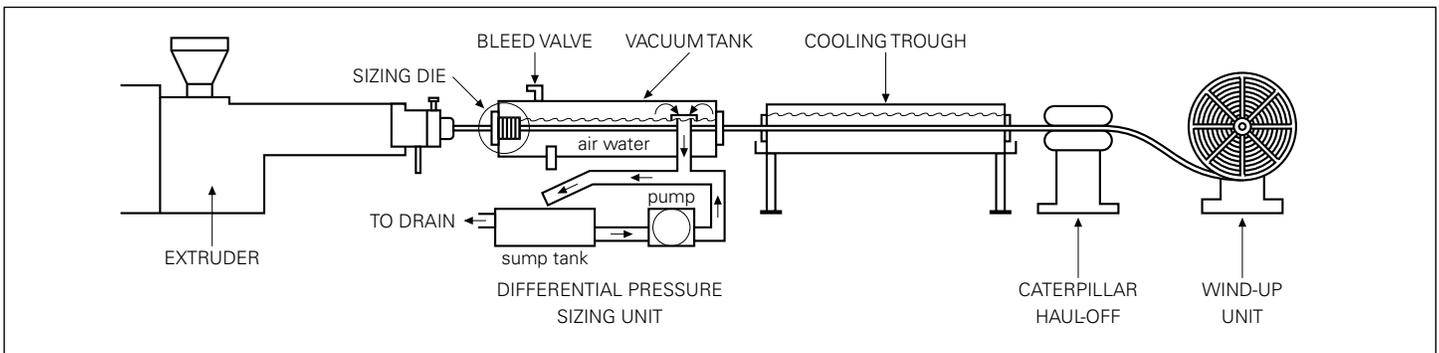
### Vacuum calibration (sizing) of tubing

The free extrusion method is not generally used for medium and hard Hytrel® grades, and particularly in sizes of 6 mm diameter and above because roundness is difficult to control. The standard procedure for sizing larger tubing is the differential pressure or vacuum sizing technique. This process works well with Hytrel® grades of 45D hardness and above.

A typical set-up is shown in **Figure 8**. This method requires no internal gas pressure to support the tube, permitting the tubing to be cut to any length without disrupting the process.

One method of calibration is by means of a stack of aluminium or brass plates each containing a precisely drilled hole, but the preferred system is a tubular sleeve sizing die generally fabricated from brass with peripheral holes or slots in the sleeve wall to allow the surrounding vacuum to act on the extruded tube. The inside surface should be sandblasted and may also contain shallow grooves or “rifling” to decrease the surface drag of the extruded tube. Whether the plate die or tubular sleeve die is used, the bore should be between 3% and 15% oversize to compensate for shrinkage of the extrudate. The correct dimension depends on several factors, the most important being tube diameter and line speed. **Figure 9** shows how to calculate correct sizing die diameter for a particular tube size and line speed, and may be used for Hytrel® grades of 55D–72D hardness.

**Figure 8. Tubing extrusion with differential pressure sizing**



The fine adjustment to tube dimension is achieved, however, by adjustment to the vacuum applied to the vacuum bath.

Suitable lubrication must be provided between the extruded polymer and the metal surface of the calibrator. Usually, this can be accomplished by a fine water flow through small holes or slits at the front of the tubular die, or through an annular water ring device at the entrance to the die.

The correct design of vacuum sizing die is very important, and designs used for other polymers (nylon, polyethylene, etc) may not necessarily be suitable for Hytrel®.

A sizing die which has been found to work well with all Hytrel® grades over 40D hardness is shown in **Figure 10**. Please contact your DuPont representative for more detailed design information.

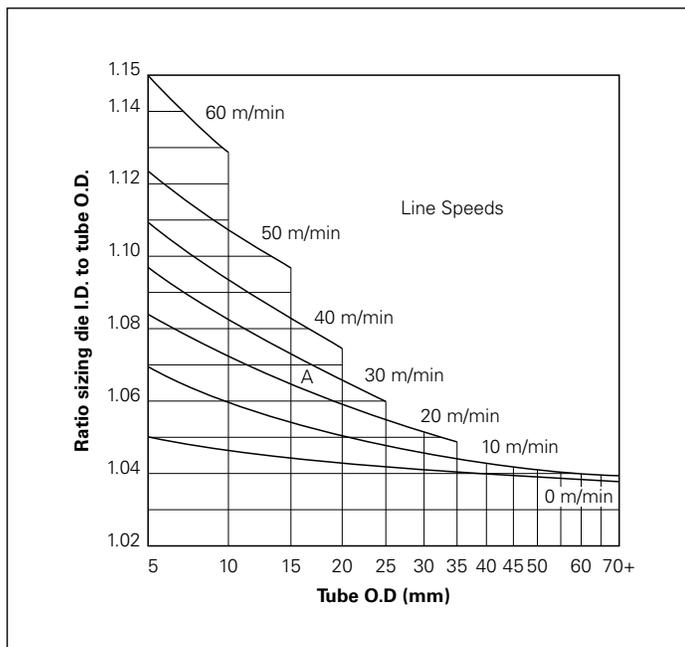
In all vacuum sizing operations there should be provision for fine adjustment of the vacuum, in order to accurately control and maintain the external diameter of the extruded tube. Vacuum of 0.10 to 0.30 bar has been used successfully to maintain accurate dimensional control of small diameter Hytrel® tubes.

In general, 35D and 40D Hytrel® grades cannot be run successfully by the differential pressure sizing method using either plate die or the tubular die. Their very rubbery nature and slower crystallization rate causes the polymer to bind or "grab" in the sizing die. In certain cases, however, it has been possible to extrude large diameter, thin-walled tubes in these grades, using a calibration die under very low vacuum. Such tubes are used as liners for fire hoses and other lay-flat hoses.

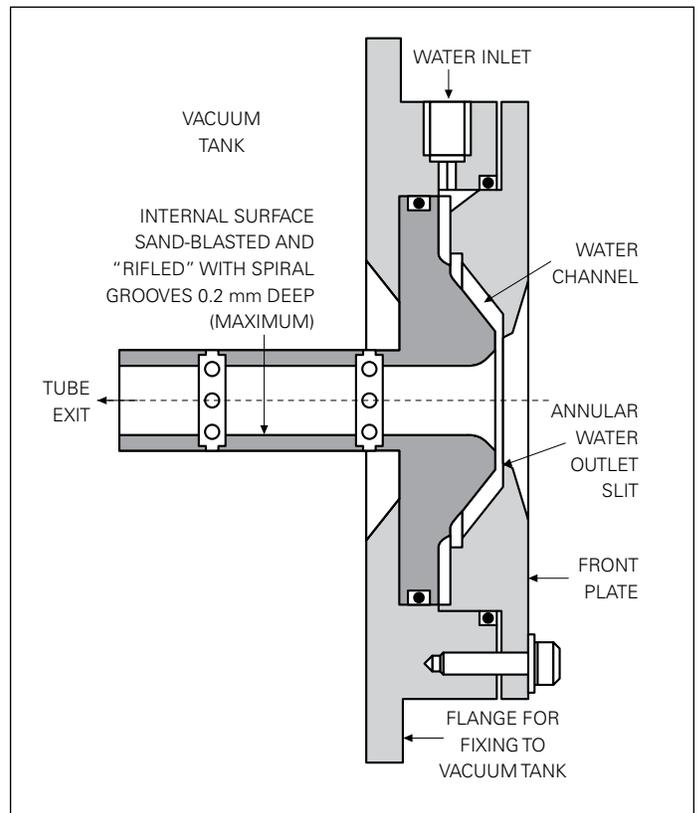
The correct extruder die and pin for tube extrusion should be selected as follows:

1. The extruder die should be between 2 and 3 times the required tube outside diameter, for tubes up to about 20 mm diameter. Where larger diameters are to be made, the extruder die should be between 1.5 and 2 times the tube outside diameter.
2. The extruder pin should then be selected so that the pin should be larger than the required inside diameter by a factor which is slightly less than that which was used to determine die diameter in (1) above.

**Figure 9. Selection of vacuum die dimensions**



**Figure 10. Tubular sizing die**



For example, to extrude a tube having 8 mm outside diameter and 6 mm inside diameter, at a line speed of 30 m/minute:

Extruder die diameter =  $8 \times 2.5 = 20$  mm

Therefore extruder pin =  $6 \times 2.3 = 13.8$  mm

Selection of pin and die according to these guidelines will result in a Draw-Down Ratio in the range of 4 to 9, which has been found to be optimum for Hytrel® tubing extrusion using the vacuum sizing technique. Draw-down ratio (D.D.R., see formula below) is defined here as the ratio of the cross-sectional area of the extrudate at the extrusion die-face, to the cross sectional area of the finished tube.

$$\text{D.D.R.} = \frac{Dd^2 - Dm^2}{Dt^2 - Db^2}$$

Another important factor when choosing dies and mandrels for making tubes from Hytrel® is the Draw-Ratio Balance (D.R.B., see formula below). The ideal D.R.B. for Hytrel® is 1, meaning that the inside surface of the molten tube is drawn the same amount as the outside surface. The range 0.9 to 1.1 is acceptable.

The draw-down ratio and the draw-ratio balance can be calculated as follows:

$$\text{D.R.B.} = \frac{Dd \div Dt}{Dm \div Db}$$

Where Dd = Diameter of die

Dm = Diameter of mandrel or torpedo

Dt = Diameter of tube

Db = Internal tube or bore diameter

Other factors which are worth special attention in vacuum calibration of tubes in Hytrel® include:

- Optimum screw design to give constant output rate and minimum variation in melt temperature. Please refer to section on Screw Design on page 4. This is particularly important for high speed tubing extrusion, and where accurate dimensions must be maintained over long lengths. A special barrier screw design has been developed which has been found to give excellent results with Hytrel® in tubing extrusion and other critical processes. Details can be provided through your DuPont representative.
- Use of a melt pump can also help to ensure constant output and hence accurate dimensional control.
- Lubricating water supply at uniform pressure to the sizing die. Ideally this should be from a header tank situated at least one meter above the die, with flow being controlled and monitored through a suitable flow meter.
- Non-turbulent water flow in the cooling bath — water inlet pipes should not be directed so as to cause turbulence, air bubbles or waves near the calibration die.

- Constant water temperature (max. 5°C variation) in the vacuum bath.
- Uniform temperature and moisture content of granules in the feed hopper. This is best achieved by the use of a hopper drier system or a drier with automatic transfer of granules at constant temperature/moisture level to a sealed hopper.

## Cover extrusion “cross-heading”

Most Hytrel® grades have been used to cover various types of product, including hoses, ropes, cables and wires. The basic equipment required includes an extruder fitted with a suitably designed cross-head, a pay-off (or unwinding) system, incorporating a brake or tension control device, a water cooling bath, and a variable speed haul-off or capstan, followed by an automatic wind-up or coiling system.

The cross-head and die arrangement can be one of two types:

- Cross-head with pressure (packing) die, or
- Cross-head with tubing (sleeving) die.

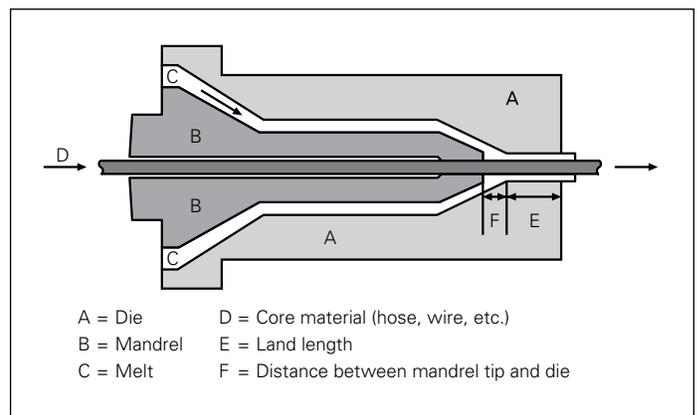
*Pressure Die* extrusion involves the extruded material coming in contact with the core (e.g.: hose, wire etc) within the die itself, which results in some pressure being applied to the melt so that it tends to penetrate any interstices in the core material. This technique is preferred where:

- Good adhesion is required, or
- A smooth, regular, outer cover diameter is required over an irregular or rough inner core material.

**Figure 11** shows a typical pressure die arrangement.

The diameter of the die should be approximately equal to the required cover diameter. The land length (E) should be between 1 and 3 times the final cover diameter, but considerably less for very thin covers (where thickness is <0.5 mm).

**Figure 11. Extruder head for pressure die covering**



The clearance between the core to be covered and the bore of the “torpedo” or mandrel should be between 1% and 5% of the core diameter (depending on the material and regularity of the core surface).

The distance “F” between mandrel tip and die entrance should be adjustable, but is normally set to be equal or greater than the cover thickness being applied.

**Tubing Die** (sleeving) extrusion is illustrated in **Figure 12**.

In this technique, Hytrel® is extruded in the form of a tube, and is “drawn down” to meet the surface of the core material. This is often achieved with the assistance of a vacuum which is applied to the inside of the torpedo and acts through the bore of the mandrel (pin). The optimum draw-down ratio (D.D.R.) is in the range of 5–20:1 and the die diameter and mandrel diameter can be calculated from the selected draw-down ratio as follows:

$$\text{D.D.R.} = \frac{D_d^2 \div D_m^2}{D_c^2 \div D_w^2}$$

Where Dd = Die diameter  
 Dm = Mandrel diameter  
 Dc = Diameter of covered core (rope, cable, etc.)  
 Dw = Diameter of uncovered core

The advantages of tubing die extrusion are:

- Better control of cover wall thickness
- Easier to “strip” cover from core (eg: wire coating applications).

Some other points which may be important in cross head extrusion with Hytrel®:

- If a thermally conductive core material is being covered (e.g., electrical conductor, steel braid hose, etc), it may be necessary to pre-heat the core by passing through a flame or hot-air tunnel before entering the cross-head. This will help to prevent too rapid freeze-off of the melt when it comes in

contact with the metal core, which may result in insufficient penetration of the core material. Similarly, fibrous cores (e.g., textile rope or braiding) may need to be dried by passing through a hot-air tunnel, or by storing the core material in a warm, dry area prior to covering, in order to prevent moisture blisters appearing through the Hytrel® cover.

- For high speed extrusion, particularly with thin wall covering, it may be necessary to raise the head and die temperatures, and possibly also the barrel temperatures, to achieve sufficient flow rate at acceptable melt pressure. Melt temperatures up to 40°C above the nominal melting point of most Hytrel® grades can be safely used to reduce melt viscosity, providing the design of adaptor, head, and associated parts does not give rise to any hold-up spots where thermal degradation might take place.
- Although water temperature in the cooling bath is generally not critical (10–20°C is typically used), there may be cases where a more gradual cooling of the Hytrel® cover by using hot water (e.g., 60°C) may be beneficial. This is sometimes the case with optical fibre sheathing, or small diameter electrical conductors, where too fast crystallization caused by cold water may result in undesirable stresses in Hytrel® when it cools.

## Cast film, sheeting and fabric coating

### Cast film

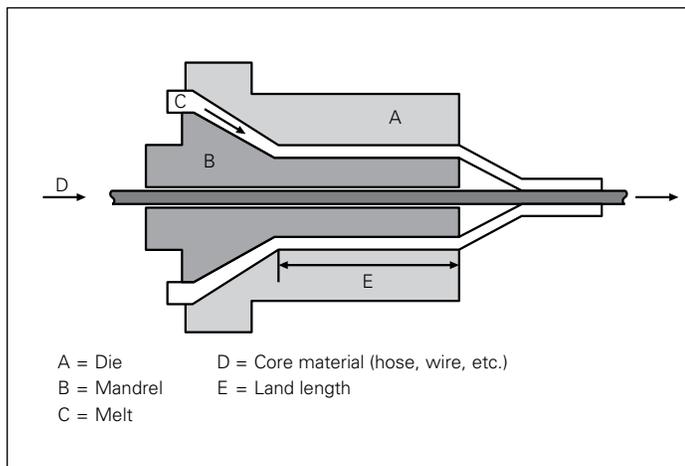
In the cast film process, molten Hytrel® is extruded through a slit die onto a polished metal roll — usually known as the “quench roll” or “chill roll” — which serves to quench the hot melt. From the quench roll, the film passes around a series of other rolls designed to guide it and keep it wrinkle-free at wind-up. **Figure 13** illustrates a typical cast film line.

Films of Hytrel® thermoplastic polyester elastomer as thin as 0.013 mm have been made by this method.

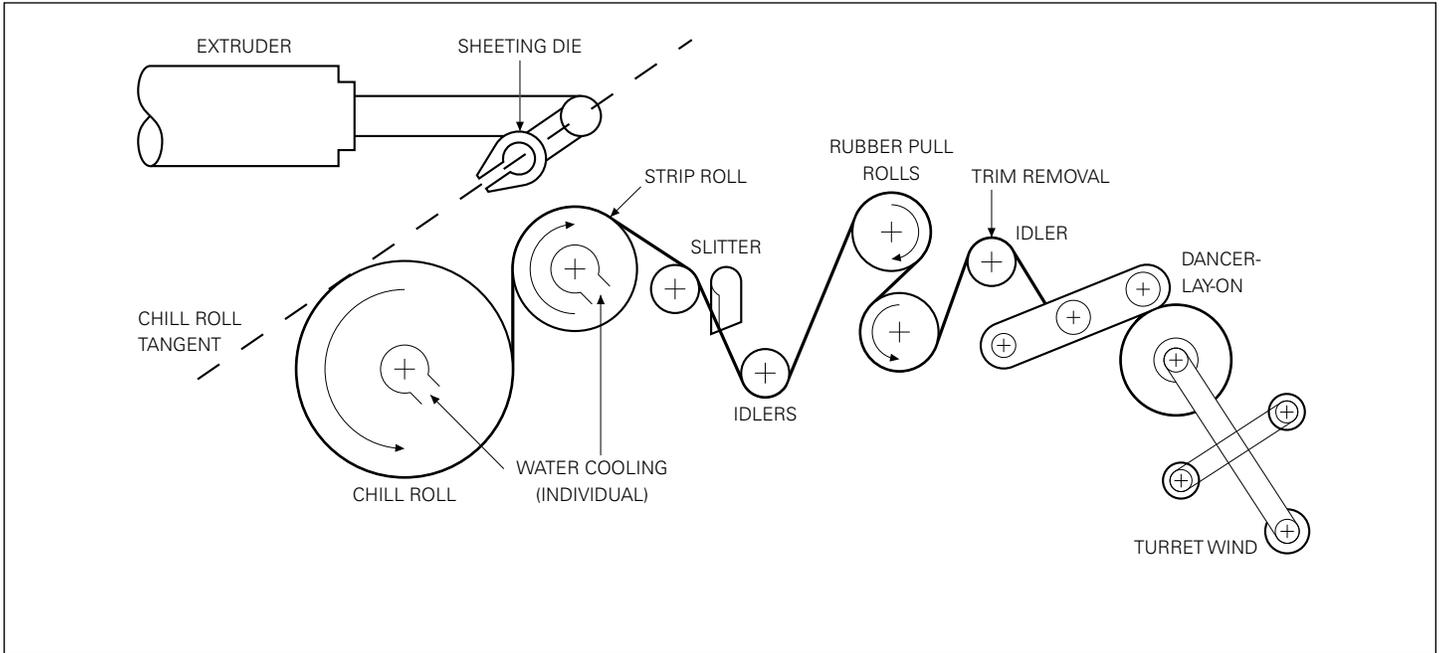
Film thickness is controlled by the relationship between the extruder output and surface speed of the quench roll.

The quench roll is normally cooled internally by water. With Hytrel®, hot water or hot oil may be used to control quench temperature for some applications (see discussion on effect of quench temperature on film properties, below). Tension control must be precise, and should be very light with the more flexible grades of Hytrel® in order to produce wrinkle-free film with good roll conformation (flatness across the roll). Air jets can be used to pin the edges of the melt web to the chill roll, in order to minimise edge weaving and reduce neck-in. Take care that none of the air flow reaches the die lips, since this could result in uneven cooling of the die and poor dimensional uniformity in the film.

**Figure 12. Extruder head for tubing die covering**



**Figure 13. Typical cast film unit setup**



The processing conditions of melt temperature, quench temperature, air gap and extrusion rate all influence film properties, but quench temperature and air gap have the greatest effect. The following general statements indicate how each of these conditions affects film properties; the degree to which the property is affected depends on the grade of Hytrel® used:

*Increasing melt temperature:*

- Increases transparency
- Increases gloss
- Decreases haze
- Decreases modulus and tensile strength

*Increasing quench temperature:*

- Increases modulus and tensile strength
- Increases haze
- Decreases gloss
- Decreases transparency

*Increasing air gap:*

- Increases haze
- Decreases gloss and transparency
- Increases modulus and tensile strength

*Increasing extrusion rate:*

- Decreases haze
- Increases transparency and gloss

To avoid sticking, quench temperature should not exceed 50°C for soft Hytrel® types (35D and 40D hardness). 80°C for 47D to 63D hardness grades, or 100°C for 72D and harder grades.

## Sheeting

The term “sheeting” normally describes material that is 0.25 mm or greater in thickness. Sheetting of Hytrel® up to about 0.5 mm thick can be produced on the same equipment used to make cast film, depending on the die dimensions and the ability to achieve a slight, controlled sticking to the quench roll.

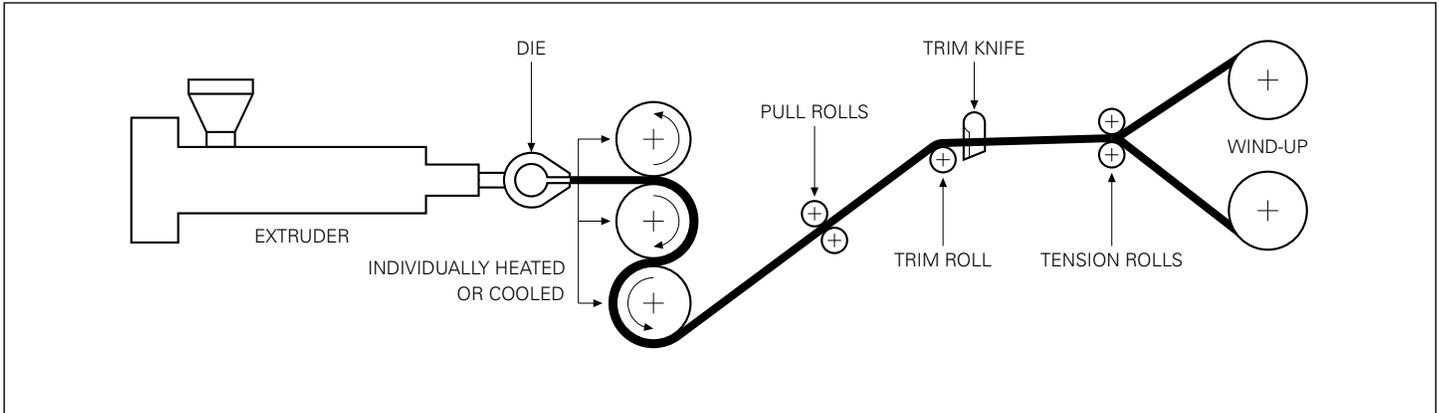
Sheetting of greater thickness is made on a three-roll finisher, as shown in **Figure 14**.

As in other extrusion processes, die design and temperature control are critical.

Both mechanical and air pressure systems for applying roll force have been used. The sheeting thickness is controlled by adjusting the “nip” between first and second rolls, while controlling extruder output in order to maintain a small “rolling bank” of molten melt in the nip.

The die lip opening should be 20 to 50% greater than the sheet to be produced; a thinner sheet (within limits) can be made with the same die opening by increasing roll speed. Melt temperature should be kept as low as possible consistent with uniform extruder output, melt quality, and head pressures. The air gap should be the minimum permitted by equipment geometry. The melt “puddle” or bank of surplus resin between the roll “nips” should also be kept small to minimise oxidative degradation, but if the bank is too small, it may result in “starving” of the rolls which will cause erratic dimensional variations in the sheet.

**Figure 14. Extrusion of sheeting**



Roll temperatures should be individually controlled. Typical roll temperatures for Hytrel® sheeting extrusion are as follows:

Type of Hytrel® (Shore D hardness)	Roll temperature, °C
35D and 40D	15 to 30
47D to 82D	40 to 70

The temperature of the first roll will be limited to the temperature at which the sheet sticks to the roll, and is normally kept as low as possible.

### Fabric coating

Fabric coating with Hytrel® can be achieved on a 3 or 4 roll coating line which is similar in principle to the sheeting extrusion line shown in **Figure 14**. The fabric is introduced above the extruded Hytrel®, between the first and second rolls. A more common set-up is the standard extrusion coating line shown in **Figure 15**. In this process the melt is extruded through a slit die onto the fabric or other substrate in the nip between a chill roll and pressure roll.

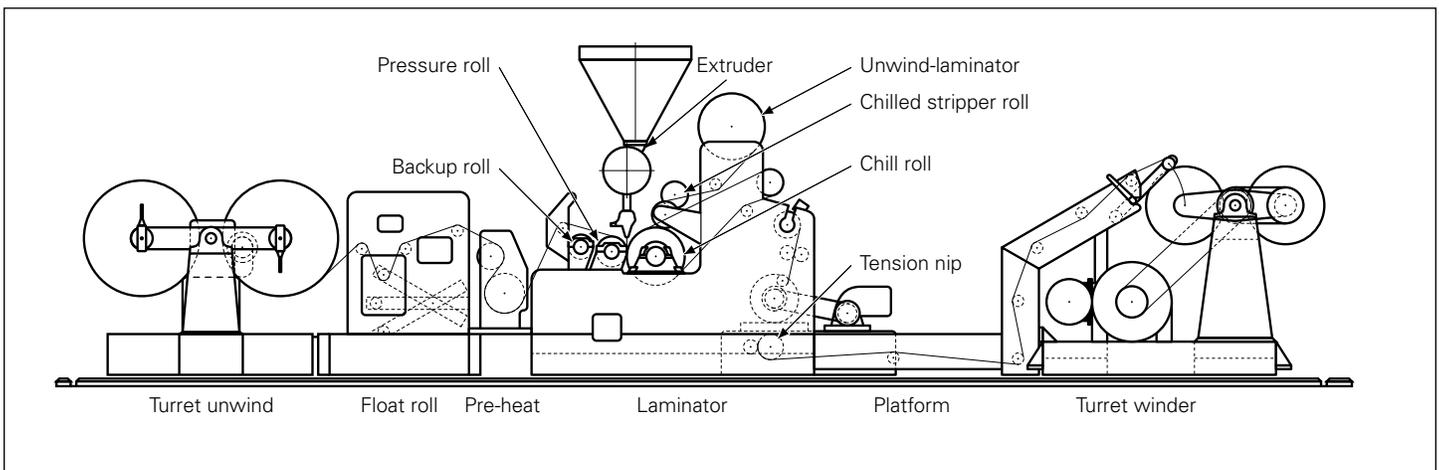
The melt contacts the substrate just prior to meeting the chill roll which solidifies or quenches the melt. The melt web usually extends beyond the edges of the substrate slightly, and therefore contacts both the chill roll and pressure roll at this point. To avoid any tendency to stick on the pressure roll, it has proved effective to cover rolls with a coating of Teflon® FEP fluorocarbon resin.

A chill roll of 300–600 mm in diameter is usually used in extrusion coating. The temperature of the chill roll is normally maintained between 20 and 40°C.

The stability of the melt web before it contacts the chill roll depends greatly on having the correct die gap set on the extruder, as well as on the melt temperature, the air gap between die and roll nip, and on the line speed.

As for cast film extrusion, careful control of tension in the windup is very important to minimise wrinkling and ensure good roll conformation.

**Figure 15. Typical extrusion coating line. Substrate is fed from the turret unwind at left. After preheating, it is coated by extruder at laminator station (center). After cooling, stock is wound at right.**

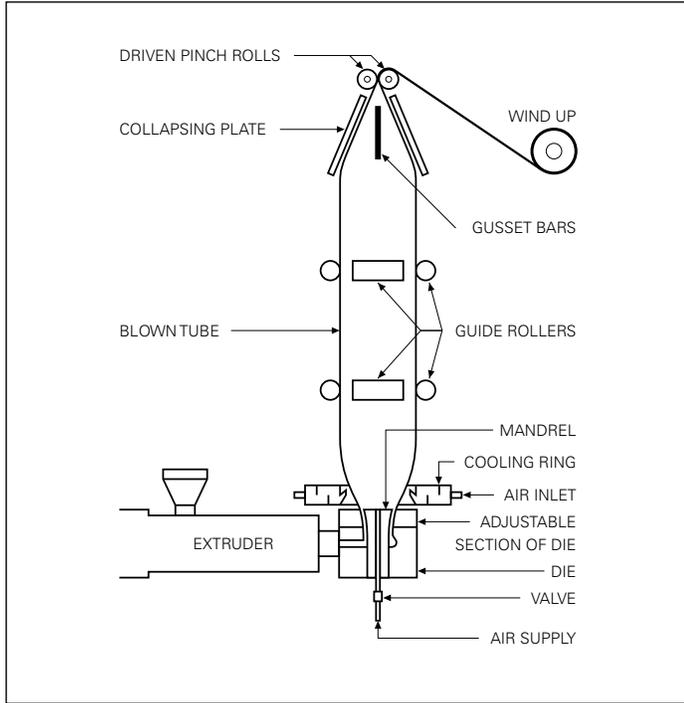


## Blown film

Suitable Hytrel® grades may be processed with standard film blowing equipment such as that shown in **Figure 16**. The higher melt strength of Hytrel® blow molding grades make them easier to handle, and allow films up to 250 microns to be produced with blow-up ratios of up to 3:1. Other Hytrel® grades,

particularly the softer 35D and 40D hardness types, can also be blown into films up to about 150 microns thick, at blow up ratios up to approximately 2.8:1, but anti-blocking agents such as Kemamide B\* or Crodamide SR\*\* (or similar products) may be required to prevent sticking of the film to itself and to the rolls.

**Figure 16. Extrusion of blown film**



## Lay-flat hose liner extrusion

Thin wall Hytrel® hose liners are used for lining fire hoses and other lay-flat hoses. Typical diameters range from 20 to 300 mm, and thickness from 0.2 to 0.7 mm. These thin-wall liners can be made by either a horizontal vacuum sizing technique, or by a vertical extrusion system. Normally, the softer Hytrel® types are used, as well as the blow molding grades, which have been found particularly suitable because of their high melt strength properties.

General extruder design for both processes is according to the recommendations discussed elsewhere in this manual, and temperature settings for particular Hytrel® grades are typical for tubing extrusion.

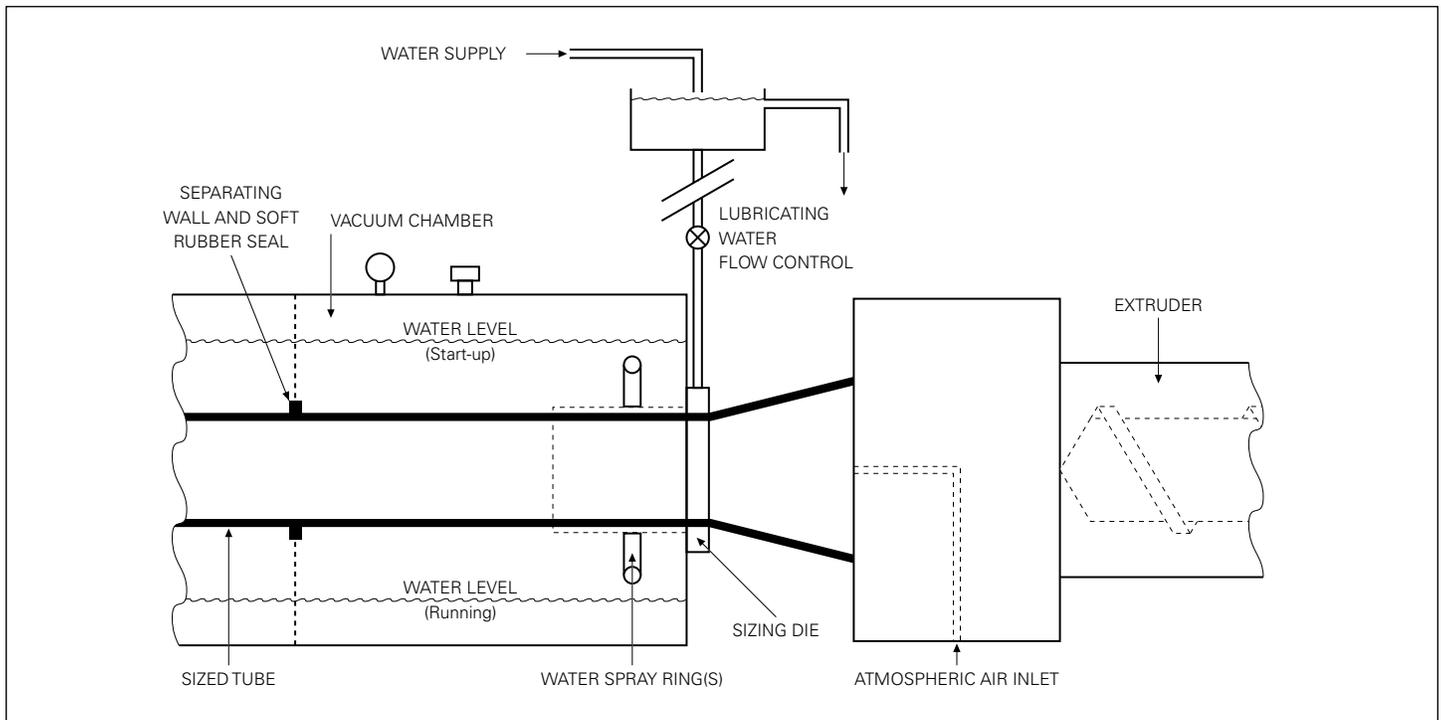
In the horizontal extrusion process, a large diameter tube of melt is extruded, which is then drawn down into a calibrating die located in a vacuum tank, and then cooled.

Accurate sizing is achieved by the differential pressure of the atmospheric air inside the tube and the vacuum applied to the outer surface. The general principles are shown in **Figure 17**.

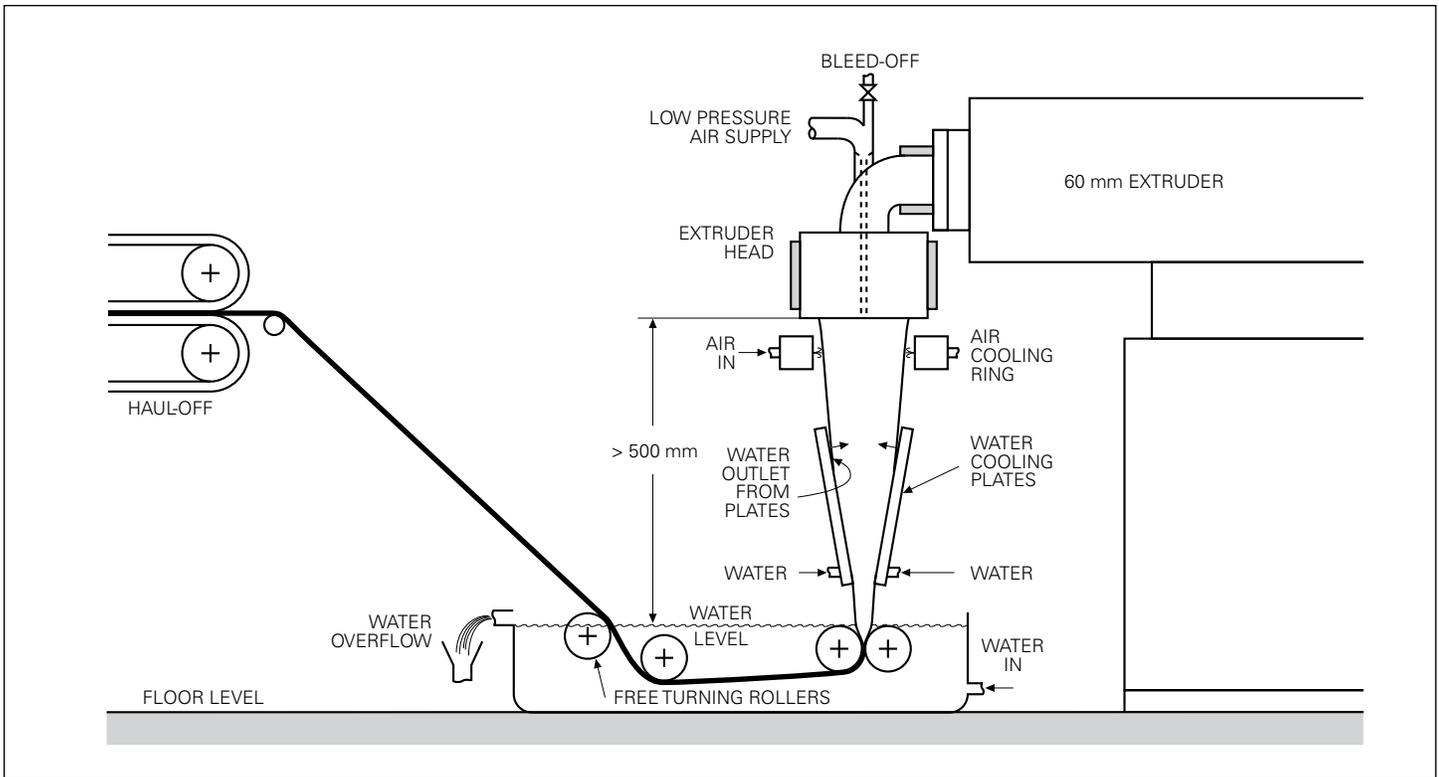
\* Product of Witco/Crompton Corp., USA

\*\* Product of Croda International Plc., England

**Figure 17. Horizontal extrusion of thin wall hose liners**



**Figure 18. Equipment for vertical extrusion**



In practice, it has been found that due to the combination of the large diameter and thin wall section, flotation occurs after the extrudate leaves the sizing die. This can create a problem in maintaining a good vacuum seal.

To overcome this, a high water level is used at start-up and when a vacuum is achieved and the tube is inflated, water is then drained to below the level of the sizing die. Cooling is effected by water sprays which are directed onto the sizing die and tube surface within the front tank section. No further cooling through the tank is required. There are several factors which are critical in order to achieve a consistent product. Of prime importance are:

- a. Correct draw-down ratio (normally between 10 and 15:1). Extruder die diameter should be approximately 2.5 times tube diameter.
- b. Correct design of sizing die.
- c. Even flow and distribution of lubricating water at sizing die entry point.

- d. Good vacuum control (liquid manometer used).
- e. Constant cooling water temperature.

In the vertical extrusion process, an extruder head is used which turns the melt stream vertically downwards, and a tube is extruded and fed into a cooling tank located beneath the head. This method is shown diagrammatically in **Figure 18**.

The diameter and wall thickness of the liner are controlled by applying a low positive air pressure through the center of the mandrel (pin), and by adjustment of the extruder output relative to the take-up speed. Various size liners can be made using the same pin and die arrangement.

The positive air supply used is very low pressure (approximately 25 kPa). A low pressure, high volume air blower is preferred for this purpose, rather than a pressure reduced compressed air supply. Control of the internal pressure is then achieved by means of a bleed-off valve fitted at the connection between the low pressure air supply and the extruder head. A further improvement is the provision of a return air channel through

the mandrel which allows air to circulate within the extruded tube, thereby giving improved internal cooling as well as better pressure control. Best results are obtained by drawing down the extruded tube to a smaller size than the extruder die, rather than trying to increase its diameter with air pressure.

Further details of both processes for extrusion of fire hose and other thin wall hose liners can be obtained from your DuPont representative.

## Co-extrusion

Hytrel® may be co-extruded with several other resins for use in many applications such as hose and tubing, profiles, sheeting and film. In this technique, Hytrel® is brought together with the other resin (or resins) in a specially designed co-extrusion head where the different materials are fused to form distinct, well bonded layers in a single extruded product.

By using co-extrusion, the finished product can combine the advantages of two or more polymers in a cost effective way.

The successful processing of Hytrel® in co-extrusion depends on the following factors:

- Compatibility of the two resins in terms of fusion or “weldability.”
- Closeness of melting points, or normal processing temperatures.
- Similarity of flow properties within the die (viscosity and viscosity/shear rate relationship).
- Design of the co-extrusion die.

Experience has shown that Hytrel® is extremely compatible with most rigid and flexible PVC compounds, and equipment normally used to co-extrude rigid and flexible PVC can give good results where the flexible PVC is replaced with Hytrel®. The lower melting point grades of Hytrel® must be used, however, as it is normally necessary to limit the temperatures in the co-extrusion head to around 180°C in order to prevent degradation of the PVC.

Other polymers which have been successfully co-extruded with Hytrel® include Alcryn\*, TPU, ABS, polycarbonate, PBT and PET. Resins which are not normally considered to be compatible may still be co-extruded, provided that a suitable intermediate “tie layer” is used to provide a bond between the layers. DuPont supplies a number of these “tie layer” resins in the Bynel® range.

## Co-extruded profiles

One of the best applications for Hytrel® in coextrusion is as a flexible hinge between rigid profiles in PVC, ABS or PC. In this case, a grade such as 4056 may be co-extruded in such a way that the rigid materials are vacuum calibrated (in the normal way) but the Hytrel® section is simply water cooled and kept free from contact with the calibration die in order to prevent sticking.

## Co-extruded (multilayer) tubes and pipes

Hytrel® can be successfully incorporated as one or more layers in a multilayer tube or pipe. It may be combined with other grades of Hytrel®, or perhaps with Crastin® PBT or even Zytel® nylon (with a suitable tie layer) to provide a specific combination of properties in the finished product.

It may also be co-extruded as a thin layer in a PVC hose, for example to provide a barrier against fluid permeation into the PVC, or to prevent plasticizer extraction from the PVC. It could also add heat resistance to the overall tube or hose.

In tube or pipe co-extrusion, the head and die temperatures must take into account the melting point of each resin, and their acceptable “processing windows.” Also, the design of the flow channels must respect each resin’s viscosity characteristics — if their rheologies are not sufficiently close, it may be difficult to ensure even flow behavior at the interface between the polymers. As with co-extrusion of any material combinations, the design of the co-extrusion head is critical.

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\*Registered trademark of Advanced Polymer Alloys (APA)

## Troubleshooting Guide for Extrusion of Hytrel®

<b>Problem</b>	<b>Probable Causes</b>	<b>Suggested Solutions</b>
Blisters (on surface) when coating substrates	1. Substrate contains volatile substances (water, oil, etc.). (For example, when coating textile materials)	1. a. Pre-heat or dry substrate b. Clean substrate c. Increase extrusion rate d. Decrease air-gap before cooling stage e. Apply vacuum through mandrel
Bubbles	1. Resin degradation due to high temperatures and/or long hold-up time, perhaps after extruder stoppage  2. Air entrapment  3. Moisture in resin	1. a. Reduce temperatures b. Increase extrusion rate c. Check for stagnation (dead spots) in extruder or die d. Check operation of heaters, controllers, thermocouples e. Use smaller extruder to decrease hold-up time f. Use proper screw g. Allow extruder to run for several minutes after start-up 2. a. Increase rear barrel temperature b. Use proper screw (compression ratio too low, or other design fault) c. Increase back pressure on screw d. Check temperature controllers 3. If resin has become wet, dry it before extruding
Buckled extrudate	See: Out of roundness — folded or buckled extrudate	
Concentricity, lack of	See: Out of roundness — poor concentricity or deformed extrudate	
Cone breaks	See: Pinholes, lumps, tears, splits or cone breaks	
Contamination	1. Improper resin handling 2. Extruder not properly cleaned  3. Poor regrind quality 4. Inadequate screen pack	1. Keep resin clean 2. Clean extruder, remove all traces of other resins or degraded Hytrel® 3. Use only clean dry regrind 4. Fit proper screen pack
Deformed/Folded/Warped extrudate	1. Uneven/insufficient melt tension  2. Burr or other imperfection on die face or pin 3. Draw rate too high (wire covering)  4. Draw-down ratio too high (tubes) 5. Uneven shrinkage (profiles)	1. a. Increase draw-down ratio b. Reduce melt temperature c. Adjust die centering (tubes) d. Hole in mandrel may be too large and should be made smaller (cross-head covering) 2. Remove imperfection 3. Reduce draw rate by lengthening cone (reduce vacuum) 4. Reduce draw-down ratio 5. a. Improve support of profile in water bath b. Reduce melt temperature c. Change to low shrinkage/slow crystallizing grades of Hytrel®

<b>Problem</b>	<b>Probable Causes</b>	<b>Suggested Solutions</b>
Frozen particles in extrudate	See: Unmelted or frozen particles in extrudate	
Diameter variations along the extruded length (round profiles, tubing or cross-head covering)	<ol style="list-style-type: none"> <li>1. Extruder output surging</li> <li>2. Variations in take-off speed</li> <li>3. Temperature cycling</li> <li>4. Draw-down ratio too low</li> <li>5. Variations in diameter of substrate (covering)</li> <li>6. Excessive drag on sizing die or sizing plates (tubing extrusion)</li> </ol>	<ol style="list-style-type: none"> <li>1. See "Surging"</li> <li>2. a. Check speed control of haul-off b. Increase pressure on haul-off belts if slipping, or reduce belt pressure if overloaded</li> <li>3. a. Check operation of controllers including setting of proportional band. b. Check constant material temperature in hopper</li> <li>4. Increase draw-down ratio</li> <li>5. Check substrate</li> <li>6. a. Check design of sizing die b. Reduce vacuum on sizing die c. Adjust air gap before entering sizing die d. Increase lubricating water flow to front of die</li> </ol>
Internal "ripples" on surface of tubing	<ol style="list-style-type: none"> <li>1. Excessive water turbulence in cooling bath</li> <li>2. Mechanical vibrations in equipment</li> <li>3. Stick-slip action in sizing die</li> </ol>	<ol style="list-style-type: none"> <li>1. Eliminate all water turbulence</li> <li>2. Eliminate vibrations</li> <li>3. a. Reduce vacuum on sizing die b. Sandblast inner surface of sizing die c. Increase draw-down ration (change extruder pin/die)</li> </ol>
Loose coatings (cross-head covering)	<ol style="list-style-type: none"> <li>1. Too rapid cooling</li> <li>2. Cone too long (cools before drawn down onto core)</li> </ol>	<ol style="list-style-type: none"> <li>1. a. Lengthen air gap b. Reduce extrusion rate</li> <li>2. Shorten cone by increasing (or applying) vacuum through pin/mandrel</li> </ol>
Out of roundness — poor concentricity or deformed extrudate (tubes or cross-head covering)	<ol style="list-style-type: none"> <li>1. Die out of round, pin/mandrel out of round or bent</li> <li>2. Covering sags before freezing or extrudate sags before entering water bath or sizing die</li> <li>3. Pressure applied by haul-off belts or other take-up equipment is too high, and causes deformation of the extrudate</li> <li>4. Uneven cooling rate</li> <li>5. Die out of adjustment</li> <li>6. Pin or mandrel too flexible (this will generally give variable concentricity)</li> <li>7. Hole in mandrel too large for core or wire being covered</li> </ol>	<ol style="list-style-type: none"> <li>1. Replace or re-machine die or pin</li> <li>2. Generally, increase melt tension: <ol style="list-style-type: none"> <li>a. Reduce temperature of melt</li> <li>b. Increase the rate of draw-down by either: increasing extrusion rate, shortening the cone length or increasing the draw-down ratio (pin/die change)</li> </ol> </li> <li>3. a. Reduce belt pressure b. Reduce payoff tension in cross-head extrusion c. Increase cooling capacity to ensure that extrudate is cold before it reaches haul-off/wind-up equipment</li> <li>4. a. Adjust die centering b. Ensure uniform water cooling around extrudate</li> <li>5. Adjust die centering</li> <li>6. a. Redesign pin b. Use shorter pin</li> <li>7. Reduce size of hole in mandrel</li> </ol>

<b>Problem</b>	<b>Probable Causes</b>	<b>Suggested Solutions</b>
Overloading of extruder drive (excessive torque)	<ol style="list-style-type: none"> <li>1. Rear barrel temperature too low</li> <li>2. Other temperature too low</li> <li>3. Incorrect screw design</li> <li>4. Flow restrictions in head or adaptor/ screen area</li> </ol>	<ol style="list-style-type: none"> <li>1. a. Raise rear barrel temperature b. Check thermocouple and controller of rear zone</li> <li>2. Raise temperature</li> <li>3. Use correct screw design</li> <li>4. a. Check for cold plugs, etc. b. Increase adaptor/head temperatures c. Check design of head</li> </ol>
Pinholes, lumps tears, splits or cone breaks	<ol style="list-style-type: none"> <li>1. Contamination</li> <li>2. Excessively high draw-down ratio</li> <li>3. Temperature of extrudate too low</li> <li>4. Die emperature too low</li> <li>5. Poor dispersion of fillers or pigments or too high loading thereof</li> <li>6. Lumps of degraded resin released from within head or die</li> </ol>	<ol style="list-style-type: none"> <li>1. See "Contamination"</li> <li>2. Reduce draw-down ratio</li> <li>3. Raise melt and/or die temperature</li> <li>4. Increase die temperature</li> <li>5. Improve blending procedures</li> <li>6. a. Clear head and die b. Ensure head design is streamlined with no "hold-up" spots</li> </ol>
Shrinkback, excessive (wire covering and other cross-head operations)	<ol style="list-style-type: none"> <li>1. Orientation too great during draw-down/cooling stage</li> </ol>	<ol style="list-style-type: none"> <li>1. a. Decrease draw-down ratio b. Decrease quench rate (lengthen air-gap/use hot water quench) c. Preheat wire/core material d. Raise melt and/or die temperature</li> </ol>
Splits	See: Pinholes, lumps, tears, splits	
Surface roughness	<ol style="list-style-type: none"> <li>1. Melt fracture ("sharkskin" or roughness on surface)</li> <li>2. Die imperfections</li> <li>3. Contamination</li> <li>4. Wire or core vibration (cross-head covering)</li> <li>5. Core/substrate not smooth</li> <li>6. Build-up (deposits) on die face</li> <li>7. Resin degradation — to determine if roughness is due to melt fracture or degradation, significantly slow down the extrusion. If there is melt fracture, it will disappear. Symptoms of degradation (bubbles and discoloration) will persist or become worse</li> <li>8. Moisture in resin</li> <li>9. Die temperature too low</li> </ol>	<ol style="list-style-type: none"> <li>1. Generally, reduce shear in die by: <ol style="list-style-type: none"> <li>a. Reducing extrusion rate</li> <li>b. Increasing die temperature</li> <li>c. Increasing melt temperature</li> <li>d. Increasing die opening/increasing draw-down ratio</li> </ol> </li> <li>2. a. Check for burrs, etc., and remove b. Check for good finish on die and pin</li> <li>3. See "Contamination"</li> <li>4. Use guides or pads to dampen vibration</li> <li>5. Check core/substrate</li> <li>6. a. Keep die face cleaned b. Increase die temperature</li> <li>7. See resin degradation as a cause of "bubbles"</li> <li>8. See "bubbles"</li> <li>9. Increase die temperature</li> </ol>

<b>Problem</b>	<b>Probable Causes</b>	<b>Suggested Solutions</b>
Surging	<ol style="list-style-type: none"> <li>1. Irregular feeding</li> <li>2. Inadequate melt back-pressure</li> <li>3. Temperature variations</li> <li>4. Barrel or screw wear</li> <li>5. Slippage in haul-off, or speed variations</li> </ol>	<ol style="list-style-type: none"> <li>1. a. Use higher temperature in first barrel zone b. Check feed throat is water cooled c. Check screw design is suitable (may need screw with longer feed section)</li> <li>2. Install finer mesh screen pack</li> <li>3. Check temperature controllers correctly tuned, thermocouples properly located, and heater wattage suitable for zone being heated</li> <li>4. Refurbish barrel and/or screw</li> <li>5. Tighten belts, check speed, etc.</li> </ol>
Tears	See: Pinholes, lumps, tears, splits or cone breaks	
Unmelted or frozen particles in extrudate	<ol style="list-style-type: none"> <li>1. Barrel temperature setting too low</li> <li>2. Granules not melted early enough in screw</li> <li>3. Heater watt density too low (one or more heaters is "on" most of the time)</li> <li>4. Compression ratio of screw too low</li> <li>5. Inadequate screen pack</li> <li>6. Cold spot(s) in extruder</li> </ol>	<ol style="list-style-type: none"> <li>1. Raise controller settings</li> <li>2. a. Change temperature profile raising rear reducing front temperature if necessary to keep acceptable melt temperature b. Change screw to recommended design</li> <li>3. Change heater band to increase wattage</li> <li>4. a. Change screw to recommended design b. Increase screen pack density</li> <li>5. See 4 (b)</li> <li>6. a. Check operation of heaters, controllers, and thermocouples and repair/recalibrate if necessary b. Raise temperatures or supply additional heater capacity to barrel extension, adaptor, breaker plate or die; use separate controllers for each of these zones c. Increase back pressure with screen pack</li> </ol>
Voids (in center of section)	<ol style="list-style-type: none"> <li>1. Too rapid cooling</li> <li>2. Section too thick</li> <li>3. Use of high shrinkage/fast crystallizing grade</li> </ol>	<ol style="list-style-type: none"> <li>1. Reduce cooling rate (use air cooling or alternate water/air cooling)</li> <li>2. Redesign to reduce thickness</li> <li>3. Use lower shrinkage or slower crystallizing grade</li> </ol>

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**Europe, Middle East & Africa**

DuPont de Nemours International S.A.  
Geneva, Switzerland  
Customer Information Center  
Toll-free +800 3 876-6838  
Tel: +41 22 717-5950  
Fax: +41 22 717-5948  
E-mail us at: [eu-info@dupont.com](mailto:eu-info@dupont.com)

**North America**

DuPont Engineering Polymers  
Wilmington, Delaware, USA  
Toll Free: (800) 441-0575  
Tel: (302) 992-4592  
Telefax: (302) 992-6713  
E-mail us at: [Web-inquiries.DDF@usa.dupont.com](mailto:Web-inquiries.DDF@usa.dupont.com)

**South America**

DuPont do Brasil S.A.  
Al. Itapecuru, 506 Alphaville  
06454-080 Barueri-Sao Paulo  
Tel: (5511) 4166-8299  
Fax: (5511) 4166-8513

**Asia Pacific**

DuPont Kabushiki Kaisha  
Sanno Park Tower, 11-1  
Nagata-cho 2-chome  
Chiyoda-ku, Tokyo 100-6111  
Japan  
Tel: (81-3) 5521 8500  
Fax: (81-3) 5521 2595

**DuPont China Holding Co. Ltd.**

15/F, Shui On Plaza  
333 Huai Hai Road (Central)  
Shanghai 200021  
China  
Tel: (86-21) 6386 6366  
Fax: (86-21) 6386 6333

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