# Dynisco The Dynisco Extrusion Processors Handbook 2nd edition

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# SECTION 1: INTRODUCTION TO EXTRUSION

SINGLE SCREW EXTRUSION

➤ TWIN SCREW EXTRUSION

**> EXTRUSION PROCESSES** 

≻ SAFETY

# SINGLE SCREW EXTRUSION

Extrusion may be defined as a process for making a product (an extrudate) by forcing a material through an orifice or die to form a shape, or alternatively, the production of finished, or semi-finished products, using an extruder.

# Scope of the Handbook

This book will be mainly concerned with the extrusion of thermoplastic products because of their importance in extrusion processes. Thermoplastics are by far the largest group of plastic materials extruded; approximately 65% of all plastics pass through an extruder. As the most popular type of extruder is the single screw machine, this book will mainly concentrate on processes based on that type of machine. Single screw machines are the most popular because they are relatively simple, cheap and easily give a continuous output.

#### **Extrusion and Thermoplastics**

In extrusion of a thermoplastic, heating first softens the material so that it can be shaped. The extrusion machine, or extruder does this process. This heat softening is called by various names, such as 'plastication', 'plasticization' or 'thermal softening'. Most extruders are single screw machines. It is the screw which forces the material towards, and then through, the die. Shape is imparted by the die, and/or by post-extrusion forming, and then the product is set to shape by cooling it while maintaining its shape. The equipment that does this is called the post extrusion equipment, while the whole system is called an extrusion line.

# **Extruder Classification**

Extruders may be classified by three figures, for example, 1-60-24. The first number states how many screws the machine has, the second number specifies the screw diameter in millimeters (mm) and, the third number specifies the effective screw length as a multiple of the screw diameter. In the example given therefore, a single screw machine is being described that has a screw of diameter 60 mm and a length of 24 screw diameters (that is, a L/D ratio of 24/1).

#### **Machine Construction**

A cut-away drawing of a simple single screw machine is shown in **Figure 1**. This shows the arrangement of the different parts of the machine. The screw and barrel are the two units that interact to convey the plastic material, melt the material and then force it through the die. The electric motor drive unit and gearbox rotate the screw at a predetermined speed. Temperature controllers

are connected to heating/cooling elements on the barrel to hold the temperature at the set-point temperatures. The ability of the screw and barrel assembly to extrude a given material is dependent on the characteristics of the plastics material, the characteristics, or construction, of the screw and barrel, and the conditions under which the system is operated.



Figure 1. Single Screw Extruder (courtesy of Dr. Harold Giles)

#### **Post Extrusion Equipment**

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Once the extrudate leaves the die it can either be set to the shape to be produced or have its shape altered and then set to shape. The equipment that does this is called the 'post extrusion equipment' or the 'haul off' and in terms of size it is usually far larger than the extruder. One reason is that plastics take a long time to cool. This cooling process often determines how fast the line will operate.

#### **Products of Extrusion**

The products of extrusion include:

- Feedstock for Other Plastics Processes: Extruders are widely used as compounders, or mixers. The output from an extruder compounder is granulated or chopped to form the feed for another process, such as, injection molding or extrusion
- · Plastic Film: This is usually used for packaging or sealed into bags
- Plastic Pipe: Used for gas, water, drains, etc.
- Plastic Tubing: Used for hose and tubing for automobiles, laboratories, etc.
- Plastic Insulated Wire and Cable: Used in the home and industry for appliances, for electric power distribution, communications etc.
- Profile: Used for tracks, windows, doors, home siding, gaskets, etc.
- Filaments: Used for brushes, ropes, twine, etc.
- Sheet: Used for signs, lighting, glazing, etc.
- Nets: Used for packaging, soil stabilization, etc.
- Plastic Coated Paper and Metal: Used for packaging

#### **Co-Extrusion**

Co-extrusion is a process where two, or more, melt streams are combined in a die to produce an extrudate formed from two, or more, materials. The process is now associated with thermoplastics materials although it was first practiced

with rubbery polymers to produce an extrudate with different colored layers. With thermoplastics, the layers of materials are commonly combined in the die. The simplest example combines just two layers, for example a colored layer on a natural core, which saves on colorant costs. In the packaging industry, films based on three or more layers are now common. Laminating two or more layers of different polymers together can produce a product that has barrier properties far superior to those obtained when only one polymer is used. Frequently, one of the polymers used is a gas or moisture barrier layer based on polyvinyl alcohol (PVAL). To produce three layered structures, two or three extruders are usually used. In the blow-molding process it is possible to combine co-extrusion with bi-axial orientation to produce strong, lightweight bottles which give a long storage life to the products.

Layered co-extrusion is not the only type of co-extrusion. By arranging for the materials to be combined one after the other, a sequential co-extrudate may be produced. This may consist of a hard thermoplastic material joined to a soft thermoplastic material. Alternatively, two materials may be extruded side-by-side, such as a clear material alongside an opaque material, to produce sheet to make thermoformed trays.

#### Vented Extrusion

A vented extruder is an extruder that contains a vent, which is used to extract volatiles from a plastic material during the extrusion process. The water (moisture) and volatiles content in a plastic material may be reduced to acceptable levels by the use of a vented machine. Part of the way along the extrusion barrel the melt is decompressed by reducing the screw root diameter. A vent, through which the vapor escapes, is located at this point in the barrel (the vent may be plugged and not used when venting is not required). The vapor-free melt is then conveyed towards the die and re-compressed by increasing the screw root diameter. To ensure that melt does not escape through the vent, a dam or torpedo section is incorporated on the screw just before the vent. Because of the way that vented machines are constructed (a high L/D ratio and a variable screw geometry) and operated, it has been found that they produce very good mixing. It should be remembered, however, that pre-drying of the polymer may be more appropriate, since heating some plastics in contact with water may cause their decomposition, or degradation.

# **> TWIN SCREW EXTRUSION**

Twin screw extrusion machines or, twin screw extruders, are the simplest example of multi-screw machines (Figure 2.). The two screws may rotate in the same direction (co-rotation) or they may rotate in opposite directions (counter or, contra-rotation). The flights of the screws may intermesh or they may not intermesh. As intermeshing, or partially intermeshing, types of machines are the most popular the discussion which follows is restricted to this type of machine.

#### **Uses of Twin-Screw Extruders**

Twin-screw machines have always been popular for certain processes, for example, where there is a need for a compounding step as well as an extrusion step. This is particularly true for un-plasticized polyvinyl chloride (UPVC). This material is often stabilized against heat degradation by the use of heavy metal compounds (like organic tins, or lead) and such stabilizers are expen-



Figure 2. Twin Screw Extruder (courtesy of Krupp Werner & Pfleiderer)

sive. Thus, for economic reasons, the amount of these heat stabilizers must be kept as low as possible. One way of doing this is to compound and extrude in one step. This saves a further heating stage (if the material is first compounded, cooled and then re-extruded). Twin screw machines are widely used to make UPVC pipe and profiles. Today they are often used to compound other plastics, or resins, with additives to make compounds for use in other extrusion or injection molding operations.

#### **Operation and Characteristics**

With a single screw extruder the hopper is filled and the screw takes in the material at the rate it wants. Such a scheme, called flood feeding, is often not possible with twin screw machines. This is because the very positive feeding characteristics of twin screw machines may result in very high forces being generated. These forces may be so high that thrust-bearing failure is a real danger. Twin-screw extruders are therefore often starve fed, resulting in a through-put independent of screw speed. The average residence time is inversely proportional to the screw speed and feed rate and, therefore, decreases with increasing output. The Specific Energy Consumption, or the energy input per unit mass of material, also decreases with increasing throughput. The output is often virtually independent of the size of die used. Because of the mixing action, twin screw machines can achieve more melting, mixing and conveying in a shorter machine length than a single screw machine. They are, however, much more expensive.

#### **Co-Rotating or Counter-Rotating Machines**

Both types of twin screw extruders have distinct advantages that lead to their use in specific applications. Counter-rotating machines are used for the extrusion of UPVC and co-rotating machines are used for compounding applications. The counter-rotating machine has very positive material feed and conveying characteristics. The residence time and material temperature control in the machine are also uniform. However, air entrapment, generation of high pressure, low maximum screw speed and output are disadvantages usually encountered. The advantages of the co-rotating machine are that the screws wipe each other clean (self-wiping), high screw speeds and high outputs are possible, particularly for materials which are not very shear, or heat, sensitive (such as PE). Less screw and barrel wear are also found. The output is, however, dependent on die-head pressure and at high pressures the material residence time distribution becomes broader (the clearances between the flanks of the screws are usually greater for this type of machine). At the high shear rates possible in this type of machine, this non-uniform residence time can result in decomposition of heat sensitive materials. However, the greater interchannel flow results in better mixing or compounding.

# **Tapered Machines**

Because of the very positive feeding characteristics of counter-rotating twin screw machines, very high forces may be generated. These forces can be so high that thrust-bearing failure is a real danger. Such machines therefore often have to be starve fed and/or run at low screw speeds. This is because it is difficult to incorporate adequate thrust bearings in the small space available. Tapering the screw diameter from the feed section to the die can increase the space available. Thus the screws have a larger diameter at the feed end and larger thrust bearings can then be incorporated, permitting higher outputs. The shear seen at the screw tips is also reduced.

# **Compounding Extruder**

The mixing efficiency of a twin screw extruder can be increased by incorporating mixing elements along the screws. These and other elements may be slotted onto a central shaft to build up the screw section required. On some machines the length, number, and form of the elements used may be easily changed. The elements may take various forms such as reversed screw flights, kneading discs, pins, etc. Thus, in the process, the material may be heat-softened in an extrusion section, passed into the mixing section, and then conveyed into another section. This process may be repeated several times, for example in devolatilizing extruders or machines used to carry out chemical reactions.

# Cascade Extruder

The simplest type of cascade extruder consists of two single screw extruders (two screw and barrel assemblies, one mounted above the other) where the output (melt) from the first machine feeds the second. A passageway that may be vented for devolatization connects the two screw assemblies. This type of machine may also be classified as a vented extruder. It offers the advantage that each screw may be driven at a separate speed, so their output can be matched. Valves may be also used to optimize the output from each stage.

# Gear Pump

A gear pump is a very simple twin-screw extruder that moves a fluid material through the action of two intermeshing gears (Figure 3.). A gear pump extruder may be used to produce fibers from polymers that have low viscosities at their processing temperatures. Examples of this type of polymer are polyamides, polypropylenes, or PET.

A gear pump fitted between the end of the barrel and the die of a single-screw extruder will produce a steady high pressure and a more consistent and higher output from the die. Thus, a gear pump can make the output of an extruder virtually independent of back-pressure and, within reason, of screw wear. As the accuracy of output is improved, significant down gauging of the extrudate is possible (approximately 10%). Since the gear pump is used to build up pressure in the die (>70 MPa/10,000 psi), the screw is not required to develop high pressures. This usually results in a reduction in melt

temperatures (approximately 10°C/18°F). The combination of down gauging and melt temperature reduction results in material savings and a higher output, as cooling rate often limits output (See Breaker Plates, Screen Packs and Gear Pumps in Section 4.).



Figure 3. Gear Pump

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# **> EXTRUSION PROCESSES**

Although a single screw or a twin screw extruder may be used to produce the products described in this section, most commonly single screw machines are used as they are simpler and cheaper. For un-plasticized polyvinyl chloride (UPVC), however, twin screw machines are widely used as they can both compound and then shape the material.

#### **Tube or Pipe**

Tube or pipe is extruded from an annular die, usually mounted as an in-line extension (Figure 4.). Within this extension is a mandrel which forms the inner surface of the tube. Low air pressure is usually applied through the mandrel. The tube is held to shape by this internal air pressure as it leaves the die. The molten tube proceeds to a sizing arrangement that sets its dimensions and then to a water-bath or a series of air-cooling rings that fix its dimensions. The sizing equipment, used to control the external diameter of the tube, is normally a water-cooled cylinder (that is, a "sizing die"), which is positioned close to the extruder die. For small-diameter tube or pipe an arrangement of sizing plates or rings, located in the cooling bath, are used instead. Internal air pressure forces the molten tube outward against the inner surface of the sizing die, plates or rings.

With larger tubes or pipes the circular profile can be maintained by applying a partial vacuum through fine holes in the inner surface of the sizing die. For optimal control of OD and roundness vacuum sizing tanks are often used, which combine accurate sizing with controlled product cooling. After passing through the cooling bath, the tube goes to a haul-off, a unit based on rubber caterpillar bands or a system of moving clamps, and then to winding equipment or to a cutting device.



Figure 4. Pipe Extrusion

#### **Blown Film**

Film can be extruded either as a thin-walled tube, cooled by air, or as flat sheet cast onto chilled rolls or into water. In the blown film process (**Figure 5.**) the melt is extruded through an annular die to produce a tube of controlled diameter and wall thickness. Vertical extrusion, where the die is mounted in a right-angle cross-head, is generally used. The extruded melt is partially cooled in the vicinity of the die, often by passing the tube through a carefully controlled stream of air from a cooling ring. The tube of film is then inflated to a bubble of the required diameter, by low-pressure air introduced through the mandrel in the annular die. The film is hauled off between a pair of nip rolls, located 1 to 10+ meters (3 to 30+ feet) from the die. Thus, the inflation air is trapped within the bubble formed between the nip rolls and the die. After passage through the nip rolls, the cold film is reeled up under constant tension.



Figure 5. Blown Film Extrusion

It is important to maintain a steady bubble that feeds to a constant position in the nip rolls. The diameter of the bubble and hence the width of the flattened tube (the "lay-flat" width) is determined by the amount of inflation. The thickness of the film depends on the output from the extruder, the bubble blow ratio (diameter of bubble: diameter of die) and the film haul-off rate. Thus, by varying the blow ratio and the haul-off rate, a range of lay-flat widths and thickness can be produced from the same die. Changing the extrusion conditions may also vary the optical and mechanical properties of film produced from a given polymer.

"Blocking" during the manufacture of tubular film is one of the most serious limitations to high-speed production. "Blocking" is the tendency of opposite faces of films to adhere to each other, thus making separation difficult. If sufficient air-cooling time is not allowed before the tube of film is closed at the nip rolls, then blocking will result. Blocking additives in the polymer such as erucamide or  $TiO_2$  can also be used to alleviate this problem.

#### Flat Film

In the extrusion of flat film the melt is extruded from a straight slit die and cooled either by quenching in water or by casting onto highly polished watercooled chill rolls (**Figure 6.**). Both systems involve a rapid cooling of the film, after which it is wound up on take-off rolls. In the casting process, high temperatures may be used because of the fast cooling possible with chill rolls. Therefore, much higher output rates can be achieved than in the tubular process. The slit die, which is usually center-fed, is fitted with heaters along its length. When a water-bath is used for cooling, carry-over of water can be a problem. Hence, the chill-roll process, which involves no direct water contact with the film, is usually preferred. The chill rolls must be highly polished because the film surface is an exact reproduction of the roll surface.



Figure 6. Sheet and Film Extrusion

#### **Extrusion Lamination**

The application of polymers such as polyethylene or polypropylene to paper, fabrics, metal foils and various other flexible substrates by the extrusion-lamination process is a rapid and economical method of producing laminates for packaging. The thermoplastic material is extruded in the form of a flat film through a slit die. As the very hot film leaves the die lips, it is cast into a niproll assembly formed by a water-cooled steel casting roll and a rubber-covered pressure roll. The substrate to be coated is fed continuously over the rubber pressure roll into the nip, which presses the molten film on-to and into the substrate to form a laminate. As the laminate runs around the steel roll the plastic layer is cooled and the laminate is taken off by wind-up mechanisms. The extruder output rate and the speed of the substrate control the thickness of the applied coating.

#### Wire Coating

In the wire coating process wire or a bunched cable core passes through a mandrel, mounted in a cross-head, which directs the conductor centrally into the die orifice. Here the conductor comes into contact and is coated with softened plastic. The wire is drawn away from the die under conditions that produce a predetermined insulation thickness (Figure 7.). The position of the tip of the mandrel relative to the die parallel is very important for most materials, because it controls the tightness of the covering of the wire. Some machines use a vacuum system to control the tightness of this covering. The insulated conductor then passes through one or more cooling baths to a haul-off, or capstan, maintained at constant speed, and from there to a wind-up drum. Generally, an accumulator is inserted between the capstan and the wind-up arrangements to allow completed reels to be changed without interfering with the extrusion operation. Cooling must be gradual, for if the insulation is cooled too rapidly the outer layers will freeze and contract, resulting in voids between the insulation and the conductor.

Foamed coatings can be extruded directly onto wire using a compound containing a blowing agent; a chemical compound that will liberate nitrogen or some other inert gas on heating. Extrusion conditions should be such that the blowing agent decomposes on the last few flights of the screw. The melt issuing from the die is then in an un-blown state and expands to its final dimensions as it leaves the die. Premature expansion leads to a product with a rough external finish.



Figure 7. Wire Coating

#### Sheet

Sheet is extruded through a slit die (See Figure 6.). After leaving the die it is cooled gradually by passing it over a serpentine of water-cooled rolls. With some materials, for example, polypropylene and polystyrene, it is desirable to use a series of polishing rolls to achieve sheet with a good surface finish. The haul-off equipment is designed to prevent distortion of the sheet during cooling and to minimize draw-down. Sheet is generally cut to length with a travelling circular saw or shear.

Incorporating a corrugation unit in the haul-off produces corrugated sheet, with transverse corrugations. A system is also available for forming longitudinal corrugations by using water-cooled forming dies.

Embossed sheet, such as embossed acrylic sheet for lighting fittings, is produced by passing the extruded sheet between a pair of embossing rolls mounted close to the die. The sheet is air cooled as it leaves the die in order to retain a sharp impression.

# <sup>10</sup> Profile

Profiles of open or hollow cross section can be extruded from specially designed dies. Each shape presents its own problems of die design and postextrusion handling. Profile dies are frequently intricate and thus difficult and expensive to make. Unless CAE is used, a lengthy process of trial and error is required to establish both die design and extrusion conditions (A case in point, is to prevent preferential flow in the thicker sections).

Such dies can be used for small sections, but a simpler technique is desirable for larger profiles (for example, for diffusers for fluorescent lighting fittings). One technique involves post-forming sections extruded from tube dies. Compared with profile dies, these large tube dies can be made at low cost and their symmetry facilitates uniform flow.

The inner lip of the die can have a reeded surface that imparts a pattern to the extrudate. A knife mounted in the die face, slits the emerging reeded tube and the tube passes to a shaping and cooling jig. Alternate inner and outer formers do the shaping with polished forming surfaces. Cooling is accomplished by internal and external perforated copper rings through which is directed a gentle flow of air. The haul-off may be the rubber caterpillar-band type, and an on-demand knife cutter can cut off sections. Trial and error will again be necessary to establish conditions and former design, but adjustments to formers are made more quickly and cheaply than changes to a profile die.

#### **Extrusion Blow Molding**

Hollow articles (such as bottles) are produced using the blow molding process. This process normally starts with the extrusion of a short thickwalled tube, or "parison", from a tube die mounted in a cross-head. This tube is extruded vertically downward between the two halves of a split, watercooled mold. The two halves of the mold are closed, sealing the ends of the molten tube. Air is then applied to the interior of the tube, blowing it outward against the inner surface of the mold. Frequently, as one molding is cooling another is made ready to receive a fresh parison. The split molds may be mounted on a turntable below the extruder die. Alternatively, the melt can be delivered to two or more static molds by arranging for melt to be diverted, in turn, to different dies connected to the same extruder. All extrusion-blown moldings involve welds of the open-ended parison which are potential sources of weakness. Thus care must be taken in the design of the mold to ensure the formation of strong, bead-type, welds. In the blow-molding field it is possible to combine co-extrusion with bi-axial orientation and so produce strong, lightweight bottles which give a long storage life to the products.

# Fiber from Film

The extrusion process can be used to produce fibers or tapes from film. Such products are usually based on polyethylene or polypropylene and they may be used as string, rope or as a woven product (such as a sack). The polymer may be extruded on a single screw machine into a flat sheet using the chill roll process. The sheet may is then slit into tapes by either stationary or rotating knives. Godet rolls are then used to stretch the tape (mono-axial orientation) while it is heated in a hot air oven or on a hot stretching table. In the production of film tape, the width of the tape may decrease to 1/3 of its original slit width as a result of this stretching process. This imparts a significant strength to the tape, in the machine direction. Annealing may then be performed on the tape before it is wound into packages suitable for weaving.

#### Nets

Polymer nets are commonly used in the packaging industry to package fruit. The polymer may be extruded through slots cut in a circular die. By rotating the outside of the die, relative to the center, a circular net may be produced in a wide range of patterns. If the slots are large then heavy gauge nets may be produced which are suitable for landscaping and soil stabilization applications.

# **Product Identification**

In order to ease recycling, the above products or components are frequently marked with a symbol or abbreviated term identifying their component polymers. Legends suitable for the generic identification and marking of plastics products have been suggested by standards organizations such as the International Standards Organization (see ISO 1043). In the simplest case, a series of letters associated with a plastic material **(See Table 1. on page 17)** are stamped onto a product between inverted (reversed angle) brackets (for example, >ABS<).

ISO also suggests how to identify fillers, flame retardants, plasticizers etc. For example, the presence of a flame retardant may be indicated by a molded or printed legend which contains FR as in >PA 66-GF30-FR(52)<. This legend shows that the product is made from a nylon 66 material that contains 30% glass fiber. The FR(52) indicates that the nylon contains also a red phosphorous (code number 52) type flame retardant. Other flame retardants have their own numbers.

# ≻ SAFETY

This section is written to help an operator, who runs an extruder or an extrusion line, to work in a safe way. It cannot cover all the possible hazards, as it is only a general guide. Safety is the responsibility of everyone. It is very important that everyone who is concerned with an extrusion process fully appreciates the possible hazards. This means that safety education and training are very important.

# A New Machine

Before a new extruder is put into service a responsible person must check that the machine is acceptable from a safety point of view and that it conforms to local and national safety codes and practices. This means listening to the advice of the machine supplier, local government officers, etc. A local plastics association, or federation, will offer useful advice in regard to whom should be contacted and what codes of practice should be followed. Any legislation in force should be considered, as the minimum required. It is very important to ensure that all those concerned with extrusion processing develop a common sense, responsible attitude, and are trained to increase their safety awareness.

# **Materials Handling**

It must be emphasized that most thermoplastic materials do not present problems during normal handling as they are usually supplied as granules or pellets. However, to prevent material contamination, the thermoplastic material should not be touched or handled, with the bare hand. Remember, it makes sense not to allow any material to come into contact with the skin or eyes, particularly if the material is dusty (for example, the material may have been dusted with a lubricant or coloring system). If during normal operation, the atmosphere becomes dusty then the operating procedure should be reviewed 12 and modified until the contaminated atmosphere is eliminated.

When handling a thermoplastic material, the biggest danger is often one of spilled material. Because of the shape of the pellets, the spilled material makes the floor very slippery and can cause falling injuries. Therefore, any spilled material must be collected immediately and disposed of in an approved way. This usually means sweeping up the spilled material and placing it an appropriately labeled bag for reclamation.

#### Burns

Some extruders operate at temperatures of as high as 750°F/400°C or above. As any temperature above 140°F/60°C is unpleasant to touch, burns must be recognized as a common source of accidents in the extrusion processing industry. It is one of the reasons that a cover or guard usually protects the extruder barrel. Like any other guard it should not be needlessly opened, or removed, as it is there for protection of both the operators and the equipment.

At the processing temperatures employed, hot thermoplastic materials (resins) behave like hot melt adhesives and can be very difficult to remove from the skin. These resins are organic materials that have high specific heats and low thermal conductivities. Thus, as the hot melt contains a large amount of heat, severe burns can result. The burning process can be very painful as the hot melt releases the heat it contains very slowly. An instinctive reaction, when hot melt comes into skin contact, is to pull the hot melt away with a bare hand, but this simply transfers the hot, sticky resin to another area. One should quickly scrape off the resin, plunge the covered area into cold water and obtain medical advice.

#### **Resin Decomposition and Fumes**

Thermoplastic materials (resins) are organic materials, which, generally, have low thermal stability. At the processing temperatures employed they can easily be degraded to give unpleasant, irritating odors and, if seriously overheated, they can produce a large quantity of noxious gas. Such gases should not be inhaled, and treated as harmful. This means that if, for example, the extruder barrel is stripped for cleaning it should be done in an extremely well ventilated area. Gloves, long sleeved overalls and a facemask should be worn when the barrel and screw are stripped and cleaned.

If the gas produced by resin decomposition becomes trapped inside the machine, it can cause serious accidents. It can blow hot resin (plastic or melt) from the nozzle or even through the hopper. In the worst case, it could produce an explosion and cause damage to the machine, such as shearing the bolts holding the die/nozzle. Thus one should be careful and follow the correct operating procedures, particularly at the start up of an extrusion operation.

#### Rolls

Rolls, such as the rubber covered rolls for pulling and steel rolls for cooling, are widely used in extrusion processing. The gap between them, called the 'nip' or 'bite', is very dangerous, as it is capable of trapping fingers or clothing causing serious injury. Such nips must be guarded, treated with respect, and avoided. Long hair should be kept covered; neckties and other loose clothes should not be worn as they can also become trapped. Be particularly careful if you have to wear gloves as these can become trapped; it is probably best to make the gloves as small as is convenient (for example, slit the backs and remove the cuffs). Learn a start-up technique that avoids the possibility of trapping a hand in the nips. Above all, find out what to do if something, or

somebody, does get trapped. This means knowing how to stop the machine in a hurry and how to release or open the nip.

#### The Screw

Normally the only part of the screw that the operator sees is that which is exposed when the machine runs out of material, that is at the base of the hopper. It looks fairly harmless but it is very dangerous and will easily remove your fingers. So, keep your hands out of the hopper and only put them into this danger area when you are satisfied that it is safe to do so; when the screw is stopped, the power is off and the machine is isolated. Always check that the machine is isolated *yourself*.

To protect the expensive screw, do not put metal tools into the hopper when the screw is turning. There should be a guard between the base of the hopper and the screw (to prevent finger trapping) and ideally, the screw should not turn if the hopper is removed. Do not remove the hopper, and expose the screw, until the extruder is stopped and you have checked that the machine is isolated.

#### The Die or Nozzle

The die (sometimes called the nozzle) is often the part of the extruder that an operator sees the most during machine operation. Accidents involving the die are a major hazard because of failure to protect the operator against danger from burning or finger trapping. Therefore, do not reach over moving equipment and *never* remove a guard or, attempt to bypass a guard.

When cleaning or stripping the die make sure that the equipment is locked in the non-running mode and that the power (electric) supply is disconnected. Specific cleaning instructions from the die maker should be followed to prevent danger to the operator and damage to the forming surfaces of the die.

# **Other Equipment**

Cutting, grinding, sawing and chopping equipment must, of course, be well guarded. They usually are the most common dangers that arise when a safe working approach is ignored. Do not reach over moving equipment and *never* remove or attempt to bypass a guard. If you have been specifically authorized to do something like this, then make sure that the equipment is locked in the non-running mode and that all electric power is disconnected.

# Work Handling Devices

A work-handling device, also known as a robot, is an item of parts handling equipment (PHE). When guarding a parts handling device, such as a robot, consideration should be given to the area covered by the machine, the speed of operation of the arm, the speed of acceleration/deceleration of the arm, the complexity of the movements involved and the time-scale over which the movements occur. This last point is very important as often an operator can be mislead into believing that the PHE is in-operative as it appears to be switched off when it is standing still.

When guarding a parts handling device (or a robot) both the downstream operations and the working area of the PHE must be considered. Special custom-built enclosures must often be built for each application.

# **Electrical Equipment**

Most extrusion equipment is heated by electric resistance elements and driven

14 by electric motors. Machine control cabinets are often kept locked to protect the operator from coming into contact with the main electrical supply. Leave such equipment alone unless you are trained and authorized to inspect, repair or adjust it. Report any damaged, or frayed, wires or cables immediately, and do not disconnect the equipment ground wires. Any damaged plugs or connectors should be reported.

# Safety Guards

An extrusion line contains dangerous machinery that must be guarded appropriately. Safety guards cover many mechanical, electrical and heat hazards and these must be kept in place. Remember: the guards are there for your protection. Machine guarding is just one way of making the process safer and should not be relied upon to the extent that inspection, maintenance and training are neglected.

# SECTION 2: MATERIALS AND THEIR FLOW PROPERTIES

- > POLYMERS AND PLASTICS
- > THERMOPLASTIC MATERIALS
- > VISCOSITY AND VISCOSITY TERMS
- **> FLOW PROPERTIES MEASUREMENT**
- > ELASTIC EFFECTS IN POLYMER MELTS
- ► DIE SWELL
- MELT FRACTURE
- SHARKSKIN
- ► FROZE-IN ORIENTATION
- ► DRAW-DOWN

# POLYMERS AND PLASTICS

A polymer is a chemical compound made up of a large number of identical molecular repeating units. A plastic material is a polymer, generally modified with additives, which is capable of being shaped or molded under reasonable conditions of temperature and pressure. It generally is solid at room temperature and is distinguished from a rubber/elastomer by having a higher stiffness/modulus and a lack of reversible elasticity. A thermoplastic is a plastic material that will soften (melt) on heating and harden on cooling. This softening (or melting) by heat and hardening by cooling can be repeated. This allows thermoplastic materials to be recycled.

#### **Synthetic Polymers**

Most commercial polymers and rubbers/elastomers are composed of molecules based on the element carbon; that is to say, they are organic. They are synthesized, or made, from simple, oil-based raw materials called monomers. A process, known as polymerization, connects these simple low molecular weight materials together to form polymers, which consist of long chains containing many identical repeat units. Polymers and rubbers based on one monomer are known as "homopolymers", while those based on two (or more) monomers and are known as 'copolymers'. They may be linear, branched or "cross-linked" (i.e., two or more polymer chains are connected). Usually, polymer chains of a wide range of lengths (chains of varying molecular weight) are present in commercial materials.

Since the final molecular weight (and size) is generally large, these materials are frequently referred to as "high polymers" or "macromolecules". Although a high molecular weight is necessary to obtain useful properties (such as stiffness and strength) in a plastic material, it also means that the material is difficult to process.

All plastics are polymers but not all polymers are plastics. Cellulose, for example, is a polymer, but it cannot be processed like a plastic material unless it is modified.

#### Long Chain Structure

The "high polymer molecules", or "macromolecules", that are used in plastics are incredibly long. There may be, for example, 50,000 atoms linked together in a long chain-like molecule or structure. Because of the structure of the carbon to carbon bond on which they are based, they generally are not straight, but are twisted or coiled. As a result of the nature of the polymerization reactions, the chains also have a distribution of different lengths.

When there is no order in the system (an amorphous material), the chains prefer to exist in a random, coiled-up state (just like the shape obtained when a piece of string is dropped on the floor). The separate long chain molecules (or "chains") are also normally intertwined one with the other. If crystallization of the polymer is possible, then the chains, or portions of the chains, may either fold back on themselves or lie side-by-side in a more ordered fashion. The great length of the chains and the entanglements between them prevent thermoplastic materials from crystallizing completely on cooling. This is why they are called "semi-crystalline thermoplastics". Such materials contain both crystalline and amorphous regions.

#### **Amorphous Thermoplastics**

Thermoplastic materials may be divided into amorphous materials and semicrystalline materials. An amorphous thermoplastic is usually a glassy, hard material that, in the unfilled state, is transparent and rigid. This type of plastic material does not have a sharp melting point, but softens to a melt over a broad range of temperature. It, therefore, can be molded or processed over a comparatively wide temperature range. A major advantage of amorphous polymers is their low and consistent shrinkage, relative to that of semi-crystalline materials. As the natural color of an amorphous material is generally a clear, water-white, a very wide range of transparent or opaque colors may be produced.

Typical amorphous polymers include polystyrene (PS), polymethylmethacrylate (PMMA), styrene acrylonitrile (SAN), polycarbonate (PC), polysulfone (PSU or PES) and polyvinyl chloride (PVC). Modified polyphenylene oxide (PPO-M) and acrylonitrile butadiene styrene (ABS), although not usually transparent, are based on amorphous thermoplastic materials. Some widely used abbreviations for thermoplastic materials are shown in **Tables 1**. **and 2**..

#### Semi-Crystalline Polymer

The amount of crystalline material (% crystallinity), in an extrudate made from a polymer that will form crystals, is typically not 100%. Amorphous areas are always present. Thus these materials are referred to as semi-crystalline. Typical semi-crystalline polymers are polyacetal (POM), polyamides (PA 6 and PA 66), polybutylene terephthalate (PBT), polyethylene terephthalate (PET), low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyphenylene sulfide (PPS), and polypropylene (PP).

Semi-crystalline polymers are usually malleable, tough, durable, and have a good chemical resistance. Usually they are not clear because the crystal structures that they contain scatter light. Semi-crystalline polymers may have a sharp melting point or, more usually, a fairly narrow melting point range. Considerable care is required in their processing. The properties of

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formed components may be greatly affected by the degree of crystallization and crystalline morphology (structure) that occurs during solidification. In addition, they exhibit greater shrinkage variations than amorphous polymers. Therefore a greater emphasis on the uniformity of cooling is required when processing a semi-crystalline material.

#### Orientation

The most commercially important polymer conversion processes involve meltprocessing methods in which the polymer is heated and made to flow. In the melted form all polymers are amorphous; that is they have no structure. When the polymer flows, the polymer chains slip, or slide, over each other while rotation around the carbon-to-carbon bonds also occurs. Layers slip one over the other in what is called laminar or shear flow. As the individual polymer molecules move relative to each other, they may change their direction or orientation as a result of chain entanglements and friction between layers. The chains thus become "drawn out" in the direction of flow. Because of the rapid cooling often employed in polymer processing, this orientation may be "frozen-in" and the product will contain, what are referred to as, frozen-in stresses or, frozen-in strains. These products thus contain molecules that are orientated in the flow direction, which means that there is a "grain effect" present – just like that which exists in wood. As the result of the grain, the wood is stronger in one direction that it is in another.

Because of orientation, thermoplastic products may be much stronger in one direction than in another; that is, the product is anisotropic. Commercial use is made of this "uni-axial" effect in extrusion in the production of the tape used for strapping packages. Biaxial orientation (orientation in two perpendicular directions) allows the production of strong, stiff, film from brittle polymers, such as polystyrene (PS).

# Table 1.Standard (Based on ISO and ASTM) AbbreviationsFor Selected Thermoplastics

Name
acrylonitrile-butadiene-acrylate (was A/B/A in ISO)
acrylonitrile-butadiene-styrene
See ABAK
See ACS
acrylonitrile-chlorinated polyethylene-styrene (was A/CPE/S in ISO)
acrylonitrile-ethylene-propylene-diene-styrene (was A/EPDM/S in ISO)
See AES
See AMMA
acrylonitrile-methyl methacrylate (was A/MMA in ISO)
acrylonitrile-styrene (SAN is more usual in Europe)
acrylonitrile-styrene-acrylate
cellulose acetate
cellulose acetate butyrate
cellulose acetate propionate
cellulose nitrate
cellulose propionate
See EEAK
ethylene-ethylene acrylate (was E/AK in ISO)
See EMA

18	Abbreviation	Name
	EMA	ethylene-methacrylic acid (was E/MA in ISO)
	E/P	See EP
	EP	ethylene-propylene (was E/P in ISO)
	EPDM	ethylene-propylene-diene
	E/TFE	See ETFE
	ETFE	ethylene-tetrafluoroethylene (was E/TFE in ISO)
	EVAC	ethylene-vinyl acetate
	EVAL	See EVOH
	EVOH	ethylene-vinyl alcohol (was EVAL in ISO)
	FEP	fluorinated ethylene-propylene
	LCP	liquid crystal polymer
	MABS	methyl methacrylate-butadiene-styrene
	MBS	methacrylate-butadiene-styrene
	PA	polyamide
	PAEK	polyaryletherketone
	PAI	polyamideimide
	PAK	polyacrylate
	PAR	polyarylate
	PAUR	poly(ester urethane)
	PB	polybutene-1
	PBAK	poly(butyl acrylate)
	PBT	poly(butylene terephthalate)
	PC	polycarbonate
	PCTFE	polychlorotrifluorethylene
	PE	polyethylene
	PEBA	poly(ether block amide)
	PEEK	polyetheretherketone
	PEEKK	polyetheretherketoneketone
	PEEST	polyetherester
	PEI	poly(ether imide)
	PEK	polyetherketone
	PEKEKK	polyetherketoneetherketoneketone
	PEKK	polyetherketoneketone
	PEOX	poly(ethylene oxide)
	PES	poly(ether sulfone)
	PESTUR	poly(ester urethane)
	PET	poly(ethylene terephthalate)
	PEUR	poly(ether urethane)
	PFA	perfluoro alkoxyl alkane
	PFEP	perfluoro(ethylene/propylene)
	PI	polyimide
	PIB	polyisobutylene or polyisobutene
	PMMA	poly(methyl methacrylate)
	PMP	poly-4-methylpentene-1
	PMS	poly- $\alpha$ -methylstyrene
	POM	polyoxymethylene or, polyacetal or, polyformaldehyde
	PP	polypropylene
	PPE	poly(phenylene ether)
	PPOX	poly(propylene oxide)
	PPS	poly(phenylene sulfide)
	PPSU	poly(phenylene sulfone)
	PS	polystyrene
	PSU	polysulfone
	PTFE	polytetrafluoroethylene
	PUR	polyurethane

Abbreviation	Name
PVAC	poly(vinyl acetate)
PVAL	poly(vinyl alcohol)
PVB	poly(vinyl butyral)
PVC	poly(vinyl chloride)
PVDC	poly(vinylidene chloride)
PVDF	poly(vinylidene fluoride)
PVF	poly(vinyl fluoride)
PVFM	poly(vinyl formal)
SAN	styreneacrylonitrile
S/B	styrenebutadiene
S/MS	styrene- $\alpha$ -methylstyrene
SMA	styrenemaleic anhydride
VC/E	See VCE
VC/MMA	See VCMMA
VC/VDC	See VCVDC
VC/VAC	See VCVAC
VC/MA	See VCMAK
VC/OA	See VCOAK
VC/E/MA	See VCEMAK
VC/E/VAC	See VCEVAC
VCE	vinyl chloride-ethylene (was VC/E in ISO)
VCEMAK	vinyl chloride-ethylene-methyl acrylate
	(was VC/E/MA in ISO)
VCEVAC	vinyl chloride-ethylene-vinyl acetate
	(was VC/E/VAC in ISO)
VCMAK	vinyl chloride-methyl acrylate (was VC/MA in ISO)
VCMMA	vinyl chloride-methyl methacrylate
	(was VC/MMA in ISO)
VCOAK	vinyl chloride-octyl acrylate (was VC/OA in ISO)
VCVAC	vinyl chloride-vinyl acetate (was VC/VAC in ISO)
VCVDC	vinyl chloride-vinylidene chloride

The above abbreviations may be modified by the addition of up to four specified letters after the abbreviation for the polymer under discussion. For example, PS could become PS-HI when high impact polystyrene is being described. In the case of PE, this would become PE-LD when low-density polyethylene (LDPE) is being described.

When mixtures are made from two or more polymers (blends or alloys), ISO 1043 suggests that the symbols for the basic polymers be separated by a plus (+) sign and that the symbols be placed in parentheses. For example, a mixture of \* polymethyl methacrylate and \* acrylonitrile-butadiene-styrene should be represented as (PMMA+ABS)

When a copolymer is being discussed, it is recommended (ISO) that an oblique stroke / be placed between the two monomer abbreviations. (For example, E/P for an ethylene propylene copolymer.) The oblique strokes may be omitted when common usage so dictates according to ISO 1043-1:1987 (E).

# > THERMOPLASTIC MATERIALS

Over the past 50 years the usage of plastic materials throughout the world has increased dramatically and now stands at well over 100 million tons. In terms

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20 of tonnage, thermoplastics are by far the most important, as they are approximately 80% of all plastics used throughout the world.

#### **Advantages of Plastics**

Plastic materials are widely used because they can be formed into complex components, or shapes, relatively easily. They also have a useful combination of properties that can be modified for use in a wide range of applications.

Some of these desirable properties (which are not possessed by any one plastic) include physical strength, resilience, corrosion resistance, elasticity, electrical insulation, wide color range, thermal insulation, lightness in weight, chemical resistance and moldability. These materials are therefore versatile in their applications. Their use frequently simplifies production, due to the fact that they often allow several discrete parts to be incorporated into one component. The ease with which complex parts may be produced from plastic compositions at high speed, in many cases outweighs the disadvantages of plastic materials. Attractive decoration of such products adds to their sales appeal.

However, thermoplastic materials sometimes lack strength, stiffness, temperature resistance, and fire resistance. Not only do some plastics burn relatively easily, but also they often evolve large quantities of smoke and fumes when they do burn. Many plastic components will creep or change their dimensions if subjected to relatively small loads for prolonged periods of time. Environmental stress cracking (ESC), changes of dimensions with temperature and humidity, and a sudden change from tough to brittle behavior are other factors that should be investigated before thermoplastics are used.

# **Major Thermoplastic Materials**

There are two main categories of plastic materials, thermoplastics and thermosetting plastics (thermosets). Because of the commercial importance of thermoplastic materials this book will concentrate on their extrusion processing. This important group of materials has been divided into four groups:

- 1. Commodity or Bulk Thermoplastics
- 2. Engineering Thermoplastics
- 3. Thermoplastic Elastomers or Rubbers
- 4. Blends and/or Alloys

Commodity thermoplastics, such as the polyolefins (polypropylene and polyethylene), polyvinyl chloride plastics and styrene-based plastics comprise, the major tonnage of material produced.

# **Commodity or Bulk Thermoplastics**

Because of their relative ease of manufacture from readily available monomers, their low cost and versatility, usage of polyolefins has grown dramatically in recent years. Both homopolymers and copolymers in this family are available, as are rubbers and thermoplastics materials. The members of the polyethylene family include; linear low-density polyethylene (LLDPE), low density polyethylene (LDPE), high-density polyethylene (HDPE) and very low density polyethylene (VLDPE). The term polyethylene (PE) therefore covers a range of materials. A new class of these resins has recently been produced "using "metallocene" or "single site constrained geometry" catalysts. These catalysts allow the production of homopolymers and copolymers with molecules of more uniform size and structure.

The term polyvinyl chloride (PVC) plastic refers to homopolymers and copolymers, both of which may be used with or without plasticizers. This gives rise to unplasticized grades (UPVC) and plasticized grades (PPVC). The term styrene-based plastic also refers to a range of materials. These include the homopolymer polystyrene (PS), the copolymer styrene-acrylonitrile (SAN), the rubber-toughened material called high impact polystyrene (HIPS) and acrylonitrile-butadiene-styrene (ABS).

The importance of commodity thermoplastics cannot be overemphasized because of their high volume of production and usage. The advent of new catalyst systems (for example, metallocenes) will further increase the usage of bulk thermoplastics, as new types of polymer may be produced. It is also now being realized that the properties of this class of materials can be up-graded, so they can compete with many more expensive engineering thermoplastics.

#### **Engineering Thermoplastics**

The term engineering thermoplastics generally refers to materials that can be used to replace metal, glass or wood in manufactured products. They include both amorphous and semi-crystalline polymers. These plastics, characterized by properties such as high strength, stiffness, impact resistance, temperature resistance and fire resistance are widely used in automotive, transportation, appliance, electronics, communications and aerospace applications. These materials are produced in smaller volumes than commodity plastics, but they command a much higher price. Materials that are considered to be engineering thermoplastics include: polycarbonate (PC), polyethelene terephthalate (PET), polybutylene terephthalate (PBT), polyoxymethylene (acetal), polysmide (nylon), polymethylmethacrylate (PMMA) and acrylonitrile-butadiene-styrene (ABS).

#### **New Material Supply**

Raw material manufacturers or their distributors can supply a material as, for example, either prime grade material or as transitional material. Some may also supply reclaimed material. Prime grade material, also known as virgin material (or as first pass material or as first grade material) is a thermoplastic material (in the form of pellets or granules) that has not been subjected to use, or processing, other than that required for its original manufacture.

Transitional material is material that does not meet the manufacturer's specification for prime grade material: it is off-spec or substandard material. Such material may be obtained from a continuous compounding line when a change is made from one grade to another. This means that the melt flow rate and the base color may be somewhat variable. The cost of virgin material is greater than that of these off-spec materials.

# **Thermoplastic Grades**

A large number of grades may be available for any one material. These grades may differ in molecular weight, molecular weight distribution, chain branching, purity and the presence of additives or modifiers. Therefore, any properties quoted in the literature should be used only as a general guideline, as each of the many plastics available consists of a family of materials. Thus it is possible to get a wide range of properties for each material group. The properties of these thermoplastics may also be dramatically changed by the processing conditions employed. Processing may alter the material's color, its physical properties, amount of molecular orientation and even the type, and amount, of crystallinity.

# Additives

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It is generally understood in the thermoplastics industry that most plastic materials used contain small amounts of additives, as most of these materials cannot be commercially used without them. The properties of a particular material are altered by the use of these additives. Since additives are essential in commercial plastics, the size and scope of the additives industry is now enormous. It is estimated that approximately 7 million tons of additives of various kinds are used in plastics annually. In the thermoplastics industry, the largest amount of additives is used in polyvinyl chloride (PVC).

With most thermoplastic materials, variations of the basic formulas are available to provide improved properties, such as, improved heat resistance or weatherability, by the use of additives. Processing and performance additives such as antistatic, nucleating and mold release agents may also be added to an existing material. These additives may form part of the color masterbatch (a masterbatch is a concentrate). Concentrates containing foaming agents, for structural foam molding, are also available for many materials.

Examples of other additives are reclaimed material, fillers, reinforcements, impact modifiers, colorants, antioxidants, heat stabilizers, light stabilizers, lubricants, coupling agent, flame retardants, cross-linking agents, plasticizers, extenders, nucleating agents, antistatic agents and blowing agents.

# Material Modification

The use of the term "materials modification" usually means the addition of larger amounts (greater than a few percent) of an additive. An additive is used in relatively large concentrations may be referred to as a modifier. Modifiers may include elastomers, flame-retardants and fillers. Material modification with rubber impact modifiers, fibers or fillers has been extensively used with engineering plastics to obtain a desirable combination of properties. These formulations may offer improved impact strength, tensile strength and heat distortion temperature together with ease of flow.

# Trade Name/Trademark Usage

It is common within the thermoplastics industry to refer to a plastic material by a trade name because they are often simpler, and more widely known, than the chemical name of the polymer. The same trade name/trademark, however, is often used for a range of materials, the properties of which may vary widely. Such materials are usually based on the same type of thermoplastic material, for example, acrylonitrile-butadiene-styrene (ABS). However, one material may be largely unmodified whereas another, with the same brand name, may contain large amounts of glass fiber or other additives (Table 2.).

# Table 2.Common Abbreviations and Trade Names/Trademarksfor Thermoplastics and Thermoplastic Elastomers

Abbrevia-	Common
tion	Name
ABS	Acrylonitrile butadiene styrene
AMS	Alpha methyl styrene
ASA	Acrylonitrile styrene acrylate (AAS)
BDS	Butadiene styrene block copolymer
CA	Cellulose acetate
CAB	Cellulose acetate butyrate

Common Trade Names or Trade Marks Cycolac; Lustran Elite HH Luran S K Resin; Styrolux Cellidor; Tenite Cellidor; Tenite

Abbrevia- tion	Common Name	Common Trade Names or Trade Marks
CAP	Cellulose acetate propionate	Cellidor <sup>.</sup> Tenite
CN	Cellulose nitrate	Celluloid: Xylonite
	Collulose propionate (CAP)	Collidor: Topito
CDE	Chloringtod polyothylong (DE C)	Boyor CM: Tyrin CM
UPE ODVO	Childrinaleu polyethylene (PE-C)	Bayer Givi, Tyrin Givi
UPVU	Chiorinated polyvinyl chioride (PVC-C)	Lucalor
EA-MPR	Elastomer alloy melt processable rubber or melt processable rubber	Alcryn
EA-TPV	Elastomer alloy thermoplastic	Lamadi Cantanrana
FOM	Vullanizate	Carilan, Katanay
	Ethylene-Garboli monoxide	Callion, Relonex
EIFE	letratiuoretnylene-etnylene copolymer	Tetzei
EVA	Ethylene vinyl acetate copolymer (EVAC)	Evatane
EVAL	Ethylene vinyl alcohol copolymer	Clarene; Eval
EVOH	Ethylene vinyl alcohol copolymer	Clarene; Eval
FEP	Fluorinated ethylene propylene	Toflop EED
	(IFE-FIFF)	
HUPE	High density polyethylene (PE-HD)	Lupolen HD; Rigidex
HIPS	High impact polystyrene (TPS or TPS)	Lustrex; Polystyrol
LCP	Liquid crystal polymer	Vectra; Xydar
LDPE	Low density polyethylene (PE-LD)	Alathon; Hostalen
MBS	Methacrylate butadiene styrene	Paraloid
MDPE	Medium density polyethylene (PE-MD)	Fortiflex
MPR	Melt processable rubber or, elastomer	
	alloy melt processable rubber	Alcryn
PA	Polyamide or nylon	
PA 6	Polyamide 6 or nylon 6	Akulon K; Ultramid
PA 11	Polyamide 11 or nylon 11	Rilsan B
PA 12	Polyamide 12 or nylon 12	Rilsan A; Grilamid
PA 46	Polvamide 46 or nylon 46	Stanyl
PA 66	Polyamide 66 or nylon 66	Maranyl: Zytel
PA 610	Polyamide 610 or pylon 610	Brulon: Perlon N
PAA 6	Polyaryl amide or poly-m-yylene-	
	adinamide (PA MYD6)	lvof
DAN	Polyaonylopitrila	Aorilan: Paray: Orlan
	FOIYaci yioiiiiiie Dalubutulana taranbthalata	Actiliati, Datex, Utilit
PBI		Pocali, valox
PC	Polycarbonate	Lexan; Makrolon
PUT	Polycyclonexanedimethylol	
	terephthalate	Thermx; Valox
PCTFE	Polychlorotrifluorethylene	Hostaflon C2; Kel-F
PE	Polyethylene	Alathon; Lupolen
PEBA	Polyether block amide (TPE-A)	Pebax
PEEK	Polyether ether ketone	Victrex PEEK
PEEL	Polyether ester (TPE-A or YPBO)	Arnitel; Hytrel
PE-HD	Polvethylene-high density	Lupolen HD: Rigidex
		HDPE
PEI	Polyether imide	Ultem
PEK	Polyether ketone	Hostatec
PEKK	Polyether ketone ketone(from DuPont)	
PE-LD	Polvethylene- low density	Alathon: Lupolen LD
PF-MD	Polvethylene- medium density	Fortiflex
PF-VI D	Polyethylene- very low density	Norsoflex
PFT	Polyethylene terentthalate	Arnite A: Techetor F
DEC	Polyether culphone (culfone)	Victrov
	roiyeniei sulpiione (sullone) Dolvimido	Victita
	FUIYIIIIUUU Delykatope	Vespei
۲K	Polyketone	Garilon

24	Abbrevia-	Common	Common Trade Names
		Nallie Delymethyl metheorylete (servic)	Diskon: Disvision
	PIVIIVIA	Polymethyl methaciylate (acrylic)	Diakon, Piexigias
	PIMP	Polymetnyi pentene	TPX; Grystalor
	POM	Polyoxymetnylene or, acetal	Dell's dissistences
		or, polyformaldenyde	Deirin; Hostaform
	POM-H	Acetal homopolymer	Delrin and Delrin II
	POM-CO	Acetal copolymer	Hostaform; Ultraform
	PP	Polypropylene	Profax; Propathene
	PPE	Polyphenylene ether (see PPO)	
	PPO	Polyphenylene oxide-usually modified	
		polyphenylene oxide (PPO-M)	Luranyl; Noryl
	PPS	Polyphenylene sulfide (sulphide)	Fortron; Ryton
	PPPS	Polyphenylene sulfide sulfone	Ryton S
		(polyphenylene sulphide sulphone)	
	PPVC	Plasticized polyvinyl chloride (PVC-P)	Solvic; Vinnol
		Plasticised polyvinyl chloride (PPVC)	
	PS	Polystyrene (GPPS or PS-GP)	Lustrex; Polystyrol
	PSU	Polysulfone (polysulphone)	Udel
	PTFE	Polytetrafluoroethylene	Fluon; Teflon
	PVC	Polyvinyl chloride	Corvic; Geon
	PVDC	Polyvinylidene chloride copolymers	Saran
	PVDF	Polyvinylidene fluoride	Dvflor: Kvnar
	PVF	Polyvinyl fluoride	Tedlar
	SAN	Styrene acrylonitrile copolymer	Lustran SAN: Tvril
	UPVC	Unplasticized polyvinyl chloride	,,,,
		(PVC-U) Unplasticized polyvinyl	
		chloride (UPVC)	Corvic: Geon
	RMPP	Rubber modified polypropylene a TPO	
		(also known as RRPP or PP/FPDM)	Uniroval TPR: Keltan
	SBS	Styrene hutadiene styrene block	onnogai n'ny nonan'
	000	conclymer or thermonlastic elastomer	
		styrene based (TPF-S)	Cariflex TR: Solprene
	SEBS	Styrene hutadiene styrene block	
	OLDO	Conclymer (saturated) or thermonlastic	
		Elastomer styrene based (TPF-S)	Cariflex
	TPF	Thermonlastic elastomer (rubber)	ourmox
	TPE-A	Polyether block amide ( $PEB\Delta$ )	Pehav
	TPE-F	Thermonlastic elastomer - ether ester	Τούαλ
		or polyether ester elastomer	Arnitel: Hytrel
		Thermonlastic elastomer - nolvolefin	Amilio, mytro
	II L OAL	hased with crosslinked rubber	Levafley: Santonrene
	TDE_C	Thermonlastic electomer styrene	Levanez, Jantoprene
	TFL-5	hasad (usually styrang bytadiang	
		styrene block conclymer)	Carifley TP: Solorene
		Thermonlactic polyurethana (TPU)	Elactollan: Ectano
		Thermoplastic polyclefin may be a	Elasioliali, Estalle
	IFU	hland (and DMDD) a thermoniantia	
		vulconizate (and TDV) or a reactor	
		vuicaliizate (see IPV) of a feactor	
	тор	Thermonlactic rubber (cleatemer)	
		Thermoniastic rubber (elastomer)	Flastallan, Fatara
		Thermoniastic polyurethane	Elastolian, Estane
	IPV	(a TDE with acception with an)	Lamade Cartanina
		(a THE WITH CROSSIINKED RUDDER)	Lomod; Santoprene
		Very low density polyethylene (ULDPE)	Nereefley
	VLUPE	very low density polyethylene (ULDPE)	INDISOTIEX

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# > VISCOSITY AND VISCOSITY TERMS

To shape a polymer or plastic into a part requires that the material first be softened or melted and then forced to flow through a die or into a mold. Thus it becomes very important to understand how polymer and plastic materials flow. Shear flow is one of the most important types of flow occurring in polymer melts during conversion processes. In this type of flow, one layer of melt flows over another on application of a shearing force. This process is described by the relationship between two variables, the shear rate and the shear stress.

# Shear Rate

Shear rate is usually designated by the Greek letter gamma with a dot above the letter  $\langle \hat{\gamma} \rangle$ . No matter what system of units is used, it is represented by 1/time (in seconds). That is 1/sec or s<sup>-1</sup>. The larger the number the faster is the material being sheared.

The shear rate (the true shear rate) at the wall of a die  $(\dot{\gamma}_w)$  is given by:

ý <sub>w</sub> = <u>(3n'+</u> 4n'	<u>1) (40)</u> πR³	(Eqn. 1)
where,	n' T <sub>w</sub> P R L	<ul> <li>= [dlog(PR/2L)]/[dlog(4Q/(πR<sup>3</sup>)]</li> <li>= shear stress at the wall of the die = PR/2L</li> <li>= measured pressure</li> <li>= die radius</li> <li>= die length</li> </ul>
	Ϋ́w Q	= shear rate at the wall of the die = volumetric output rate

By plotting shear stress (Pa or Nm $^{\rm 2}$ ) against shear rate (s $^{\rm 1}$ ) a flow curve may be obtained.

# **Apparent Shear Rate**

The expression for the shear rate at the wall of a tube or die is rather complex and in practice it is often simplified to give  $\dot{\gamma}_{wa} = 40/\pi R^3$ , where  $\dot{\gamma}_{wa}$  is the apparent (or uncorrected) shear rate at the wall of a die.

For Newtonian fluids, the apparent (or uncorrected) shear rate at the wall of a die ( $\dot{\gamma}_{w,a}$ ) is equal to the true shear rate ( $\dot{\gamma}_w$ ) However, with non-Newtonian fluids, such as polymer melts, the two are not equal but are related by the equation:

 $\dot{\gamma}_{w} = [(3n' + 1)/4n'] \dot{\gamma}_{w,a}$  (Eqn. 2)

where,  $n' = [dlog(R\Delta P/2L)]/[dlog(4Q/(\pi R^3)]]$ 

# Shear Stress

Shear stress is the stress or force applied to a body in the direction of one of its faces. In rheology, shear stress is that force that is applied to a material to cause shear flow. Thus, it may be defined as the stress (the force per unit area – F/A) required to make one layer of melt to flow over another at a specific rate. It has the dimensions of force per unit area and is usually designated by the Greek letter tau ( $\tau$ ).

 $\tau_{\rm w} = \text{PR/2L} \qquad (\text{Eqn. 3})$  The shear stress at the wall of the die (given the symbol  $\tau_{\rm w}$ ) and is

where, P = measured pressure

R = die radius, and L = die length

Shear stress has the units of force per unit area. 1 Nm<sup>2</sup> = 10 dyn cm<sup>2</sup> = 0.000 145 lbf in<sup>2</sup> (psi). By plotting shear stress (Pa or Nm<sup>2</sup>) against shear rate (sec<sup>-1</sup>) flow curves may be obtained. Dividing the shear stress by the shear rate gives the viscosity ( $\eta$ ) with units of (Pa-sec) or (Nsm<sup>2</sup>).

#### **Apparent Viscosity**

Viscosity is defined as the resistance to flow. By dividing  $\tau_w$  (the shear stress at the wall) by the apparent wall shear rate  $(\gamma_{w,a})$  one may obtain the viscosity  $(\eta_a)$  at a particular shear rate. This is the apparent viscosity. (In practice the word "apparent" is often omitted and the symbol  $\eta$  is used in place of  $(\eta_a)$ . Another form of viscosity, the kinematic viscosity is obtained by dividing the viscosity (the "coefficient of viscosity") of a fluid by its density. The units are  $m^2s^{-1}$ .

Dynamic viscosity is an alternative name for the viscosity or coefficient of viscosity of a fluid. The usual symbol is  $\eta$  but  $\mu$  is sometimes used. In the cgs system, the units are dyne sec cm<sup>2</sup> and such units may be referred to as "poise" (P). One poise = 1P =  $10^{-1}$  Pa-s =  $10^{-1}$  Nsm<sup>2</sup> = 0.000 014 5 s ft<sup>2</sup>. That is, one poise = 1P = 0.1 Pa-s = 0.1 Nsm<sup>2</sup> and 1 Pa-s = 1 Nsm<sup>2</sup> =  $1 \text{ kgm}^{1}\text{s}^{-1}$  = 10P = 0.020 88 lbf s ft<sup>2</sup> = 0.000 145 lbf s in<sup>2</sup>.

Units Used Property	Symbol	System of Measurement			
		cgs	fps	ips	S.I.
Shear stress	τ	dyn cm <sup>-2</sup>	lbf ft <sup>-2</sup>	lbf in <sup>-2</sup>	Nm <sup>-2</sup>
Shear rate	Ϋ́	S <sup>-1</sup>	S <sup>-1</sup>	S <sup>-1</sup>	S <sup>-1</sup>
Apparent viscosity	$\eta_a$	dyn s cm <sup>-2</sup>	lbf s ft-2	lbf s in-2	Pa-s

cgs. - abbreviation used for centimeter-gram-second.

fps. - abbreviation used for foot-pound-second.

ips. - abbreviation used for inch-pound-second.

SI. - abbreviation used for Système International d'Unitè.

#### **Flow Curve**

.. .. .. .

Polymer melts are not Newtonian, but pseudoplastic fluids (i.e., the viscosity decreases with increasing shear rate or stress.). Since there is not a linear relationship between pressure and flow, the flow properties cannot be represented meaningfully by a single number. Therefore, flow testing over a range of conditions is required. The information so obtained is usually presented in the form of a series of flow curves where each one represents data obtained at a particular temperature. **Figure 8a.** shows individual flow curves for a number of commodity resins while **Figure 8b.** shows similar curves for some engineering polymers.

A flow curve is sometimes referred to as a rheogram. It is generally a plot of apparent shear stress ( $\tau_{w,a}$ ) against apparent shear rate at the wall of a die ( $\dot{\gamma}_{w,a}$ ). This is done because the expressions for the true shear stress and shear rate at the wall of a die are rather cumbersome. In practice it is often just as useful to use the much simpler expressions  $\tau_{w,a} = R\Delta P/2L$  and  $\dot{\gamma}_{w,a} = 4Q/\pi R^3$ . It may be shown that, as with the true shear rate, there is a true shear stress at the wall of the capillary. To obtain this from capillary rheometer measurements one must correct for the pressure drop caused by the polymer entering the die. (This is called the Bagley correction.) Flow curves generated in most industrial laboratories are, however, generally obtained by plotting  $\tau_{w,a}$  against  $\dot{\gamma}_{w,a}$ .



Figure 8a. Viscosity Curves for Some Commodity Thermoplastics



Figure 8b. Viscosity Curves for Some Engineering Thermoplastics

By plotting shear stress in Pa ( $Nm^2$ ) against shear rate in s<sup>-1</sup>, a viscosity in Pa-s ( $Nsm^2$ ) may be obtained by from the slope of the flow curve. The shear stress is generally plotted on the vertical axis and the shear rate plotted on the horizontal axis. Conversion from shear rate to shear stress is also obtained for plastic melts by reading from such a flow curve. Flow curves are used because the equations, used to describe the flow behavior of pseudoplastic fluids, can be imprecise. Log-log plots are generally use to easily read the curves over several decades of shear rate.

Although the wall of the die is specified in the measurement, this is simply a place at which the shear stress and shear rate may be calculated from knowledge of flow rates and pressures. The relationship between  $\tau_w$  and  $\dot{\gamma}_w$  will, however, be independent of the position in the flow system and indeed will be equally applicable to flow in other geometries such as slits.

# ► FLOW PROPERTIES MEASUREMENT

The information necessary to generate a flow curve may be obtained from a variety of types of machines. These include cone and plate and parallel plate rheometers, co-axial cylinder viscometers and capillary rheometers. Because of the similarity of its operation to that of many conversion processes and its ability to generate rheological information at processing conditions, the capillary rheometer is possibly the most useful of these instruments.

# **Capillary Rheometer**

A capillary rheometer (Figure 9.) is a test instrument that is used to measure the rheological properties of polymer melts. It does so by using a piston to forcing polymer melt, from a reservoir, through a capillary die. This type of rheometer is of greatest interest to the plastic technologist as it, in many respects, is similar to injection molding and extrusion machinery and provides data in the shear rate range used in these processes (in fact, early extruders and injection molding machines used rams rather than screws to move the molten resin). The data so obtained may in turn be used, for example, to size dies or runner systems. A capillary rheometer, when used in the shear rate range seen in melt processing, may sometimes be called a high shear rate rheometer.

The analysis of shear flows in a capillary rheometer employs some important assumptions. Of these the most important are:

- 1. The flow is isothermal.
- 2. There is no slip on the die wall.
- 3. The melt is incompressible.
- 4. The flow pattern is the same throughout the die.

5. Dissipation of energy at the die entrance, or due to chain uncoiling, is negligible.

# Measuring $\dot{\gamma}_{\text{w,a}}$ and $\tau_{\text{w}}$

When the above assumptions are considered to be valid, then the shear stress and shear rate may be calculated as follows. If the piston speed of the rheometer is s (measured in mm/s) and the barrel diameter is D (measured in mm), then the volumetric flow rate Q is

#### $Q = \pi SD^{3}/4 \qquad (Eqn. 4)$

This is the same in both the barrel and the capillary. The apparent shear rate at wall is given by

 $\dot{\gamma}_{w,a} = 4Q/\pi R^3$  (Eqn. 5)

Where R is the radius of the capillary in mm.

Combining the above equations and simplifying,

$$\dot{\gamma}_{w,a} = SD^{3}/R^{3} s^{-1}$$
 (Eqn. 6)

If the barrel diameter is 9.550 mm (the standard size) then, the formula becomes

#### $\dot{\gamma}_{w,a}$ = 91.203S/R<sup>3</sup> s<sup>-1</sup> (Eqn. 7)

(when the capillary radius is in mm and the ram speed is in mm/s).

Since the shear stress at the wall is given by  $\tau_{\rm w}$  = PR/2L then, for a 20:1 die,  $\tau_{\rm w}$  = P/80

(where P is the recorded pressure at the entrance to the capillary). By plotting  $\tau_w$  against  $\dot{\gamma}_w$ ,a a flow curve may be obtained.



Figure 9. Capillary Rheometer

#### **Flow Data**

Below are shear flow data obtained from a capillary rheometer. Such information is available in several material databases, such as those maintained by Moldflow Inc. These data are used, for example, to help predict how an injection mold will fill. For this application the effect, on the viscosity, of changing shear rate at a constant temperature is required. In addition, the effect of changing temperature a constant shear rate is required. A die with a high L/D ratio, for example 20/1, is generally used.

The viscosities (in units of Pa-s ) at 1,000 s<sup>-1</sup> for the following grades of PA 6 (Akulon from Akzo) at a range of temperatures are:

Grade	240°C/464°F	260°C/500°F	280°C/536°F	300°C/572°F
Easy flow	98	78	63	50
Medium flow	149	115	89	69
Stiff flow	394	319	259	210

The viscosities (Pas) at a temperature of 240°C/464°F for a range of shear rates (s<sup>-1</sup>) are:

	100s <sup>-1</sup>	1,000s <sup>-1</sup>	<b>10,000s</b> -1	100,000s <sup>.1</sup>
Easy flow	197	63	20	6
Medium flow	323	89	25	7
Stiff flow	1220	259	55	12

#### **Ease of Flow**

The data in the above table were obtained when samples of nylon 6 (PA 6) were tested. They clearly show that the three injection molding grades tested have very different viscosities. The first grade, which has the lowest viscosity, (it is the easiest flowing grade) can be used where mold filling is difficult, or where long flow lengths are involved. The viscosity of all three materials falls as the shear rate is increased. Thus mold filling becomes easier. Raising the melt temperature, while keeping the shear rate constant, reduces the amount of injection pressure required to maintain a certain rate of flow. This is because most plastic melts become less viscous (flow more easily) with increasing temperature.

Such information is frequently presented in tabular form in the "Flow Properties" section for a material. Although the viscosity of most plastics falls as the temperature is raised, with some materials the effect is more pronounced. Thus, for materials whose viscosity falls markedly with a rise in temperature (for example, PA 66, PC and POM) the ease of mold filling is helped significantly by a moderate increase in melt temperature. For materials where the effect of temperature increases is less pronounced, it is more useful to increase the mold temperatures and/or the molding pressures to ease the filling of a mold.

#### **Flow Property Assessment**

Because most methods of shaping plastics are melt processes, such as extrusion or injection molding, the measurement of melt flow properties is extremely important. A large number of tests have been devised, many of them empirical, to measure these properties. Such tests may be roughly grouped into low shear rate tests and high shear rate tests. A low shear rate test, such as the melt flow rate (MFR) (or melt flow index (MFI)) test, is one of the most widely used tests (See the Section 3 on Testing).

# > ELASTIC EFFECTS IN POLYMER MELTS

The long chain molecules that make up polymeric materials become distorted when they are subjected to shearing forces during processing operations. Such shearing tends to stretch the molecules. However, when the shearing process ceases the molecules tend to coil up again, providing they are still molten. If cooling occurs rapidly after shear, this re-coiling may not be complete. Such un-coiling/re-coiling processes can give rise to a number of effects, which are often referred to as elastic effects. The most important elastic effects are die swell, melt fracture, sharkskin, frozen-in orientation and draw down.

# ► DIE SWELL

When polymer melt emerges from a die the extrudate may swell, so that its cross-section as it leaves the die is greater than that of the die orifice. For a capillary die, the ratio of the extrudate diameter to the die diameter is known variously as the die swell ratio, swelling ratio or as the puff-up ratio. For a slit die, the relevant ratio is thickness of extrudate/depth of slit.

#### **Causes of Die Swell**

Die swell (Figure 10.) occurs because shearing of the melt as it passes through the die causes the molecules to become extended (with the greatest
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orientation near the die wall). On emergence from the die, the molecules tend to coil up (recoil) resulting in a contraction in the flow direction and an expansion in directions perpendicular to the flow. If an extrudate is cut at the die face, it will be seen that the leading edge of the extrudate is convex. This indicates that the greatest contraction in the flow direction is nearest to the wall where the shear has been greatest.



Figure 10. Die Swell

## **Reducing Die Swell**

For a given material, die swell may be reduced by:

- Decreasing the extrusion rate
- Increasing melt temperature
- Increasing the length of the die parallel or lands
- Streamlining and/or reducing the die entry angle
- · Increasing the draw down ratio

Often the addition of a lubricant will reduce the die swell. The lubricant will cause slip at the wall and ease the flow of polymer through the die. Low molecular weight polymer functions as a lubricant and so broadening the molecular weight distribution of a material will also reduce die swell.

## **Changing Shape**

It is common industrial practice to compensate for die swell by stretching, or drawing down, the extrudate so that it can just pass through a sizing die. When this technique is used it is not necessary that the two be exactly balanced. It should, however, be noted that drawing down causes molecular orientation, which gives rise to an increase in strength in the flow direction and a decrease in directions transverse to the flow. This may or may not be desirable. The method is also somewhat limited where the solid extrudate has varying section thickness. Shear rates, and hence die swell, will be higher at the thinner sections. These thin sections may also have a shorter die parallel, to 32 ensure that extrusion rates are constant across the cross-section, and this will increase the die swell even further.

In the production of pipe and tubing, the situation is further complicated by the fact that the extrudate is usually inflated to the dimensions of a sizing die. In this case it may be assumed that on emergence from the die, the wall thickness will expand appropriate to the die swell for the shear rate used. Subsequently, the wall thickness will be reduced proportionally to the amount of inflation given by the ratio diameter of the sizing die/ the external diameter of the pipe die.

### **Die Entry Angle**

The die entry angle is the angle of convergence provided for the melt entering the extrusion die. As the melt leaves the extruder barrel the shape of the flowing melt stream must be changed to that of the shaped extrudate. This transition takes place in the die adapter and is best done as gradually and as streamlined as possible. The higher the viscosity of the melt, the more streamlining is required. It is also necessary to keep the lengths of the flow paths, to each part of the die, the same in the streamlined region, as this will reduce subsequent extrudate shape changes.

The die entry angle for a particular material can be found practically by using a plate die, which has a properly shaped die orifice, in a nonstreamlined die holder. Plastic is then run through the die, followed by a change in the color of the material. When the second color starts to extrude from the die, the machine is shut down. The hot die and the die holder are removed and cooled and the cross section of the cold material is examined. The color changes seen in the cross section will show the streamlined shape required. A thermoplastic material, which contains a heat fugitive dye, may also be used to show the streamlining required.

#### **Predicting Die Swell**

Computer programs are available that simulate the flow of polymeric materials through a die and will predict die swell. This type of program takes die swell into account in predicting the die shape and size required to produce a desired profile shape and size. To do this, the program incorporates a materials database that contains the data (thermal, rheological and viscoelastic properties) needed to do a flow analysis. Such software allows the design of a die that is balanced to produce uniform flow. Methods for the measurement of die swell are given in a later section.

## MELT FRACTURE

It is frequently observed that when extrusion is carried out at high rates, a distortion of the extrudate may occur. The distortion may be due either to the phenomenon known as melt fracture (also known as elastic turbulence or bambooing) (Figure 11.) or, it may be due to sharkskin. Although not fully understood, these do appear to have different origins.

#### **Critical Shear Rate**

Melt fracture occurs when the shear rate in a die exceeds a critical value for the polymer melt at a particular temperature (the critical shear rate). There is also a critical shear stress. The point on the shear rate-shear stress diagram (flow curve), corresponding to these two values is known as the critical point. Melt fracture is believed to occur in the die entry region where material is being funneled from the melt reservoir into the capillary or die. In a full-scale extruder, this would correspond to the point where melt moves into the die parallel portion of the die. Some further complicating effects may occur at the wall of the die.



#### Figure 11. Melt Fracture

#### **Form of Distortion**

The form of the melt fracture distortion varies from one polymer type to another, but it is generally helical in nature. With materials such as polyethylene and polypropylene a distortion like a screw thread may appear, while with polystyrene the extrudate may form a spiral. Other polymer melts may show ripples, or bamboo-like repetitive kinks. With all melts, the helical nature becomes obscured by severe distortion that looks random, at rates well above 33

34 the critical point. Melt fracture is most likely to occur where small diameter extrudates are being extruded at high rates. The most notable example occurs with wire covering.

## **Factors Affecting Melt Fracture**

Since it is easily observed in the laboratory, melt fracture has been widely studied and experiments have shown that:

- The critical shear rate  $(\dot{\gamma}_{c})$  increases with increasing temperature
- The product  $\tau_w M_w$  is a constant. Melt fracture will start at lower shear stresses and shear rates as the molecular weight of the material is increased ( $\tau_w$  is critical shear stress and  $M_w$  is weight average molecular weight)
- Two polymers differing in their levels of branching, but which have similar melt viscosities tend to have similar critical points
- The extrudate quality may be markedly improved by tapering the die entry. When this is done externally, undistorted extrudates may be obtained at rates well above the critical point. However, there is some evidence that there may still be some internal distortion
- There is some evidence that increasing the length to diameter (L/D) ratio of the die parallel increases the critical shear rate. Tapering the die parallel may also substantially increase the critical point

The above factors have now been well known for some years and as a result high speed wire covering, and other operations involving high shear rates are operated without undue trouble from melt fracture effects.

## ≻ SHARKSKIN

The phenomenon of sharkskin has been less widely studied than melt fracture, but it is probably a greater problem in industrial extrusion.

## **Melt Tearing**

In the case of sharkskin, the distortion consists of transverse ridges (as opposed to helical distortions). It is believed to occur as a result of the melt tearing as it extrudes from the die. This occurs because, within the die, melt close to the wall is moving very slowly (in the case of the layer next to the wall this movement is zero). As the melt emerges, the whole extrudate moves away from the die face at a constant speed so that the outer layers are suddenly stretched and may tear.

## Incidence of Sharkskin

Experimental studies indicate that the critical shear rate for onset of sharkskin  $(\dot{\gamma}_{c.sh})$  is inversely proportional to the die radius (R); that is,  $\dot{\gamma}_{c.sh} R = a$  constant. This means that the critical shear rate is much lower with larger diameter dies. One result of this is that the small dies (such as those used in typical laboratory rheometers) generally show melt fracture occurring at shear rates below those for the onset of sharkskin, while the reverse may be seen with typical full-scale industrial dies.

## **Critical Linear Extrusion Rate**

It can be shown, as a consequence of the above relationship, that sharkskin is likely to occur above a critical linear extrusion rate irrespective of the die size.

For example, for a particular hypothetical polymer melt, it may occur at an extrusion rate of approximately 30 in./min whatever the die size.

#### **Conditions Favoring Sharkskin**

Sharkskin is most pronounced when the melt is partially elastic and has the consistency of a friable cheese. Improved results may sometimes be obtained by reducing the melt temperatures, so that the melt is more strongly elastic as it emerges from the die. Alternatively, some improvement has been seen when the die is heated at the point of exit. This makes the surface layers of melt more fluid, thus making melt tearing less likely. The severity of sharkskin may vary enormously. At one extreme, the distance between a ridge and adjacent trough may be 1/3 of the extrudate cross-section. At the other extreme, the effect may be barely detectable to the naked eye, but shows up as a matt finish or, may be felt by running a fingernail over the surface. In blow molding, a rough surface on the inside of a bottle is indicative of sharkskin (The outside is usually flattened against the wall of the blow mold).

#### **Molecular Factors**

The only molecular factor within a polymer type that appears to greatly influence sharkskin is molecular weight distribution (MWD). A broad distribution is generally effective in reducing any tendency to show sharkskin effects.

## ► FROZEN-IN ORIENTATION

In the molten state, and when not subject to external stresses, polymer molecules tend to coil up. That is, they prefer to exist in a random coil configuration. When external stresses are applied (as occur during extrusion, molding and other shaping operations) the molecules become distorted from their randomly coiled state and tend to become oriented. In most processing operations it is generally desirable to "set" the polymer as soon as possible after it has been shaped; usually by cooling in a water bath after the extrusion die. In such circumstances, the polymer molecules may not have time to coil up (recoil) completely before the melt freezes. This results in the effect known as frozen-in orientation.

## Anisotropy

As a result of frozen-in orientation, plastic products may be anisotropic in their behavior. That is, their properties differ if measured in different directions. For example, tensile strength is greatest in the direction of orientation. Impact strength is also affected by frozen-in orientation. When the sample has been injection molded with the gate at one end of the sample, the molecules will be roughly aligned with the long axis of the sample. Thus to break the sample, in a standard lzod test, requires fracture across the elongated molecules, which gives a higher impact strength is measured by dropping a weight onto a flat plate, lower impact strength will be seen with more oriented moldings. In this case fracture can occur more easily parallel to the direction of orientation since this largely requires fracture between, rather than across, molecules.

## <sup>36</sup> Biaxial Orientation

Increased orientation may be introduced into a system by stretching the polymer melt just before it freezes. Uniaxial orientation, that is stretching in one direction, is important in the manufacture of tapes and fibers, while biaxial orientation, simultaneous stretching in two directions, is important in film production. It may also be desirable to build-in biaxial orientation into piping, bottles, and other hollow containers, in order to enhance the hoop strength and fracture resistance of these products.

## Desirable or Undesirable?

Frozen-in orientation may thus be either desirable or undesirable according to circumstances. It is greater when the melt is subjected to higher stresses and when there is a short interval between shearing and freezing (or setting) of the melt. Such conditions are obtained with low melt temperatures and low cooling temperatures after shaping, as found with low injection mold temperatures or low extrusion cooling bath temperatures.

## ► DRAW DOWN

In a number of extrusion processes, such as the manufacture of film, the extrudate may be subject to extensive stretching after leaving the die. In other cases, like chill-roll casting of film, it is important that the extruded web does not tear on stretching.

## **Viscous Behavior**

In cases where stretching is important, viscous rather than elastic behavior is often more important. It is reasonable to expect that, although the melt should have some strength and elasticity, the main requirement is that the molecules of the melt can flow past each other.

## Neck-In

One phenomenon associated with chill roll casting is that of neck-in. The edge of the extruded web tends to shrink inward towards the center of the web and, at the same time, tends to become thicker than the bulk of the film. It is found here that more elastic melts, which can maintain a tension in the extrusion direction, are less susceptible to neck-in.

## Parison Sag

Elastic effects may also influence the parison sag that occurs during blow molding. Parison sag is the thinning of the parison caused by the stress of its own weight as it leaves the die. Part of the sag may be due to an elastic effect (chain uncoiling) and part due to viscous flow as the molecules slide past each other. It is reasonable to assume that the elastic component of the sag increases as a proportion of the total as the:

- Molecular weight, and hence viscosity, increases
- · Melt temperature decreases (increasing viscosity)
- The length of parison per unit weight increases. This is because an elastic deformation under a standard load depends on the length of the part being stretched, whereas the viscous flow does not (as long as the weight of the parison is constant).

## SECTION 3: TESTING

TESTING AND STANDARDS

MATERIAL INSPECTION

- DENSITY AND DIMENSIONS
- ➤ TENSILE STRENGTH
- FLEXURAL PROPERTIES
- IMPACT STRENGTH
- HARDNESS AND SOFTNESS
- > THERMAL PROPERTIES
- FLAMMABILITY TESTING
- ► MELT FLOW RATE
- ➤ MELT VISCOSITY
- MEASUREMENT OF ELASTIC EFFECTS
- CHEMICAL RESISTANCE
- > ELECTRICAL PROPERTIES
- > OPTICAL PROPERTIES
- MATERIAL IDENTIFICATION

## ➤ TESTING AND STANDARDS

The word test means an examination performed to evaluate performance or capability of a material, a compound, a test piece or a component. A test may be performed on a raw material (in powder or granular form), a piece specifically made for testing, or a formed part.

## **Reasons for Testing**

Testing is often time consuming and expensive, so, before considering specific tests, the purpose or object of the testing in general has to be decided. Testing is nothing new. Over the years, many tests have been devised to enable judgements to be made on the:

- Quality and consistency of raw materials
- Quality and consistency of associated products
- Merits of new or modified materials
- Suitability of a particular design

The need to carry out adequate fitness-for-purpose, and quality control testing, has increased greatly in recent years as a result of legislation on liability for product performance. New legislation is making the risk of punitive damage claims for product liability a very real possibility that manufacturers and suppliers must face and guard against. They must ensure that their materials and products are not in any way defective.

## <sup>38</sup> Standards Organizations

Tests are performed as specified by a test standards **(Table 3.)** which are issued by national or international standards organization (see separate Appendix). An example of a national standards organization is the American Society for Testing of Materials (ASTM). The International Standards Organization (ISO) is an international organization.

It should be noted that the same answers may not automatically be obtained if a material/test piece is tested by one standard and then by another, for example, by an American Society for Testing and Materials (ASTM) method and then by an ISO method. Differences in the reported values are not necessarily due to changes in the material, but may be due to differences in test piece preparation, dimensions, conditioning or to changes in the actual test method used.

## **Factors Influencing Test Results**

There are a large number of factors that will influence the results obtained from a test such as test machine design, test piece size, shape and method of production, conditioning of the samples, temperature during the test, etc. Standardization of test methods is essential, if people in the same laboratory or in different laboratories, are to obtain the same result on the same material. Since there are a large number of factors that will influence the results obtained, it is obvious that standardization of test methods is essential.

Standard test sample conditioning must be employed. Often this conditioning is simply storing the specimens at a standard temperature, for a specified time, before testing. Despite its simplicity, such physical aging is important as post-molding changes commonly occur. For many materials, such as some engineering thermoplastics, the specimens should be stored for a specified time under conditions of specified humidity.

## Test Results and SI

Most test results are measured and quoted in the units specified by the Système International d'Unité (SI). The formal content of SI is determined and authorized by the General Conference of Weights and Measures. The units issued by the SI are a coherent international system of units now widely used for scientific and technical work. As there is coherence between units, this removes awkward factors in calculations involving quantitative relations.

The seven basic units are the meter (length), kilogram (mass), second (time), ampere (electric current), degrees Kelvin (temperature), mole (amount of substance) and the candela (luminous intensity). There are supplementary units for plane angle (radian) and solid angle (steradian) and some SI derived units that have special symbols. These SI derived units that have special symbols include the Newton and the Pascal. Certain other non-SI units are permitted, for example, cm and °C. SI units were derived from the MKS system and are sometimes occasionally known as MKSA metric.

## Families of Materials

Each of the many plastics and rubbers available is, in effect, a family of materials that differ in such factors as molecular weight and molecular weight distribution. What this means is that it is possible to get a wide range of properties for each of the materials groups. Properties quoted in the literature should, therefore, only be used as a general guideline as, for example, where the exact composition or structure of material tested is not known. The properties of plastics may also be dramatically changed by the processing conditions employed and by the use of additives. With many materials, variations of basic formulations are available using additives to provide for improved properties such as, heat resistance or, weatherability. Some formulations offer improved impact strength while others, which contain fillers, are used where the moldings require tensile strength or or higher heat distortion temperature. Processing and performance modifiers such as anti-static or nucleating agents may be added. These additives may form part of the color masterbatch.

#### **Restriction of Testing**

Most of the tests described here, although standardized, are usually only suitable for relatively simple purposes such as for checking the quality of a material being produced. Even so, such testing is expensive and it is therefore important to restrict it as much as possible. Unnecessary testing, on materials or products, should not be performed. (For example, the testing of gloss, or finish, on parts, which are to be hidden in service, would seem to be pointless.) Before a test is performed it must always be decided what will be done, with the results and with the components, after testing. It is very important that test specifications are drawn up at a very early stage in the production process. The use of written specifications and procedures is vital. Once production has started, periodic reviews of the tests are essential to ensure that the test programs continue to provide what is required.

#### Table 3. Standard Tests Used for Property Guidelines

	ASTM	ISO	BS	DIN	Other
<b>Conditioning and Test Sai</b>	mple Prepar	ation			
Cond. of materials		291	2782 Pt10		
Test piece preparation			2782 Pt9		
Cond. of test pieces			2782		
Density & Dimensions					
Density/rel. density	D 792	1183	2782 Pt6	53479	
Dimensions			2782 Pt6		
Apparent bulk density			2782 Pt6		
Bulk factor			2782 Pt6		
Short Term Mechanical Te	ests				
Tensile strength	D 638M	R527	2782 Pt3	53455/7	
Elongation at break	D 638M	R527	2782 Pt3	53455/7	
Tensile yield strength	D 638M	R527	2782 Pt3	53455/7	
Tensile modulus	D 638M	R527	2782 Pt3	53455/7	
Specific modulus	See Tensile	strength a	nd Density/rel	. density	
Compressive strength	D 695	604	2782 Pt3	53454	
Compressive modulus	D 695	604	2782 Pt3	53454	
Flexural strength	D 790	178	2782 Pt3	53452	
Flexural modulus	D 790	178	2782 Pt3	53452	
Izod impact strength	D 256	180	2782 Pt3		
Charpy impact strength	D 256	179	2782 Pt3	53453	
Tensile impact strength	D 1822		2782 Pt3	53448	
FWIS	D 3029		2782 Pt3		
<b>Mechanical Surface Tests</b>	;				
Abrasion resistance	D 1242				
Taber abrasion res.	D 1044	3537	2782 Pt3	52347	
Friction	D 1894		2782 Pt8		
Ball hardness		2039-1	2782 Pt3	53456	
Rockwell hardness	D 785	2039-2	2782 Pt3		
Shore hardness	D 2240	868	2782 Pt3	53505	
Scratch hardness					
Softness			2782 Pt3		

Thermal and Temperature	ASTM Dependent	ISO Properties	BS	DIN	Other
Thermal conductivity	C 177				
Coeff. of thermal exp.	E 228 or 83	1		53752	
Shrinkage - linear	D 955	294-4	2782 Pt9		
Specific heat	C 351				
Glass trans. temp T <sub>g</sub>	D 3418				
Melting point - T <sub>m</sub>	D 3418	3148	2782 Pt1		
Softening point			2782 Pt1		
Vicat softening temp	D 1525	306	2782 Pt1	53460	
HDT 1.82 MPa/264 psi	D 648	75	2782 Pt1	53461	
HDT 0.45 MPa/66 psi	D 648	75	2782 Pt1	53461	
Relative temperature index				UL 746B	
Cold flex			2782 Pt1		
MFR/MFI	D 1238	1133	2782 Pt7	53735	JIS K/210
Melt viscosity	T			54811	
Flammability and Smoke			0700 041		
	D 635		2782 PU		UL94V/ CSA
Glow wire		6458			IEC 605-
		0430			2_1
Needle flame		6458			IFC 695-
		0.00			2-2
Limiting oxygen index	D 2863	4589	2782 Pt1		
Smoke testing	D 2843				
Permanence Tests					
Water abs. in 24 h	D 570	62	2782 Pt4		
Saturation water abs	D 570	62	2782 Pt4		
Chemical resistance	D 543		2782 Pt8		
ESC		4600	2782 Pt8		IEC 538A
Ageing	G 26/G 53				
Electrical properties					
Volume resistivity	D 257	6233			IEC 93
Surface resistivity	D 257	6233			IEC 93
Relative permittivity	D 150	2067/4542	2		IEC 250
Dissipation factor	D 150	2067/4542	2		IEC 250
Dielectric strength	D 149	2918			IEC 243-1
Arc resistance	D 495				.=
CTI		5901			IEC 112
Optical Properties	D 5 40		0700 D.F	50.404	
Refractive index	D 542		2782 Pt5	53491	
Haze	D 1003		2/02 Pt5	07500	
GIOSS	D 523		2782 Pt5	0/530	
Croop				52444	
Uleep Eatigue	D 2990			03444	
raugue	U 0/ I				

## ► MATERIAL INSPECTION

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Most thermoplastic materials are supplied as granules or pellets in bags, sacks, or bulk containers. When supplied in 50 lb/22.6 kg sacks, the material is often supplied in 20 bag (1,000 lb) loads. If supplied in 25 kg/55 lb bags then, the material is often supplied in 1,000 kg/2240 lb lots. Considerable discounts may be obtained if the plastic material is purchased in bulk and supplied in bulk containers. With some materials, this form of supply may not be acceptable in such cases as when excessive water absorption may occur.

## **Preliminary Inspection**

Most plastic materials are accepted as being satisfactory until something goes wrong during a processing operation. It makes sense, however, to inspect the material before processing for color, particle size, particle size consistency and any obvious contamination. There is no point in processing 'outof spec' material as 'out-of spec' products will be the result. This applies particularly to recycled material. The feed to many extrusion processes is a mixture of virgin and regrind or recycled material.

A major problem with recycled or reclaimed material is contamination (which may take the form of discolored material and/or fines). Therefore, a sample of the material should be inspected under a consistent light source for color and contamination and the regrind should be sieved to check the concentration of fines. Only that material with a specified concentration of fines should be used. The moisture content of the material should be checked and the material dried if necessary. Blend the dried, reclaimed material with virgin material in a precisely agreed to ratio and check appropriate properties (for example, flow rate and color) before the material is certified as being good enough for processing. If there is any uncertainty in regard to the quality of the material being fed to the machine, then it should not be used.

## **Moisture Absorption**

Moisture absorption is a measure of how much water (moisture) a component, or material, will absorb under specified conditions and over a certain time. Many plastic materials absorb water (that is, they are hygroscopic). This absorbed water changes dimensions and properties including electrical insulation, dielectric properties, mechanical properties and surface appearance. The melt flow will also be affected.

In general, the level of water/moisture in the material fed to melt processing equipment must be kept very low as its presence in many polymers may lead not only to problems with processing, but also to post-extrusion problems and to chemical degradation. For instance with PC, the moisture causes chemical degradation that is detrimental to the performance of the finished product (impact strength is affected). The maximum permissible limit for moisture in the granules for production of good extrudates varies from polymer to polymer but, in general, should be kept below 0.1% as measured by the water absorption in 24 hours.

## Water Absorption in 24 Hours (24 h)

Test pieces are generally molded discs 2 inches/50.8 mm in diameter and 0.32 mm thick. Three test pieces are used. Each test piece is conditioned by being heated at  $50^{\circ}$ C/122°F for 24 hrs. The test pieces are cooled, in a desiccator, and weighed. The test pieces are then immersed in distilled water at a specified temperature and for a specified time. For example, the test pieces are placed in a container with water at  $23^{\circ}$ C/73°C: after 24 h the test pieces are wiped dry and weighed. The change in weight is usually expressed as a percentage of the original weight, for example, 0.1%. Such a test gives the 24 hr. water absorption at room temperature. In general, if the 24 hr. water absorption is greater than 0.2% then, for injection molding, drying is usually necessary. For extrusion-based processes, the level is approximately 0.1%.

An accelerated water absorption test may be performed by immersing the weighed, dried, test pieces in boiling water for 30 minutes, cooling for 15 minutes at  $23^{\circ}$ C/73°F and re-weighing.

## <sup>42</sup> Saturation Water Absorption

A saturation water absorption value is also quoted for many materials. This value is obtained by immersing the weighed and dried test pieces in water at  $23^{\circ}$ C/73°F until there is no further increase in weight upon re-weighing.

For some materials such as nylon 6, that have a very high water absorption, graphs are published by the material manufacturers that indicate how the water content varies with humidity. This may be referred to as an equilibrium water content.

## **Measuring Moisture Content**

Methods of measuring of the moisture content in a thermoplastic material include drying to constant weight and the Karl Fischer method. The Karl Fischer method, however, is time consuming, involves the use of pyridine (an unpleasant smelling liquid) and requires considerable technical skill. Drying to constant weight is also time consuming and often not satisfactory where very low levels of moisture need to be measured.

Consequently proprietary instruments, which require little skill and which give a direct reading of moisture content in less than 30 minutes have been developed. An example is the OmniMark moisture analyzer. A separate weighing facility as well as the instrument is required. Ideally such an instrument should be sited close to the processing operation, rather than in a remote quality control laboratory, so that the moisture content of the material being processed may be readily checked. See **Table 4.** below for recommended moisture limits for extrusion processes.

## Table 4. Moisture Content Limits for Extrusion

Moisture Content Limit %
0.05
0.02
0.40
0.25
0.01 - 0.10
0.02
0.10
0.08
0.10
0.01

## DENSITY AND DIMENSIONS

Most tests involve the measurement of one or more dimensions. The density of a material is of importance since materials are usually bought by weight and sold by volume. Apparent density measurements may be performed on material before an extrusion operation, as a quality control check. Melt density can be measured during a melt flow rate test (See Method A to B Conversion in Flow Rate Testing).

## Units of Density and Specific Gravity

Density is defined as mass per unit volume. When expressed in grams per cubic centimeter (g/cc) it is numerically equal to relative density (specific

gravity or SG). It is often given in kg/m<sup>3</sup> (kilogram per cubic meter) although grams per cubic centimeter is more common (g/cm<sup>3</sup> is the same as g/cc and gcm<sup>3</sup>). It is suggested, for ease of understanding that when density is discussed, the units used should be Mg/m<sup>3</sup> rather than kg/m<sup>3</sup>. This gives values that have the same numerical values as the well established g/cm<sup>3</sup> values or SG values. 1 Mg/m<sup>3</sup> = 1 g/cm<sup>3</sup> = 1,000 kg/m<sup>3</sup>.

Relative density is more usually referred to as specific gravity (SG). Relative density (rd or RD) is the ratio of the mass of a substance to the mass of an equal volume of water, at a specified temperature. Specific gravity or relative density has no units, as it is a ratio.

#### **Density of Filled Materials**

The densities of most commercial thermoplastics are low since they are generally based on carbon and hydrogen. Addition of inorganic fillers and fibers usually increases the density of the polymer blends since these materials usually have a density greater than that of the polymer. To calculate the density of a polymer composition, divide the total mass of the polymer composition by the total volume. For example, if 100 g of unsaturated polyester resin (UP) of density 1.28 g/cc is mixed with 50 g of glass filler (density 2.55 g/cc) then the

#### Compound Density = 100g + 50g / [100g/1.28g/cc + 50g/2.55g/cc] = 1.52 g/cc. (Eqn. 8)

This equation assumes that all the air spaces, or voids, in the mixture are filled.

#### **Measurement of Density and Specific Gravity**

Relative density (RD), more commonly known as specific gravity (SG), may be obtained by dividing the weight of a sample in air by the difference between the weight in air and the weight in water. Special balances (such as the L'homme and Argy) are available for direct measurement of relative density (RD or SG) based on this principle. A measurement of RD can be used to determine if opaque test pieces contain voids.

If an accurate measurement of density is required, then this may be obtained from a flotation method. For example, the density of polyethylene (PE) may be obtained by the use of a water/alcohol mixture. Small pieces of the PE are placed in a beaker which contains a water/alcohol mixture with a density of approximately 0.9 gcm<sup>3</sup> (obtained by mixing 35 ml of methanol with 25 ml of distilled water at 23°C). Additional water is then slowly added to the beaker. When the density of the liquid is the same as the density of the PE, then the pieces of PE will remain suspended. It is important to add the additional water slowly and to keep the solution well stirred. These precautions ensure that the liquid density is uniform and that the heat of mixing does not cause the temperature to rise to an unacceptable level. The density of the water/alcohol mixture may then be determined by a weighing bottle technique.

A density gradient column may also be used for measuring density. The density of such a column gradually changes from top to bottom, so a small piece of a material dropped into it will come to rest at the point where its density matches that of the column. Glass floats of known density are used to calibrate the column. Some liquid mixtures used in such a column include water and ethanol (density range 0.8 to 1.0), ethanol and carbon tetrachloride (density range 0.8 to 1.6), water and calcium nitrate (density range 1.0 to 1.6) and, carbon tetrachloride and bromoform (density range 1.6 to 2.9). For foamed plastics, one can weigh a known volume of the material. In this case, a cube of the material is cut and weighed: the density is weight over volume.

## Melt Density

Sometimes a measure of melt density is required. This is most easily measured on an apparatus used for measuring Melt Flow Rate by what is called Method B. Method B uses an electronic method to sense the plunger displacement and thus the volume of polymer displaced. Some machines can calculate the flow data from such measurements at the same time. **(See Flow Rate)** It should be noted that, for polymers, the melt density is always lower than the solid density.

## Dimensions

Many tests require that the dimensions of the test piece are known and are known very accurately. The measurement of dimensions, such as length, width, and thickness of rigid materials is relatively easy to do and may be done with a simple external micrometer. Spring-loaded dial gauges are commonly used for soft or flexible materials and electronic non-contacting gauges are also available.

Gravimetric thickness is sometimes used, for example, to measure the thickness of an embossed component. This is a method of obtaining the thickness of a component from a determination of dimensions, mass and relative density. The gravimetric thickness in millimeters (mm) equals 10.0 W/A x RD: where A is the area, W is the mass and RD is the relative density. The gravimetric thickness in inches (in.) equals 0.394 W/A x RD: where A is the area, W is the mass and RD is the relative density.

## ➤ TENSILE STRENGTH

Tensile testing is a very widely used test as it is relatively easy to perform, gives reasonably reproducible results and yields a great deal of information. From this one test one can obtain not only tensile strength, but also elongation and modulus. The same basic machine may also be used to perform a number of other tests (for instance, flexural strength and compressive strength) with relatively simple modifications.

## **Description of the Test**

The tensile test machine is constructed so that as the test specimen is deformed at a pre-selected speed the resistance to deformation and the amount of extension are measured. In order to ensure that breaking does not occur at the grips, used to hold the test sample, the sample is usually dumbbell shaped. If a cut sample is used (for example, cut from sheet) then the surface finish of the cut edges must be controlled so as to minimize errors.

The sample dimensions in the waist region (gauge section) are measured with a micrometer or dial gauge and then the sample is firmly gripped in the jaws of the machine. An extensiometer may then be clipped to the sample. One jaw of the machine is drawn away from the other (usually by an electric motor) at the specified speed. A load cell that is connected to one of the jaws, measures the resistance to deformation. A load/extension curve is usually produced automatically by the machine. Five specimens are normally tested and the average result(s) quoted together with the standard deviation.

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## **Stress and Strain**

The application of the tensile stress causes the specimen to stretch from its original gauge length (I<sub>o</sub>) to a longer length (I<sub>i</sub>). Tensile strain ( $\Sigma_1$ ), commonly referred to as strain, may then be defined as the change in length/original length which is

 $\Sigma_1 = (\mathbf{I}_1 - \mathbf{I}_0)/(\mathbf{I}_0)$  (Eqn. 9)

Percentage strain = strain x 100.

If sufficient stress is applied then the sample will break. The percentage elongation at break  $(\Sigma_{\rm s})$  is usually expressed as a percentage of the original length and this, therefore, may be expressed as longer length (I,) minus original gauge length (I\_{\rm o}) divided by original gauge length (I\_{\rm o}) times 100.

Tensile strength at break, also called the ultimate tensile stress, may be obtained by dividing the force necessary to cause failure during a tensile test, by the cross-sectional area at break. As this area is difficult to measure, the tensile strength that is commonly quoted is that given by  $F/A_o$ . Where F is the force which causes failure and  $A_o$  the original cross-sectional area. Yield strength may be calculated from the force at which the specimen continues to elongate without additional load. The stress and strain obtained by using the original sample dimensions are sometimes referred to as 'nominal' or 'engineering' stress and/or strain.

## Young's Modulus

The ratio of stress/strain, when stress is proportional to strain, is called the elastic modulus or the Young's Modulus. It is a tensile modulus, obtained from the results of a tensile strength test. This value is commonly quoted for many materials as it gives an appreciation of the stiffness of a particular material. For materials such as steel the value of Young's Modulus, obtained from the slope of a stress/strain plot (up to the yield point), does not depend markedly on the test conditions and has come to be regarded as a material constant. This is not the case for polymeric materials, since the results obtained depend markedly on the test conditions, for example, on the speed or temperature of testing.

Young's Modulus, which is the elastic modulus for uniaxial extension, is the slope of the stress strain plot for small extensions: that is, stress divided by strain. It has the units of force per unit area, for example, N/mm<sup>2</sup> or psi. For small extensions, this elastic modulus is approximately the same as the compressive modulus.

## **Creep Testing**

A short term test, such as a tensile test, is not capable of giving information which can be used in the design of a continuously stressed component in a particular environment. It is only really suitable for quality control purposes. On the other hand, a creep test will indicate that a small load applied for a long period, to a thermoplastic material, can cause a significant deformation. Tests, such as a tensile test, are referred to as single point tests while creep tests take into consideration the effects that changes in time, temperature and environmental conditions have on the deformational properties of polymers.

Creep tests are performed by weight loading a plastic sample, maintained at a specified temperature. Periodic measurements of the extension are made, perhaps over several years. Often a dumbbell specimen is cho46 sen that has a very long parallel section, so the small deformations, which result upon loading, can be measured accurately. The material is tested at a specific temperature by applying a weight (or stress) by means of a lever system. Since large loads may produce only small extensions, a very sensitive and accurate extensiometer is required. Sample conditioning is extremely important and may involve regulation of test temperature and/or humidity for several months to ensure that the specimens are in equilibrium with their surroundings. In the case of nylon 66, such storage will allow water take-up to occur as well as allowing post-molding crystallization to develop.

The results of creep testing may be expressed in various ways, for example, a creep modulus may be obtained by dividing the applied stress by the time dependent strain. If the creep modulus is divided by the room temperature elastic modulus (the Young's Modulus) then a reduced stiffness value is obtained. Creep modulus/time curves are obtained by plotting creep modulus against log time. A family of tensile creep modulus curves, as a function of strain and log time, may be obtained.

## ► FLEXURAL PROPERTIES

Flexural properties are measured to obtain a measure of stiffness or rigidity. This may be a more appropriate test than measuring tensile strength if the extrudate is to be deformed by bending during its service life.

## **Test Specimens**

The specimens used for flexural or bending tests are simple rectangular bars the length of which must be at least 20 times the thickness. The width and thickness can vary, but the preferred dimensions are width 10 mm and thickness 4 mm. All samples should be conditioned at the test temperature and appropriate humidity for a standard time. As for tensile testing, at least five specimens should be tested and the arithmetic mean and standard deviation of the five individual results reported. Where there is anisotropy, five specimens should be tested in two directions at right angles.

## Flexural Strength and Modulus

Flexural strength is the strength determined from the load and the test piece dimensions in a flexural test. Flexural strength is also known as breaking strength and is usually measured by applying a stress at the center of a rectangular bar which is supported at two other points. That is, three-point loading is applied in a bending test. During the test, the force applied and the resulting deflection are measured. The test is usually performed on a universal testing machine (as used for tensile strength tests).

Bending the specimens in a three point bending jig (on which the span or distance between the two outer supports is set 15 to 17 times the thickness) gives a load/deflection curve or graph. From this curve flexural strength and modulus are determined.

## Calculations

If the specimen is brittle and breaks at very low strain (less than 0.05) then, the breaking or flexural strength is calculated from

#### $\sigma_{\rm B} = (3 \ {\rm F}_{\rm B} \ {\rm L})/(2 \ {\rm b} \ {\rm h}^2)$ (Eqn. 10)

Where  $\sigma_{\scriptscriptstyle B}$  is the flexural strength,  $F_{\scriptscriptstyle B}$  = load at break, L = span width, b and h are width and thickness of the specimen, respectively. If the sample does not

break but simply bends then the stress measured when the sample deflection reaches 1.5 times the sample thickness is used. The breaking strain is often given the symbol  $\gamma$  and equals 6D h/L, where D is the deflection at mid-span. The elastic modulus in flexure, symbol E, can be calculated from

#### $E = [L/(4 b h^{3})](F/Y)$ (Eqn. 11)

where F/Y is the slope of the initial linear load-deflection curve.

## > IMPACT STRENGTH

In the plastic industry the most common type of impact test is a pendulumtype test (such as the Izod or the Charpy Test). However, this is not the case for the extrusion industry where an impact test is required to assess the behavior of products such as film, sheet and pipe.

#### Falling Weight Impact Strength

The falling weight impact strength (FWIS) is the energy that on average causes 50% of test specimens to fail in a FWIS Test. This test is a very useful type of impact strength determination. It is usually performed on actual products, or on samples cut from them. The test is performed by allowing a projectile to fall on the piece to be tested. End-use service performance can, therefore, be easily simulated, as the sample will fail, as in a real life situation, along the line of least resistance. There are two main types of tests employed and these have been called the conventional falling weight and instrumented falling weight tests.

#### **Conventional FWIS Test**

In this test, widely used for testing film, sheet and pipe, the sample is struck by a weight attached to a hemispherical dart or "tup" that falls from a constant height. By testing a large number of specimens the energy, which on average causes 50% of them to fail, is determined.

When extruded film is tested the BS method requires that 14 m (45 ft) of film be available and that the film width is 260 mm. At least 60 successive but not overlapping test portions are obtained. It is usual to leave the film in the form of a strip. A vacuum is used to clamp the film taut and then a dart is dropped on it from a specified height. If the film is punctured then this is recorded as a failure and the procedure is repeated for a range of dart masses. Sets of ten test pieces are used with at least six different dart masses. The percentage of failures for each dart mass is recorded. The results are quoted as an  $M_{50}$  value. This figure is the mass, in grams, of the dart that would be expected to break 50% of a large number of specimens. For sheet the  $F_{50}$  value is quoted. This figure is the energy of the striker which caused 50% failure, that is, the mass of dart (kg) x acceleration due to gravity (m<sup>-2</sup>) x height of fall (m). In this case the result is given in Joules (J).

A graph is drawn on probability paper of dart mass against the percentage of test pieces punctured for each set of ten test pieces. The best fit straight line is drawn through the points and the  $M_{\rm 50}$  value read off. This is reported as the falling weight impact strength of the film.

#### **Instrumented Falling Weight**

In this test a dart, or tup, is used which is so heavy that it will break the specimen easily, without a significant decrease in striker speed. A transducer is mounted behind the nose of the striker and the information that this generates is used to measure the maximum force and to calculate the energy to break the specimen. Only a relatively small number of components or test specimens are required for this test.

The test is normally performed at room temperature and the signal from the transducer, as it breaks the sample, is amplified, recorded and/or displayed. To get more consistent results, and to stop the sample wrapping around the nose of the tup, a striker with a flat face is sometimes used. The speed of the tup is measured photo-electrically. It is relatively easy to perform this test with only a few specimens over a range of temperatures to obtain information about ductile to brittle transitions. To show the effect of processing on properties, the results obtained by testing the actual components may be compared against results obtained when compression molded specimens of the same thickness, are used. Compression molded samples are assumed to be free of orientation, that is, isotropic.

It has been found that a reasonably good approximation of the energy causing breakage (E\*) can be obtained by multiplying the impact speed (V<sub>0</sub>) by the area under the force-time graph up to the point where failure begins, if the speed of impact (V<sub>0</sub>) remains virtually unchanged during the impact. That is:

#### $E^{\cdot} = V_0 fdt$ (Eqn. 12)

If the dart speed changes during the time it is forcing its way through the specimen, then a correction must be applied to  $E^*$ . The corrected value of the energy of breakage (E) is then calculated from:

#### $E = E^{\cdot} (1 - E^{\cdot}/4E_{0})$ (Eqn. 13)

where  $\mathsf{E}_{\scriptscriptstyle 0}$  is the kinetic energy of the dart at the moment of impact.

## HARDNESS AND SOFTNESS

The hardness and/or softness of a material commonly means resistance to indentation or penetration by a blunt indenter (a probe or a ball) loaded with a spring. Both the indenter and the spring have precisely fixed characteristics, since indentation is dependent on the indenter profile. The loaded indenter remains in contact with the test material and the depth of penetration is measured after a fixed time. Such a test is typically used for rubbers and plastics where rapid recovery, after removal of the deforming force, makes it impossible to measure the dimensions of the indentation.

#### **Rockwell Hardness**

The Rockwell Hardness is a number that indicates the hardness of a material as measured by an indentation test. The use of different loads and procedures gives several Rockwell hardness scales. Scale C is used for metals. Those used for plastics include R, L and M. Two procedures are used. The first when there is no appreciable recovery when the indenting force is removed and the second gives  $\alpha$  Rockwell hardness values when there is recovery.

For the first procedure, the test specimen is placed on a flat anvil below the indenter, and a minor load of 10 kg is applied, forcing the indenter into the material. Within 10 seconds the scale is set to zero. A major load of 60 kg, or 100 kg depending on the scale, is applied for 15 seconds and then immediately removed, with the minor load remaining. The hardness reading is taken from the scale 15 seconds after removing the major load.

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The second method is used with the R scale only and gives  $\alpha$ Rockwell values. The indentation is noted 15 seconds after application of the major load, with the major load still applied. The  $\alpha$  Rockwell hardness value is then obtained by subtracting the indentation from 150.

#### **Shore Hardness**

The Shore Hardness is a measure of the hardness or softness of a material as assessed by the resistance (on a scale of 0 to 100) experienced by an indenter. 100 on the scale corresponds to a high resistance (that given by a sheet of glass) and 0 corresponds to zero resistance. Two different indenters are used: Type A for soft materials and Type D for hard materials. This means that there are two scales of Shore Hardness, Shore A and Shore D.

Type A measurements are made on soft materials using a truncated 35° cone with a blunt tip of 0.79 mm diameter as an indenter. Type D measurements are made on harder materials with a 30° steel cone rounded to 0.1 mm radius tip as an indenter. The depth of penetration is inversely indicated in thousandths of an inch. The harder the surface the higher the reading.

The specimen must be conditioned prior to testing and it has to be sufficiently flat over an area of at least 6 mm/0.25" radius from the indenter point to allow contact with the foot of the instrument. It must also have a thickness of at least 6 mm unless it is known that identical results can be obtained with a thinner specimen. Measurements are made at several points over the surface simply by pressing the instrument against the surface of the specimen. Readings should be taken after a standard period of time, the ISO recommends 15 seconds and the ASTM one second.

## Softness

The softness number gives a measure of softness. It is a measure of the resistance to indentation of a polymeric material, such as plasticized polyvinyl chloride (PPVC) compound. An indentor is pressed into a sheet of the PPVC compound under a specified load and the depth of penetration of the indenter is measured.

The British Standard softness number is a number that indicates the softness of a material. The higher the number the softer the material. An abbreviation used for this term is BS Softness Number. A flat sheet or disc is used which is between 8 and 10 mm thick, conditioned at the test temperature and humidity. The time and temperature of conditioning is important. For example, it is recommended that temperature conditioning be seven days at 23°C for plasticized polyvinyl chloride (PPVC) compounds. This is because the properties of such compounds are known to change over several days after molding.

The test is performed using a duro-meter. This measures the increase in depth of penetration of a steel ball, 2.5 mm in diameter, into the flexible material, when the force on the ball is increased from 0.30 N to 5.70 N. The minor or contact load of 0.30 N is applied for 5 seconds and then the load is increased to 5.7 N. The reading on the dial is noted when 5.7 N has been applied for 30 seconds. Four readings are taken at different points on the surface, away from the edge of the specimen, and the average is expressed as the BS softness number.

## > THERMAL PROPERTIES

Plastic materials are very sensitive to changes in temperature. When the temperature is raised many thermoplastic materials will soften at a comparatively low temperature. If the temperature is lowered then the material may harden at another comparatively high temperature. Thus the temperature range of use of many thermoplastic materials is limited. Such use is limited, not by chemical breakdown, but by hardening or softening. Other problems associated with the use of thermoplastics are the low thermal conductivity, high specific heat, high coefficient of thermal expansion and a high and non-uniform, shrinkage. The flammability of plastics materials and the smoke produced by their burning, may also be serious disadvantages.

## Shrinkage

Shrinkage is the reduction in volume that occurs when a material is cooled from its processing temperature (often quoted shrinkage values are based on injection molded components). Shrinkage is much greater for a semicrystalline thermoplastic material, such as high-density polyethylene (HDPE), than it is for an amorphous, thermoplastic material, such as polystyrene (PS). For PS it is about 0.6% whereas for HDPE it may reach 4%. Shrinkage may also be different from one grade of material to another and will be influenced by changing the processing conditions. It may also be significantly different in different directions (anisotropy), across the flow and along the flow. Because of this it is usual to quote a shrinkage range for each plastic material and the values quoted **(Table 5.)** should only be taken as a guide.

Mold shrinkage is defined as the change in dimensions between the size of the cavity and the molded part 24 hours after the part is ejected from the mold. Mold shrinkage is quoted as a percentage, for example, 0.4%, or as a linear shrinkage, such as 0.004 in/in or 0.004 mm/mm. A widely used test for the measurement of mold shrinkage is ASTM D 955-51. For injection molded specimens, an end-gated bar of thickness 3.2 mm is usually used. The cavity length, both parallel and perpendicular to the flow direction are measured to the nearest 0.02-mm at 23  $\pm$ 2°C. At least three test specimens are then molded. After molding the specimens are allowed to cool to room temperature. This cooling time (before initial mold shrinkage is measured) is 1 to 2 hours when the thickness is 3.2 mm. The length (both along and transverse to flow) are measured and then the specimens are returned to storage in a standard laboratory atmosphere (typical conditions are 23°C at 50% RH). Measurements are made again at 24 hours and 48 hours after molding in order to obtain 24 hour shrinkage and the 48 hour (or normal mold) shrinkage.

Results are usually given as mm/mm (same as in./in.) and this is obtained by subtracting the dimension of the sample from the corresponding dimension of the cavity and then dividing by the latter. The mold shrinkage (MS) is given as a percentage if the following formula is used:

#### $MS = [(L_0 - L_1) / (L_0)] \times 100.$ (Eqn. 14)

where  $L_0$  is the length in mm of the cavity  $L_1$  is the length in mm of the molding (to the nearest 0.02 mm).

## Table 5. Shrinkage (Mold Shrinkage) Values for Some Thermoplastic Materials

Abbrev.	Material	Linear MS	% MS
		in/in or mm/mm)	
ABS	Acrylonitrile-butadiene- styrene	0.004-0.007	0.4-0.7
CA	Cellulose acetate	0.003-0.007	0.3-0.7
CAB	Cellulose acetate butyrate	0.002-0.005	0.2-0.5
EVA	Ethylene vinyl acetate	0.007-0.020	0.7-2.0
FEP	Fluorinated ethylene propylene	0.030-0.060	3.0-6.0
HDPE	Polyethylene (high density)	0.015-0.040	1.5-4.0
HIPS	Polystyrene (toughened)	0.002-0.008	0.2-0.8
LDPE	Polyethylene (low density)	0.0165-0.040	1.65-4.0
PA 6	Nylon 6	0.010-0.015	1.0-1.5
PA 66	Nylon 66	0.010-0.020	1.0-2.0
PBT	Polybutylene terephthalate	0.015-0.020	1.5-2.0
PC	Polycarbonate	0.006-0.008	0.6-0.8
POM	Acetal	0.018-0.025	1.8-2.5
PMMA	Acrylic	0.002-0.010	0.2-1.0
PPO	Polyphenylene oxide (modified)	0.005-0.007	0.5-0.7
PP	Polypropylene	0.010-0.030	1.0-3.0
PS	Polystyrene (GP)	0.002-0.008	0.2-0.8
PTFE	Polytetrafluoro-ethylene	0.050-0.100	5.0-10.0
UPVC	Polyvinyl chloride- unplasticized	0.002-0.004	0.2-0.4
PVC	Polyvinyl chloride (plasticized)	0.015-0.050	1.5-5.0
PVF	Polyvinylidene fluoride	0.020-0.030	2.0-3.0
SAN	Styrene-acrylonitrile	0.002-0.006	0.2-0.6
Thermoplasi	tic Elastomers		
PP/EP(D)M	Rubber reinforced polypropylene	0.010-0.020	1.0-2.0
SBS	Styrene-butadiene- styrene	0.004-0.010	0.4-1.0
PEEL	Thermoplastic polyether ester	0.004-0.016	0.4-1.6
TPU	Thermoplastic polyurethane	0.005-0.020	0.5-2.0

## **Specific Heat Capacity**

Also known as specific heat and sometimes abbreviated to c or to, sp ht or  $C_p$  (when measured at constant pressure). Heat capacity divided by mass (heat capacity is the amount of heat energy required to change the temperature of a body, or of a substance, by 1°C (or 1K). The amount of heat which must be added to unit mass of a material in order to raise its temperature by one degree. Values are commonly expressed in J/kgK, Btu/lb°F or in cal/g°C. To convert from Btu/lb°F or cal/g°C to J/kgK multiply by 4186.80.

As specific heat varies with temperature, it is necessary to specify the temperature employed when quoting a specific heat value. With a crystalline polymer the heat of fusion causes a large increase in the specific heat 52 value at the crystalline melting point. Beyond that temperature the value falls again. Table 6 gives average values for some materials. The high specific heats and heat contents of plastic materials should be noted together with the difference between amorphous and crystalline materials. The specific heat of filled systems, such as rubber compounds, is a cumulative property of all the compounding ingredients.

Heat content is the amount of heat energy that a system contains. Specific heat is commonly measured by determining the heat content (enthalpy) over a temperature range (such as upper processing temperature to room temperature) by methods such as differential scanning calorimetry (DSC) method. If such an average specific beat is known, then the amount of heat that must be removed, for example, by the mold cooling system in injection molding can be estimated as:

#### Heat Content = mass x specific heat x (melt temp - mold temp) (Eqn. 15)

As the DSC method can take account of the latent heat of fusion at the crystalline melting point, no allowance need be made for it. However, if enthalpy data are available it is best to use them directly. If the information is used for injection mold cooling calculations, assume that only half the heat needs to be removed from components. This is because the cool surface layers of the molding provide sufficient rigidity so that ejection can occur even though the temperature of the inside of the molding may still be very high.

# Table 6.Average Specific Heat and Heat Content for SomeThermoplastic Materials

Material Abb.	Melt Temp	Mold Temp	Temp. Difference	Specific Heat	Heat to be Removed
	(°C)	(°C)	(°C)	(Jkg <sup>-1</sup> K <sup>-1</sup> )	(Jg⁻¹)
FEP	350	220	150	1600	240
POM	205	90	115	3000	345
SAN	240	60	180	1968	354
PC	300	90	210	1750	368
ABS	240	60	180	2050	369
PMMA	260	60	200	1900	380
PPS	320	135	185	2080	385
PS	220	20	200	1970	394
ASA/AAS	260	60	200	2010	402
HIPS	240	20	220	1970	433
PP0	280	80	200	2120	434
PA 6	250	80	170	3060	520
LDPE	210	30	180	3180	572
PA 66	280	80	200	3075	615
PP	260	20	240	2790	670
HDPE	240	20	220	3640	801

#### **Glass Transition Temperature**

The glass transition temperature, also known as  $T_0$  or the glass-rubber transition temperature is the temperature at which a polymer becomes glass-like on cooling or rubber-like on heating. It is not defined by a sharp, precisely located point, but by a temperature range **(Tables 7. and 8.)**.

At the  $T_{\rm g}$  there is a volume increase in polymeric materials on heating. This arises from segmental movements of the molecular chains. The tem-

perature at which this change in volumes occurs may be measured by dilatometry and used as a measure of glass transition temperature ( $T_{\rm q}$ ). Enthalpy changes are also used to determine  $T_{\rm g}$  as are modulus, refractive index, and thermal conductivity changes. Each method may give a different answer and such differences help account for the differences found in the literature between reported glass transition temperatures ( $T_{\rm g}$ ) which may be as high as 30°C.

## **Melting Point**

The melting point, also known as the melting temperature (strictly speaking this is the temperature at the melting point) is the temperature at which a solid material becomes a liquid on heating. It is the temperature at which the solid and liquid phases of a material are in equilibrium at a specified pressure (usually atmospheric).

Unlike low molecular weight materials, polymers do not normally have a sharp melting point. Even a semi-crystalline, thermoplastics have broad melting points of as much as 20°C. An amorphous thermoplastics does not exhibit a melting point, but on heating, but will soften, as the temperature increases, until it becomes a high viscosity melt. True melting is a first order transition and the melting temperature given the symbol  $T_m$ . A melting point approximation may be obtained by heating small pieces of the material on a hot plate and noting the temperature at which the material melts. For a more accurate determination, a capillary tube method may be used.

The capillary tube method (see, for example, ASTM D 2117) may be used to obtain an accurate measurement of the melting point of a crystalline polymer such as a polyamide (PA). A thin strip of PA is placed inside a capillary tube. The tube is then slowly heated inside a heating apparatus to the temperature where the sharp edges of the specimen become rounded. Reported results have been obtained in different ways, for example, when the last traces of material become a melt. This gives a relatively high  $T_m$  and helps account for the differences found in the literature between reported melting points **(Table 7.)**.

## Table 7.

## Glass Transition Temperatures $(T_g)$ and Melting Temperatures $(T_m)$ of Some Thermoplastic Materials

Abbreviation	Material Name	T, (°C)	<b>T</b> _m (° <b>C</b> )
ABS	acrylonitrile-butadiene-styrene	-20/105	
AS	acrylonitrile-styrene - see SAN		
CTA	cellulose triacetate	105	306
СТВ	cellulose tributyrate	183	
CAP	cellulose tripropionate		234
FEP	fluorinated ethylene-propylene		271
HIPS	high impact polystyrene	100	
IPS I	sotactic polystyrene	100	240
IPMMA	isotactic polymethyl methacrylate	45	160
PA 6	nylon 6	50	215
PA 11	nylon 11		185
PA 12	nylon 12		175
PA 46	nylon 46		295
PA 66	nylon 66	55	265
PA 610	nylon 610	40	227
PAA 6	polyaryl amie/an aromatic nylon	90	236

54	<b>Abbreviation</b> PAN PAB	Material Name polyacrylonitrile polyarylate	<b>T, (°C)</b> 80/104 D 180	<b>T</b> <sub>m</sub> (°℃)
	PR	polybutene-1	-25	126
	PBAK	polybutone i	40	195
	PBT	polybutyl dolytate	30	225
	PC	nolycarbonate	150	225
	PCTFF	polychlorotrifluorethylene	49	220
	PEHD	high density polyethylene	10	130
	PFLD	low density polyethylene	-20	120
	PFLLD	linear low density polyethylene	20	127
	PEEK	polvetheretherketone		334
	PEI	polvether imide	215	
	PEK	polvetherketone	154	367
	PEOX	polvethylene oxide	-56	66
	PET	polyethylene terephthalate	69	260
	PMMA	polymethyl methacrylate	99	
	PMP	poly-4-methylpentene-1	55	250
	PMS	poly- $\alpha$ -methylstyrene	120	
	POMCO	polyoxymethylene/acetal		
		copolymer		165
	POMH	polyoxymethylene/acetal		
		homopolymer	-13	175
	PP	polypropylene	0	165
	PPE	polyphenylene ether	210	
	PPOX	polypropylene oxide	-62	75
	PPS	polyphenylene sulfide	85	282
	PS	polystyrene	100	
	PSU	polysulfone	190/230	
	PTFE	polytetrafluoroethylene	126	327
	PVAC	polyvinyl acetate	30	
	PVAL	polyvinyl alcohol	50/70	D
	PVC	polyvinyl chloride	87	212
	PVDC	polyvinylidene chloride	-25	190
	PVDF	polyvinylidene fluoride		177
	PVF	polyvinyl fluoride	-20	200
	SAN	styrene-acrylonitrile	105	
	SP	saturated polyester - see PBT and PET		
	SPMMA	syndiotactic polymethyl methacrylate	110	>200
	(D = decompo	oses before melting)		

## **Softening Point**

The softening point is the temperature at which thermoplastic material softens by a specified amount in a softening point test. The amount of softening is usually measured by the indentation of a weight-loaded needle (a Vicat Softening Point apparatus will provide this information) or by the bending of a weight-loaded bar as the temperature is slowly increased. A softening point test is a thermal yield test.

## **Cantilever SP Test**

A common softening point test for thermoplastic materials is a cantilever or bending test. Such a test uses a rectangular strip, or bar, which is 31.8 mm/1.25" by 6.35 mm/0.25" by 1.52 mm/0.06". A 1.5 mm/0.062" diameter hole is located centrally 1.57 mm/0.25" from one end. A 20 g weight is attached to the bar by a piece of cotton thread (which passes through the

hole). The end of the strip opposite the hole is held in a clamp, so that  $25.4 \text{ mm/1}^\circ$  of the strip is protruding. The 20 g weight is attached to the free end.

Initially the weight is supported so that there is no load on the test piece. The clamp assembly is then immersed in a liquid heating bath that is approximately 30°C below the expected SP and the support removed from the weight. While stirring, the temperature of the bath is raised at 1°C/minute and the temperature noted when the upper edge of the free (weighted) end coincides with a 30° graduation on the clamp assembly. The mean of two tests is taken as the softening point.

When the clamp assembly is immersed in the liquid heating bath, the liquid must not affect the properties of the material being tested. Liquids used include liquid paraffin for cellulose acetate (CA) and glycerol for polystyrene (PS), high impact polystyrene (HIPS) and unplasticized polyvinyl chloride (UPVC).

#### **Vicat Softening Point**

This is a softening point test that is performed using a Vicat Softening Point needle instrument. This term is sometimes abbreviated to VSP. The result, the Vicat Softening Temperature (VST), is usually quoted in °C or °F. It is measured by applying a standard load (10 N or 50 N), via a circular indenter (a round flat-ended needle of 1 mm<sup>2</sup>) onto a plastics specimen. The test assembly is heated at a constant rate of either  $50^{\circ}$ C/122°F or  $120^{\circ}$ C/248°F per hour. The result is reported as  $100^{\circ}$ C (10 N,  $50^{\circ}$ C). This means that the indenter penetrated into the thermoplastic sample at  $100^{\circ}$ C/212°F for the set distance (for example, 1 mm) while carrying a load of 10 Newtons and at a heating rate of  $50^{\circ}$ C.

ISO method 306 describes two methods: Method A which uses a load of 10 N, and Method B which uses a load of 50 N. As two heating rates are allowed ( $50^{\circ}$ C/hr and  $120^{\circ}$ C/hr) this means that the results may be reported as:

- Vicat A50 = load 10 N and heating rate 50°C/hr
- Vicat A120 = load 10 N and heating rate 120°C/hr
- Vicat B50 = load 50 N and heating rate 50°C/hr
- Vicat B120 = load 50 N and heating rate 120°C/hr

## Table 8. Typical Thermal Properties of Some Thermoplastic Materials (°C)

Material	T۹	T <sub>m</sub>	SP	HD	Т	VSP
				(0.45 Mpa)	(1.8 Mpa)	
ABS	105	-	94	96	84	95
HIPS	100	-	84	85	72	86
PA 66	55	265	180	183	75	185
PE-HD		130	115	75	45	125
PE-LD	-20	120		45		85
PMMA	99		95	97	80	90
POM-H	-13	175	170	120	165	175
PP	0	165	145	140	60	150
PS	100	-	92	97	90	98

 $T_g$  = glass transition temperatures, SP = cantilever softening point test,

 $T_m =$  melting temperature

SP = cantilever softening point test, VSP = Vicat softening point test,

HDT = heat distortion temperature

## <sup>56</sup> Heat Distortion Temperature

The heat distortion temperature is also called the heat deflection temperature. However, it should more accurately be called heat deflection under flexural load or, the heat deflection under load (HDUL). It is often abbreviated to HDT (which may stand for heat distortion temperature or heat deflection temperature). In test specifications, HDT is called deflection temperature under flexural load. Two different flexural loads are generally quoted: 1.8 MPa or 0.48 MPa (264 or 66 psi).

In the HDT Test a plastic bar (for example, 110 x 10 x 4 mm which is 4.4 x 0.4 x 0.16") is subjected to three-point bending, by a load that produces a maximum stress of either 1.8 MPa or 0.48 MPa (264 or 66 psi), at its mid-point, while being heated. If the breadth (b) is 4 mm, the depth (d) is 10 mm and the stress required is 1.8 MPa then, the load (F) in Newtons which has to be applied to the mid-point (if the span (L) is 100 mm) is equal to 2Pbd<sup>2</sup>/3L. That is, 2 x 1.8 x 100/ 3 x 100 which equals 4.8 N. This load is applied and the temperature raised at 120°C per hour. When the bar deflects by 0.32 mm (0.012") that temperature, in °C or °F is recorded and called the HDT.

A comparison of some HDT temperatures for thermoplastic materials is given in  $\ensuremath{\textbf{Table 9}}$  .

## **Cold Flex Temperature**

This is the temperature measured in a Cold Flex Temperature Test. This cold flex temperature test is a brittleness temperature test used to assess the low temperature properties of plasticizers or of plasticized compounds such as plasticized polyvinyl chloride (PPVC).

A rectangular strip of plasticized PVC ( $64 \times 6.4 \times 1.27$  mm thick) is cooled in a bath of methanol and solid carbon dioxide. The temperature is raised at a rate of 20°C/minute and the strip is twisted using a specified torque: initially the strip is twisted by more than 400°. A graph is plotted of temperature against time and the temperature for a deflection of 200° is read off the graph and called the cold flex temperature.

#### Table 9. Comparison of Softening Temperatures for Some Thermoplastic Materials

Material	Abbreviation	HDT	HDT (°C)	
		( 1.8 Mpa)	(0.48 Mpa)	(10N 50°C/hr)
Polymethyl		,	,	. ,
methacrylate	PMMA	71-99	77-110	114
Polypropylene	PP	50-60	107-120	134
Unplasticized				
polyvinyl chloride	UPVC	60-77	60-82	85
Acetal	POM	85-120	154-166	160-185
Nylon 66	PA 66	74	245	240
Polycarbonate	PC	132	138	155

## **Stress Relaxation**

The stress relaxation (sometimes called reversion) is the shrinkage, or change in shape, that occurs when a thermoplastic material is heated to a specified, elevated temperature, generally a temperature above the glass transition temperature ( $T_{g}$ ). This change of shape is caused by release of molecular orientation, or stress, introduced by alignment of the polymer molecules in the direction of flow.

A measure of stress relation may be obtained in the following manner: A component is placed on a piece of paper and an image of the part is traced. The image is then cut out and weighed. The component is heated at the specified temperature for a fixed time. The distorted component is then removed, cooled and placed on another piece of paper similar to the first. The outline of the distorted component is marked and the shape cut out and weighed. A measure of stress relaxation is obtained by comparing the two weights. If the product is a plastic sheet then marking a circle on the sheet and measuring the change in length of two diameters at right angles to each other is used to assess stress relaxation. Usually the two dimensions are the machine direction and the transverse direction.

## FLAMMABILITY TESTING

The ability of a plastic material to withstand heat and flame without burning is critical to how it may be used. The requirements placed on appliances, house furnishings, electrical parts and clothing will be much stricter than those for packaging or outdoor furniture. Thus flammability testing is required to assure the suitability of a plastic material for many applications.

The flammability testing of plastics is complicated by many factors that may affect the results. The thickness of the sample, location of the flame, orientation of the test specimen, the oxygen concentration, the ambient temperature and air velocity may all have an influence on the results. Two methods for evaluating the flammability are generally used, the Limiting Oxygen Index (LOI) and the Underwriter 's Laboratory rating system (UL94). Some flammability testing results are shown in **Table 9**.

## Limiting Oxygen Index (LOI)

The LOI Test is one of the few, reproducible, flammability tests that produces quantitative results. It measures the amount of oxygen required for a material to support combustion. In this test the sample is ignited in a chamber containing a controlled nitrogen/oxygen atmosphere in which it will burn. The oxygen level is then reduced until the sample is extinguished. The LOI is the lowest percent oxygen that will support combustion of the sample. Thus this test can be used to test the performance of plastic materials that will not normally burn in air. In general, any material with an index greater than 28 (air contains 21% oxygen) is considered flame resistant.

## Table 10. Typical Limiting Oxygen Index (LOI) Results

Material	Abbreviation	Oxygen Index (%)
Acrylonitrile-butadiene-styrene	ABS	19-35
Acetal	POM	15-16
Nylon 6	PA 6	21-24
Nylon 66	PA 6	21-30
Nylon 11	PA 11	25-32
Phenol-formaldehyde	PF	35
Polyarylate	PAR	34
Polycarbonate	PC	26
Polybutylene terephthalate	PBT	18

58	Material	Abbreviation	Oxygen Index (%)
	Polyether ether ketone	PEEK	35
	Polyether sulphone	PES	34
	Polyethylene	PE	17
	Polyethylene oxide	PE0	15
	Polyethylene terephthalate 30%GF	PET 30%GF	31
	Polyimide	PI	32
	Polymethyl methacrylate	PMMA	17
	Polyphenylene oxide (modified)	PP0	28
	Polypropylene	PP	17
	Polystyrene	PS	18
	Polysulphone	PSU	30
	Polyvinyl chloride	PVC	23-43
	Polyvinyl alcohol	EVOH	22
	Polyvinylidene chloride	PVDC	60
	Polyvinylidene fluoride	PVDF	44
	Polytetrafluoroethylene	PTFE	95

## **UL94 Flame Class Rating**

The Underwriter's Laboratory has established a series of qualitative ratings of flame retardance, listed in their UL94 Bulletin, that are broadly accepted in the plastics industry. In the tests, variables such as the flame size as well as the sample form, preparation and orientation are strictly controlled. The ratings generated include:

- HB Horizontal burn (inches/min.)
- V-0 Vertical burn, average burn of 5 sec. or less
- V-1 Vertical burn, average burn of less than 25 sec. (no burning drops)
- V-2 Vertical burn, average burn of less than 25 sec. (burning drops)
- 5V~ More severe than V-0 (5 inch flame, 5 positions, 5 ignitions, no burn through, no drips)

Other generally accepted flammability tests include the ASTM D-635 Horizontal Burn Test, the ASTM E-84 Tunnel Test, the ASTM E-162 Radiant Panel Test, and the Ohio State Cone Calorimeter Test.

#### Table 11. Typical Results from a UL 94 Vertical Burning Test

Material Acetal	UL 94 coding HB
ABS	HB
ABS/PVC alloy	V-1
ABS/PC blend	V-0
Nylon 6	V-2
Nylon 66	V-2
Glass reinforced nylon 66	V-0
Glass reinforced PET	V-0
Glass reinforced PPS	V-0
Glass reinforced polycarbonate	V-0
Glass reinforced polysulphone	V-0
Polyarylate	V-0
Polycarbonate	V-2
Polyether ether ketone	V-0
Polyetherimide	V-0
Polyphenylene oxide	HB to V-0
Polyethersulphone	V-0
Polysulphone	V-0

## ► MELT FLOW RATE

The most commonly used test is usually referred to as MFR or MFI. Both terms refer to the same test and stand for melt flow rate and melt flow index, respectively. This test is popular, particularly for polyolefins, since the test is easy to do and understand and the instrumentation is inexpensive. A specified force, produced by an accurate weight, extrudes a heated plastic material through a circular die of a fixed size, at a specified temperature (Figure 12.). The amount of PE extruded in 10 minutes is called the MFR and results are reported as, for example, MFR (190, 2.16) = 2.3. This means that the temperature was 190°C and a force, producing load of 2.16 kg, was used. Under these conditions, 2.3 g of the plastic material was extruded in 10 minutes. MFR is general a low shear rate test where the shear rate may be as low as 1 sec-1. Weights larger than 2.16 kg may be used as well as different temperatures. These will depend upon the material and the grade of material being tested (Table 12.). With UPVC a weight of 20 kg may be employed, while the suggested temperature (in ASTM D 3364) is 175°C (347°F). (See Flow Rate Testing).



Figure 12. Melt Flow Rate Tester

## Method A

Method A, also known as Procedure A, is a manual cut-off operation used for materials having flow rates that fall within 0.15 to 50 g/10 minutes. The piston position during the timed measurement, (that is, the position of the piston tip) is required to be between 51 and 20 mm (2.0 and 0.8 in) above the die. The test specimen can be in any form that can be introduced into the heated barrel. The conditions of temperature and load can be selected by consulting the standard. For example, for a PE with an expected MFR of 2, the temperature would be 190°C and the load would be 2.16 kg. After packing a precisely measured amount of PE (in the range of 3 and 5 g) into the barrel, the sample is allowed to heat for 4 minutes with the piston in contact with the material.

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Then the weight (2.16 kg) is placed on top of the piston. After a further 3 minutes (usually between 2 and 4 minutes) the lower reference mark on the piston must be level with the top of the barrel. The material is then allowed to freely extrude for a specified time (such as 3 minutes), after which the extrudate is carefully cut off and saved. This should be repeated three times if possible, before the upper reference mark reaches the top of the barrel. The three samples are then individually weighed, to the nearest milligram (0.001g), and the average mass in grams is found. The flow rate is reported as the rate of extrusion in grams in 10 minutes (obtained in this case, by multiplying the average mass of polymer extruded ( $M_p$ ) by 3.33). Thus

#### $MFR = MFI = M_n (600/t)$ (Eqn. 16)

where:

t = the cut-off interval expressed in seconds (s)  $M_{\rm p}$  = the mass of polymer extruded

#### Method B

Method B is also known as Procedure B and is an automatically timed flow rate measurement used for materials having flow rates that fall within 0.50 to 1,200 g/10 minutes. To ensure reproducibility, the timing device must be accurate to within ±0.1 seconds and the position of the piston foot at the end of the test must be 25.4 mm (1 in) above the die. During the measurement, the length of timed piston movement must be measured to within 0.025 mm (0.001 in) over a prescribed distance. This measurement may be achieved by using an opague flag, hung from the piston, which interferes with the passage of light to an electronic eye. It is more common today, however, to use a mechanical encoder connected to the piston. If the system is computerized, then all the operator has to do is to select the test conditions of temperature and load by consulting the standard or the memory and load the material. The test specimen can be in any form that can be introduced into the barrel. The electronics will automatically control the temperatures and time the measurements (and even change the weight during the test run if required). The apparent melt density of the polymer (see Method A to B Conversion) at the test temperature must be entered to allow the calculation of the weight of extrudate from the piston displacement. Once the test has been performed then the electronics can calculate flow rate, flow rate ratios, viscosity, shear rate and shear stress. The electronics also may contain the statistical equations for SPC/SQC analyses of the data for quality control purposes.

#### Method A to B Conversion

Conversion from Method A to Method B may be obtained on machines where an appropriate electronic optical eye, mechanical encoder or computerized control system is used. The operator runs the test as if it is a Method A test, while the machine conducts a Method B test. Upon completion of the test the apparent density is obtained using the data from both tests and setting the equations equal and solving for melt density.

By Method A, the melt flow rate (MFR) is  $MFR = M_p$  (600/t)

Where  $M_{\rm p}$  is the average mass extruded (M) within the cut-off interval t (expressed in seconds).

By Method B,

#### MFR = $\pi R^2 L D_M$ (600/T) (Eqn. 17)

Where R is the piston radius in cm, L is the effective length of the flag in cm, A is the apparent melt density in g/cm<sup>3</sup> and T is the time taken for

the test measurement (in seconds). If the two MFR's are the same then, the equations may be solved for A **(Table 5.)**. The advantage of this system is that by using an apparent density obtained from any given machine, Method B will always equal Method A and it takes into account frictional and other variations for that specific instrument.

#### **Increasing Shear Rate and Stress**

Melt Flow Rate (MFR) testing is a low shear rate test (the shear rate may be as low as 1 s<sup>-1</sup>). Larger weights and/or different temperatures may be used depending on the material and grade of material **(Table 12.)** to produce higher shear rates or to make the test practical. With un-plasticized polyvinyl chloride (UPVC) a weight of 20 kg/44.1 lb is often employed at a temperature (ASTM D 3364) of 175°C/347°F. When the test is run with such a high load, or with the even higher load of 21.6 kg/47.62 lb, then it may be referred to as "the high load melt index" or the HLMI.

## **Flow Rate Ratio**

Since polymer viscosity varies as a function of shear rate, the single point determination by a melt indexer does not fully characterize material flow. To obtain more useful information, flow rate testing may be done with more than one load. For example, in blow molding the sag behavior of the parison is best measured by the low shear flow, while the flow through the die lips is best measured by the higher shear flow. The Flow Rate Ratio is obtained by dividing the flow rate obtained when a large load is used by the flow rate obtained under a smaller load. The load ratio, which is generally 10:1, may, on some instruments, be measured automatically during a single test run.

## **Melt Volume Index**

An abbreviation used for this term is MVI. MVI is defined as the volume of thermoplastic material extruded through a die during a specified time and is expressed in cm<sup>3</sup>/10 min (MFI is in g/10 min). It is measured by performing a Method B test and doing the calculation without using the melt density. Specifications for this test can be found in DIN 53735. A rating of MVI (250/5) means that the melt volume index, in cm<sup>3</sup>/10 min, was measured at 250°C and under a load of 5 kg.

## Table 12. Suggested Temperatures (°C) and Loads (Kg) for MFR Tests

Temp. (°C)	Load (Kg)
190	2.16 or 1.05
230	1.2 or 3.8
200	5.0
230	3.8.
190	0.325, 2.16 or 21.6
210	2.16
275	0.325
235	1.0, 2.16 or 5.0
265	12.5
125	0.325 or 2.16
190	0.325, 2.16, 10 or 21.6
	Temp. (°C) 190 230 200 230 190 210 275 235 265 125 190

) Load (Kg)
12.5
1.2
2.16
5.0
1.2 or 3.8
2.16
2.16
2.16
5.0
21.6

## MELT VISCOSITY

This is the viscosity of a polymeric material in its melted form. An abbreviation used for this term is MV. Viscosity is resistance to flow or internal friction. The term viscosity is sometimes used as an alternative way of saying coefficient of viscosity or dynamic viscosity.

#### Ease of Flow

Plastic materials differ widely in their viscosity, or ease of flow. The problem of testing such materials is made more difficult by the fact that each material is available in a range of grades, each of which also may have a different flow behavior. The situation is made even more complicated by the fact that the flow properties of plastics are non-Newtonian. In most cases, plastics are pseudo-plastic materials. This means that they become less viscous (easier flowing) when they are moved faster. Therefore, there is not a linear relationship between pressure and flow. This means that the flow properties cannot be represented meaningfully by one number. Thus flow testing over a range of conditions is required.

#### **Viscosity Units**

Shear stress is usually designated by the Greek letter  $\boldsymbol{\tau}$  and has the units of force per unit area.

 $1 \text{ dyn } \text{cm}^2 = 0.002088 \text{ lbf } \text{ft}^2 = 0.0000145 \text{ lbf } \text{in}^2 \text{ or psi.}$ 

Shear rate is usually designated by the Greek letter gamma ( $\dot{\gamma}$ ) with a dot above the letter, that is, gamma dot or g. No matter what system of units is used, 1/time (in seconds) represents the units of shear rate. That is, reciprocal seconds or sec<sup>-1</sup>.

Viscosity ( $\eta_a$ ) at a particular shear rate is obtained by dividing  $\tau$  (the shear stress) by the corresponding  $\dot{\gamma}$  (the shear rate). This is an apparent viscosity. In practice the word 'apparent' is often omitted. Apparent viscosity has the units of force per unit area times time (in seconds).

One poise =  $1P = 10^{-1} Pa-s = 10^{-1} Ns/m^2 = 0.000 014 5 lbf s in^2$ One poise =  $1P = 0.1 Pa-s = 0.1 Ns/m^2$ 1 Pa-s =  $1 Ns/m^2 = 0.102 kgf s/m^2 = 10 P = 0.02088 lbf s ft^2 = 0.000145 lbf s/in^2$ 

## **Capillary Rheometer Studies**

Viscosity values are obtained by dividing the shear stress required for polymer flow by the shear rate of that flow. Both these values may be obtained from capillary rheometer studies. A capillary rheometer is a test instrument used to study the rheological properties of polymer melts by forcing the melt through a capillary (fine) die using a piston, driven at a known speed, at a constant material temperature. The pressure opposing constant flow or the force needed to maintain the specified flow rate is measured. The piston speed is then changed and the new force/pressure (needed to maintain this speed) is measured and recorded. This procedure may be repeated at different barrel temperatures. Knowing the barrel dimensions and the ram speed, the volumetric flow rate through the die can be calculated. In turn, the shear stress and the shear rate may then be calculated and used to construct flow curves. Therefore, characterization of a material's shear flow properties may be done, over a range of temperatures and imposed rates. (Table 13.) using the laboratory capillary rheometer (LCR). (See Flow Properties Assessment in Section 2)

The shear stress and shear rate conditions used for a test may closely approximate those used in a production process, so that the measured flow properties are representative of those seen at different points in the production process.

## **Extrusion Testing**

Checks may be made on incoming raw materials and thermoplastics by using a small, single-screw extruder as a rheometer. For example, a rod die may be specified and the machine set at specified temperature. The extrusion behavior is measured over a range of screw speeds and graphs are plotted of output against screw speed. Graphs of temperature against screw speed or die swell against screw speed can yield valuable data. In this way, the flow behavior of a particular material is characterized over a series of melt temperatures.

One of the major problems is maintaining the set temperature, as any alteration in screw speed quickly alters the extrusion cylinder temperatures and then the melt temperature, as a result of shear heating. For this reason the extruder should be fitted with an efficient cooling system which can remove excess heat from the barrel.

# TABLE 13.Suggested Temperatures for High Shear RateRheometry

Polymer	Test Temperature	Range
(Abbreviation)	(°C/°F)	(°C/°F)
ABS	240/464	230-270/446-518
ASA	260/500	250-280/482-536
BDS	220/428	190-230/374-446
EVA	180/356	140-225/284-437
FEP	350/662	300-380/572-716
HDPE	240/464	205-280/401-536
HIPS	240/464	200-270/392-518
LDPE	210/410	180-280/350-536
LLDPE	210/410	160-280/350-536
PA	See PA 6, PA 11,	PA 12 & PA 66
PA 6	250/482	230-280/446-536
PA 11	255/491	240-300/464-572

64	Polymer (Abbreviation)	Test Temperature	Range
		255//01	240-300/464/572
	PA 66	280/536	260-290/500-554
	PRT	250/482	200 200/000 004
	PC	300/572	280-320/536-608
	PE	See HDPE I DPE and I I D	PF
	PEEK	370/698	360-380/680-716
	PEL	380/716	340-425/640-800
	PFT	275/527	260-300/518-572
	PES	360/680	330-380/626-716
	PMMA	240/464	210-270/410-518
	POM-CO	205/401	190-210/374-410
	POM-H	215/419	190-230/374-446
	PP	240/464	220-275/428-527
	PPF	See PPO	
	PPO	280/536	260-300/500-572
	PPS	320/608	290-360/554-680
	PPVC	180/356	175-200/347-392
	PS	220/428	200-250/392-482
	PSU	360/680	330-380/626-716
	PVC	See PPVC and UPVC	
	PVDF	225/437	220-250/428-482
	SAN	240/464	200-270/392-518
	UPVC	195/383	185-205/364-401
	RMPP	240/464	220-275/428-527
	TPE	See TPE-A, TPE-E, TPE-S,	TPE-U and TPE-OXL
	TPE-A	200/392	185-240/364-464
	TPE-E	220/428	195-255/383-491
	TPE-S	170/338	150-200/302-394
	TPE-U	200/392	180-230/356-446
	TPE-OXL	190/374	180-200/356-392

## MEASUREMENT OF ELASTIC EFFECTS

The most important elastic effects are die swell, melt fracture, sharkskin, frozen-in orientation and draw down. The conditions necessary to initiate the occurrence of melt fracture and sharkskin can easily be measured on a high shear capillary rheometer. However, melt fracture, sharkskin and draw down are often measured, or studied, on laboratory-sized extrusion equipment and the results correlated with the appropriate production-sized equipment that is running the same grades of material.

#### **Extrusion Testing**

A small, single-screw extruder may be used as a rheometer. Such a machine might have a screw size of 19 mm and a length to diameter ratio (L/D) of 20:1. Usually a rod (capillary) die is specified so the shear rate through such a die may be easily calculated. The machine is usually fitted with transducers to measure die pressures and melt temperatures.

The machine is set at a specified temperature profile and once the set temperatures have been reached the extrusion behavior is measured over a range of screw speeds. Output, screw speed and melt temperature are all measured and recorded. Samples of the extrudate may be kept and identified for subsequent examination to reveal defects such as sharkskin and melt frac-

ture. The temperatures are then changed and the procedure followed again. Graphs are plotted of output, melt temperature and die swell against screw speed. The shear rates and stresses that cause problems, then can be calculated from the equipment dimensions and the output rate.

Such an extruder can also be fitted with different dies and ancillary equipment so that cast film can be produced at the shear rates where production problems occur. However, even the use of a small extruder is expensive (in terms of both capital outlay and running costs) and so, most studies are performed on capillary rheometers (See Flow Property Assessment Section).

#### Die Swell

Die swell is usually measured on samples of circular cross-section that have been extruded downward from a capillary rheometer fitted with a circular die. Accurate measurement and interpretation of die swell measurements requires care since, the weight of the extrudate may cause drawing down of the extrudate. To ensure that a circular cross-section is obtained from the die, the extrudate must be given time to freeze while suspended, since it can distort if it is allowed to lie on a surface before it has hardened. Drawing down can be avoided by extruding into a bath containing a liquid of the same density as the polymer, which is placed immediately below the die.

Draw down minimization may also be done graphically. Measurements of extrudate diameter are made at several points away from the leading end of the extrudate (obtained by cutting the extruded sample at the die outlet). By means of a graph, the theoretical die swell at this leading edge may be found by extrapolation. Somewhat more simply, there is little loss in accuracy if the experimenter always makes measurements at the same distance from the leading edge. The distance chosen should be the point at which the die swell is approximately a maximum.

If the extrudate is smooth and circular, then extrudate measurements may be made using a projection microscope. A laser micrometer, however, allows very accurate measurements of both the extrudate and of the die. If the sample is not round and smooth (due to sharkskin, melt fracture or some other problem) the best procedure is to weigh (M) a known length (L) of the extrudate and from a knowledge of the density ( $\sigma$ ) calculate the extrudate diameter (D) by the formula D<sup>2</sup> = 4M/ $\sigma$ vL. For amorphous materials, it is satisfactory to use the density of the solid polymer. In the case of crystalline polymers, however, the density will depend on the rates of cooling from the die and for accurate measurement the density should be obtained from the extruded sample.

## **Anisotropy and Orientation**

As the result of the rapid cooling occurring in production processes, such as injection and blow molding, the moldings usually contain molecules which are orientated in the flow direction: this means that there is a "grain effect" present like the one that exists in wood. Because of the grain, the wood is stronger in one direction that it is in another, that is, the products have anisotropic properties. An easy way of quantifying the anisotropy, or orientation, is to cut samples from the product, at two directions at right angles to each other (along the flow and across the flow). Then measure a property which varies significantly with orientation, such as flexural modulus or tensile strength.

Impact strength is also affected by such frozen-in orientation. In an Izod impact test, where the sample has been injection molded with the gate at one end of the sample, the molecules will be roughly aligned with the long axis of the sample. Thus to break the sample, in a standard Izod test, would

require fracture to occur across the elongated molecules. This will give a higher impact strength than would be recorded with non-oriented samples. On the other hand, if impact strength is being measured by dropping a weight onto a flat plate, lower impact strengths will be recorded with more oriented moldings. This is because fracture can occur more easily parallel to the direction of orientation and allows fracture to occur between, rather than across, molecules.

Shrinkage caused by oven heating is also used to assess the amount of stress relaxation that will occur on heating a plastic molding or extrusion product. Such a test is used as a measure of how well the product was produced and/or, if a product will withstand use at elevated temperatures. The greater the orientation the more the component will shrink on heating. The dimensions of the product are checked after oven heating for a specified time and at a specified temperature (See Stress Relaxation in Section 2).

## CHEMICAL RESISTANCE

#### Guidelines

Generally, plastics demonstrate a good resistance to chemical attack. Of course this resistance is dependent on the plastic and the nature of the chemical agent. The reported resistance of a plastic, to attack by a chemical agent, should only be considered as a guideline. There are frequently many grades of the same material and some may display better resistance than others. In general, the chemical resistance of semi-crystalline thermoplastics is superior to that of amorphous resins. With all plastics the chemical resistance is reduced as the temperature is increased.

## **Environmental Stress Crack Resistance (ESCR)**

The short term chemical resistance of most plastics is generally well known. The long term effect of exposure to various chemical environments, sometimes referred to as Environmental Stress Crack Resistance (ESCR), however, often shows quite different results. While a plastic may appear resistant to a particular environment, such as water, a long term exposure (~100 hours or more) may show a totally different result. Even if it survives this test it may fail if the sample is under stress. The only way to obtain this information is by doing the test, usually by immersion or direct contact with the chemical agent. Sometimes the samples are stressed on special jigs. The resistance is then reported as the time to first appearance of cracking or degradation and the calculated stress on the part.

With metals the attack of a chemical agent is usually confined to the surface and involves corrosion and loss of weight. Plastics, however, may absorb the chemical reagent and display swelling and softening, accompanied by a gain in weight. In extreme cases, this may be followed by a loss in weight as the surface is dissolved, swollen or decomposed and crumbles away.

## > ELECTRICAL PROPERTIES

Some of the earliest applications for plastics was as electrical insulators. They have excellent insulating properties as well as being easily shaped.
# Resistivity

Most plastics are good insulators and have a high resistance to the passage of electrical current. Since the resistivity of the surface and bulk of a plastic may be different they are both quoted. In both cases, the higher he value, the better the insulating qualities. A good conductor, such as gold, has a volume resistivity of  $10^{-6}$  (Ohm-cm), while carbon is  $10^{-3}$ , conductive plastic ~ $10^{-2}$ , cellulose  $10^{-6}$ , PVC  $10^{-14}$  and polystyrene is  $10^{18}$ .

Insulation resistance, a combination of surface resistivity and volume resistivity, is also sometimes quoted. It is the ratio of the direct current voltage applied to the electrodes to the total current between them.

## **Dielectric Strength**

Dielectric strength is a measure of the maximum electrical load a material can withstand before failure. To test dielectric strength, increasing levels of voltage are applied to a sample until fails. It is defined as the voltage per unit of thickness that will cause a catastrophic failure of the insulating material, resulting in the flow of current through the material. It can depend on thickness. For example, the dielectric strength of a 0.001 inch film may be as much as twice that for a 0.005 inch film of the same material (25.4V/mil = 1 KV/mm). The value decreases rapidly with increasing AC frequency and for some materials the value is decreased by increasing humidity.

# Arc Resistance or Tracking

While dielectric strength measures the maximum voltage a material can withstand, and volume resistivity measures it's resistance to the flow of electricity per unit thickness, arc resistance measures a material's resistance to an applied voltage over time. It measures the time, in seconds, that an electrical spark can be applied to a material's surface before it chars. Charring is the cutting of a carbon track across the material's surface, which causes it to become conductive. Plastics that degrade on heating to produce volatiles (gases) show a higher arc (or tracking) resistance than those that do not.

## **Dielectric Constant**

The dielectric constant (also referred to as the permitivity or specific inductive capacity) is a measure of how well a material will store an electrical charge. The lower the value the better the insulating properties. It is the ratio of the storage capacity of a material relative to that of air and has no dimensions.

## **Power and Dissipation Factors**

For most insulators (dielectrics) these two factors are the same and refer to a measure of how much power is converted to heat. This generation of heat is undesirable in an insulator and should be as low as possible. The term loss factor (or dielectric loss index) is the product of the dissipation factor and the dielectric constant. As a result, PVC, which has a high loss factor can be vibration welded, while PE, with a low loss factor, cannot.

## **Improving Conductivity**

For insulation purposes the high resistivity of plastics is an advantage. However, it also has the disadvantage of the capacity for building up a high static charge. This can in turn lead to spark generation or attraction of dust. This can be eliminated by the addition of a conductive filler such as carbon black. The incorporation of lubricants can minimize the generation of static, while the addition of some semi-incompatible liquids can cause static to leak away.

## > OPTICAL PROPERTIES

Optical properties is a term often used in plastics technology to describe those tests used to characterize the appearance of a film or sheet. The properties commonly measured include refractive index, clarity (usually means seethrough clarity), gloss, haze and light transmittance. The melt and die surface temperatures employed in extrusion may have a significant effect on properties such as surface gloss. In general, the higher the melt and die surface temperatures, the better the surface gloss.

#### **Refractive Index**

The refractive index is the ratio of the sine of the angle of incidence (sin  $\alpha$ ) to the sine of the angle of refraction (sin  $\beta$ ), when light is refracted from a vacuum into a medium. (Air is often used in place of a vacuum.) As the refractive index varies with the wavelength of light, the wavelength of light used for the measurement is taken as being that for yellow light (sodium D-line = 589.3 nm) unless otherwise stated.

The refractive index may be measured approximately by using a microscope: the opposite faces of a uniformly, thick test piece is determined by focusing on opposite faces alternately and measuring the traverse distance. Refractive index is obtained by dividing the true thickness by the apparent thickness. According to ASTM, the preferred method of measuring the refractive index is by use of an Abbé refractometer. A small test piece is kept in touch with the fixed prism with a suitable liquid: the ASTM procedure suggests suitable liquids. White light and a test temperature of 23°C are used.

#### Gloss

Gloss is a measure of the ability of a material to reflect light. It is an optical feature of all materials whether transparent or opaque. When a beam of light strikes a surface, the amount reflected will depend not only on the type of surface finish, and refractive index of the material, but also on the angle of incidence and the angle of viewing of the reflected beam. Because of such variations, many methods of measuring gloss exist, varying mostly in the angle of incidence and reflection used.

The primary standard of gloss is usually a highly polished, plane, black glass surface with a refractive index of 1.567. This standard is assigned the value of 100 gloss units for each of the geometries, or arrangements, of the incident and reflected light beams. For example, in ASTM tests, 20° and 85° (to the vertical) are used. The 20° angle is used for high gloss materials and the 85° angle is used for low gloss materials.

In many instances tests are made on specimens cut from finished products. No further preparation is usually necessary unless test temperature and humidity are known to affect the result (when careful conditioning prior to testing will be needed). Specimens made for comparison by injection and/or compression-molding need to be made very carefully as the result may be affected by many factors. The following are important in injection molding: Mold temperature, the rate of mold filling and mold finish.

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# Light Transmittance

The transparency of a material is defined in terms of two parameters or measurements. These are its light transmittance and haze. Light transmittance is a ratio obtained by measuring how much light passes through a sample of a material compared to how much light is passed without the material. Light that is not transmitted is reflected, scattered or absorbed. The upper limit for light transmittance achieved by high quality acrylic sheet is about 92%.

Light transmittance is measured by placing a flat test specimen in front of, and at right angles to, a collimated (parallel) beam of light. A photocell measures the amount of light passing directly through the material. The specimen is then removed and the amount of light reaching the photocell is measured again. The ratio of the two measurements, expressed as a percentage, is the light transmission.

When the specimen is thin and flexible (thin film rather than rigid sheet) an integrating sphere haze-meter must be used. This consists of a light source and lens which gives a collimated beam that passes through the specimen into an integrating sphere. The inside of the sphere is coated with a highly reflective white powder. The sphere can be moved so that all the light entering it either passes into a light trap, or falls on the wall of the sphere where it is repeatedly reflected until it reaches a photocell. Output from the photocell is displayed on a galvanometer.

Light transmittance measurements are made by first setting the instrument so that without a specimen in position, the galvanometer reads zero (that is, all the light passes into the trap) and 100 when the sphere is tilted and all the available light reaches the photocell. Interposition of the specimen causes the galvanometer reading to fall to a value of less than 100, and this figure is the light transmittance percentage.

#### Haze

Haze is the amount of light scattered in a forward direction, either by surface irregularities and/or non-homogeneities within a translucent or transparent material. The latter can be such things as dust, filler particles, or crystalline regions of different refractive index from the rest of the material. When the percentage of haze is greater than 30% the material is considered to be translucent rather than transparent.

It is possible to distinguish between haze due to surface roughness from that due to non-homogeneities by coating a small area of the specimen on both sides with a liquid of the same refractive index as the polymer (for instance, 1.49 for polypropylene). If the transmission in this wetted area improves, then the haze is mainly due to surface imperfections.

After a light transmittance measurement, and with the specimen still in position, the integrating sphere haze-meter is set so that all the available light reaches the photocell **(See Light Transmittance)**. The galvanometer reading is increased from its value below 100 to read 100 exactly by using the sensitivity setting of the galvanometer. The sphere is then moved so that all the light except that scattered by the haze passes into the light trap. The scattered light or haze then registers on the photocell, which outputs the value directly on the galvanometer. Haze is the percentage of incident light that is scattered by more than  $2.5^{\circ}$  from the original, incident beam.

#### See-Through Clarity

See-through clarity is defined as the ability to see objects, or print, which are in contact with a thermoplastics film. It is a measure of the distortion and/or 70 obscuration of an image when seen through a material. Long distance viewing is often not possible when looking through many plastics films while contact viewing is, however, often possible. See-through clarity may be measured by assessing the optical definition of, for example, Snellen charts at a specified chart-to specimen distance. Such charts consist of sets of parallel lines that differ in line spacing. The charts are viewed with and without the specimen. The narrowest, most closely spaced lines, which can clearly be seen as lines, with and without the specimen in front of the chart is a measure of see-through clarity.

The see-through clarity may also be assessed by judging the definition of a well-illuminated, wire grid when viewed through a film. The view of the grid is compared with a set of eight photographs that cover the range of clarity usual with the material. The test film is then assigned a number (which corresponds with the photograph number that resembles what is seen). Another way of assessing see through clarity is by small-angle scatter. This assumes that small-angle scatter controls the definition of an object.

The small angle test uses a stabilized light source to illuminate a hole which is 2.03 mm (0.08 in.) diameter: another hole of the same diameter is located 1.98 m (78 in.) away. A lens located approximately halfway between the two holes, focuses the light from the first hole (the illuminated hole) onto the second hole. When the film is placed behind the lens, that is, closer to the second hole, only light which is transmitted by the film with a deviation of less than 4 minutes is measured by a photocell located behind the second hole. The photocell reading, expressed as a percentage of the intensity of the incident beam, is called the see-through clarity.

# > MATERIAL IDENTIFICATION

Complete identification of most polymer compounds is very difficult because of the complexity of the formulations. Often, however, what is required is the separation (or identification) of lots of material whose identity has been lost or an identification of the type of material used to make a molded part. Useful simple tests to identify plastic materials include a preliminary examination, density, melting point and behavior on heating.

#### **Preliminary Examination**

The material should be inspected before processing and the color, particle size, particle size consistency and any obvious contamination, noted. If a component is being examined then note size, weight, color and any features, or clues, which indicate how the component was made. By way of illustration, a gate scar would indicate injection molding whereas the presence of flash lines would indicate manufacture by compression molding. Such preliminary examinations may indicate if a material is a thermoplastic material or a thermoset.

#### Density

The densities of most commercial thermoplastic materials are low as they are usually based on carbon and hydrogen. Density is defined as mass per unit volume and therefore has the units of mass and of volume. When expressed in grams per cubic centimeter (g/cc) it is numerically equal to relative density (specific gravity or SG).

Very often the absolute density of a material is not required. What is required is an approximate value so as to assist in material identification.

An approximate measure of specific gravity/density may be obtained by observing whether the material sinks or floats in a limited range of liquids, for example, water and saturated magnesium chloride. The former has a density of 1 g/cm<sup>3</sup> and the latter has a density of 1.34 g/cm<sup>3</sup>. Most olefin-based materials will float in water (and therefore also in saturated magnesium chloride). Most unfilled, styrene-based thermoplastics will very slowly sink in water, but will float in the saturated magnesium chloride. Most UPVC materials will quickly sink in water and more slowly in saturated magnesium chloride. This test is most useful when performed in conjunction with a burning test.

If an accurate measurement of density is required, then a flotation or displacement method (See Density and Dimensions Section) can be used.

## **Melting Point**

The simplest way of obtaining an approximate value is by heating a small sample of the material on a metal hot plate while measuring the temperature. A heating rate of approximately 50°C/hour or 100°F/hour, should be used. It is useful to have a glass rod so that the sample may be moved, or prodded, during heating. Note the mid-point of the obvious softening range. An amorphous thermoplastic material will not have a sharp melting point, whereas a semicrystalline thermoplastic will usually have a relatively sharp melting point **(Table 7.)**.

#### **Behavior on Heating**

Often this test may be combined with the melting point test. A glass rod is used to move the material while it is being heated. If the material does not soften on heating then it is probably a thermoset (some heavily filled engineering thermoplastics behave in the same way). If it does soften then it is probably a thermoplastic. An amorphous thermoplastic will have a much broader softening range (not a melting point), whereas a semi-crystalline thermoplastic material will have a melting point. The way that the material burns and the fumes evolved may also indicate the type of material used.

# SECTION 4: THE SCREW AND BARREL SYSTEM

- MATERIALS HANDLING
- ► THE HOPPER
- ➤ THE BARREL
- ➤ THE SCREW
- SCREW TYPES
- SCREW MIXING SECTIONS
- BREAKER PLATES, SCREEN PACKS AND GEAR PUMPS
- ≻ SCREW DRIVE SYSTEM
- MOTOR SIZE AND THRUST BEARING LIFE

# MATERIALS HANDLING

The subject of materials and component handling is one that appears to be ignored in many extrusion shops. Thus, material and component contamination is common. The most common source of resin contamination is water. Generally oil, grease and dust are observed in the contamination of products as well.

#### **Material Feed**

The feed to machines involved in processing thermoplastics is very often a mixture of virgin (new) material, regrind, and colorant (often in the form of a master batch). All of these materials must be kept clean and dry. A controlled ratio of the materials must also be used if consistent machine operation and component quality (such as surface appearance) are to be maintained.

The extruder can be fed with plastics (resins) or compounds in various forms. The feed may be fine powder, regrind material or virgin pellets. If the material is available in more than one form, feeding problems will probably occur if a mixture of forms is used. In terms of feeding efficiency, spherical granules (of approximately 3 mm/0.125 in diameter) are the most efficient, while fine powders are usually the worst. Frequently, regrind material can be almost as difficult as fine powder. Cube-cut granules are better and lace-cut (or face-cut) granules are better still.

As a result of feeding differences, machines must be fed with a consistent raw material mix or a pre-blended masterbatch. The use of a masterbatch mixture can produce significant cost savings as a compounding step may be eliminated. However, the use of such a mixture can sometimes cause problems. There may be a color shade differences between the same material processed on different machines. Another problem is the separation of the components of the masterbatch in the hopper.

#### Stock Control

All storage and unloading areas must be kept clean and dry to minimize fire hazards. Storerooms should be separated from the processing area by fire resistant doors. The materials should be stored away from direct sunlight and on properly constructed racks. Usually, the use of unheated storage areas, with natural ventilation, is sufficient. To ensure that the material does not stagnate in the stores a strict first-in, first-out policy (Fi-Fo) policy is wise.

Strict stock control is important. Within a shipment of material there can be several different batches and one of these may be faulty. By adopting a strict stock control system, a faulty batch can be readily identified and isolated, thus preventing defective products from being produced.

#### **Condensation Minimization**

Many thermoplastic materials are supplied in pellet or granular form in sacks or bags. Before use, it is important that the granules inside the sack reach the temperature of the workshop to prevent condensation from forming on the surface of the granules. Even if a material is said not to absorb water, it is recommended that the material be stored in the extrusion shop for at least 8 hours prior to opening the sack. This advice applies particularly during the colder months and at times of high humidity. At such times, it is advisable to store the day's requirements in the shop.

Before opening the bag, wipe it clean to avoid dust being transferred with the material when the bag is tipped or inverted. After the bag has been opened and the required amount of polymer removed, reseal it immediately and label the bag with the amount remaining, the date and your signature. Only put sufficient material into the hopper for about 1 hour of operation and keep the hopper covered. Wherever possible use dried material and a hopper heater or a direct feed loader.

#### **Hygroscopic Behavior**

If a polymer or compound contains water or another low boiling point material, the temperatures seen in many extrusion processes will be above its boiling point. Visible bubbles will then form within the thermoplastic as the external pressure falls when the material emerges from the die of an extruder. Generally speaking, the higher the processing temperatures, the lower the amount of water (or other volatiles) that can be tolerated. This is because the higher temperatures will generate larger volumes of steam from the same quantity of water. The water may be introduced into the compound by additives that contain excessive water. Usually commodity thermoplastics do not suffer from water-related problems. Engineering thermoplastics, however, do often absorb water; that is, they are hygroscopic.

## Drying

While many processors have drying facilities, they rarely check the moisture content of the material, but dry it for a set number of hours at a particular temperature. Such a drying procedure may not always be satisfactory, as many factors affect the drying process.

It is a given that the higher the initial moisture content, the longer the time needed to reach the required low moisture content or limit. Therefore, there is a need to measure the moisture content of the material at various stages in the cycle; for example, as supplied and after drying. A generally standard measure of how much water a material will absorb is given by the amount it will absorb in 24 hours at room temperature. If the value is greater 74 than approximately 0.1%, drying is usually necessary before extrusion. If it is lower, drying is not normally required.

Drying should be done in a hot air oven, desiccant drier or in a vacuum drier. The last two methods are the more efficient as they are faster and reduce the water content to lower values. They take approximately half the time required by a hot air drier and, for some resins, they are the only way of getting the material dry.

## **Consistent Drying**

Many virgin thermoplastic materials do not require drying before processing; that is, they are supplied dry and are ready to use. However, hygroscopic materials need to be dried before melt processing. To reduce problems, in general, it is suggested that all materials be dried to predetermined moisture content levels as a matter of course. After drying, the material should not be placed into an open hopper for extended periods, as it will reabsorb moisture. Ideally, material usage should be calculated to determine how much should be loaded into the hopper to prevent storage for an extended length of time. Where space is at a premium and storage in the hopper is necessary, then a hopper heater should be used to prevent re-absorption of water. The region of the barrel under the hopper (zone 0) should also be held at a known, elevated temperature.

#### Table 14. Suggested Drying Conditions for Extrusion Materials

Water Abs.	Hot	Air Drying	Desid	Desiccant Drying	
(%)	(°C)	(# of hours)	(°C)	(# of hours)	
0.2-0.35	70-80	2-4	70-80	2	
0.08	70	2-3	70	1-2	
<0.2	65	3	80	1-1.5	
<0.01	65	3	80	1-1.5	
<0.2	65	3	85	1-1.5	
<0.2	65	3	85	1-1.5	
1.6	80	16	105	12	
1.5	85	16	105	12	
0.3	75	2-4	90	3-4	
-	-	-	-	-	
<0.2	65	3	80	1-1.5	
	Water Abs. (%) 0.2-0.35 0.08 <0.2 <0.01 <0.2 <0.2 1.6 1.5 0.3 - <0.2	Water Abs. Hot   (%) (°C)   0.2-0.35 70-80   0.08 70   <0.2	Water Abs. Hot Air Drying   (%) (°C) (# of hours)   0.2-0.35 70-80 2-4   0.08 70 2-3   <0.2	Water Abs. Hot Air Drying Deside   (%) (°C) (# of hours) (°C)   0.2-0.35 70-80 2-4 70-80   0.08 70 2-3 70   <0.2	

(Based on drying conditions for Injection molding materials)

#### **Materials Management**

Material provides the biggest single cost of producing an extrudate. We do not want to waste time and material by producing rejects. Even if the material can be reclaimed, we will have lost production capability and wasted money.

Machine manufacturers produce machines that can be started up quickly and which maintain consistently the preset operating conditions. The die and haul off have been designed and manufactured so that they produce extrudate at the required rate producing the minimum amount of regrind. Such efforts will be wasted unless you play your part by looking after the material.

What does this mean? It basically means that one should keep the material clean, dry and free from contamination. Remember and apply the following:

- Only open sacks when they have reached workshop temperature
- Only use material when you have double-checked that it is the correct type and grade
- Check to see that the machine has been set correctly for that material
- Reseal containers immediately after use and check that the material is labeled correctly
- Ensure that regrind is kept clean, dry and used in the correct proportions

#### ➤ THE HOPPER

The hopper is the material holding unit attached to the barrel at the feed port. It is used to hold and feed plastic material into the extruder (See Figure 1).

#### **Conical Hopper**

Usually the hopper is shaped like a tapered cone, the smaller end of which is attached to the hopper block on the barrel. The larger end, through which it is filled, is closed by a lid. At the base of the hopper is the hopper slide (throat gate) which can be used to block the material feed into the machine.

The taper angle of the hopper (usually between 20° and 45°) must be greater than the angle of repose of the material being extruded to reduce the risk of bridging. The inner surface of the hopper must be smooth and highly polished to minimize material stagnation and to prevent any material from adhering to the metal surface. Often the hopper is made from aluminum although stainless steel is preferable.

Simple gravity feed is most often used with a conical hopper, unless the material does not flow uniformly. To avoid material feed problems, such as bridging, the hopper may be vibrated and/or the material may be stirred by means of a rotating arm. It is desirable to keep the level of material in the hopper as constant as possible to assure a constant output from the die. The material temperature should also be kept as constant as possible using a heated hopper assembly.

#### **Hopper Block**

The hopper block is that part of an extruder that supports the feed hopper and contains the throat. To ensure that material freely enters the feed section of the screw, cooling water is generally passed around the hopper throat assembly. The flow rate and temperature of the cooling water is important, as these variables affect how the material is converted from solid to melt (plasticized) within the barrel. Usually, the water passing around the hopper throat is from the local supply and is, therefore, subject to a seasonal temperature variation. This variation can affect the melt temperature and cause product variation. To overcome this problem a temperature control system (similar to a mold temperature controller) can be installed with the flow rate and temperature of coolant standardized for each machine. Alternatively, a solenoid-actuated valve fit into the coolant supply can be used to provide water flow when the temperature exceeds a preset value.

#### **Hopper Heater and Drier**

A hopper drier is used for drying a material, (for example, an engineering thermoplastic) before, melt processing. Heated air is blown through the base of the hopper and filters through the material. Part of this air is bled-off during the drying process to prevent it from becoming saturated with moisture. With such a system, drying times are measured in hours (See Table 6.), but this can be reduced by the use of a desiccant drier or vacuum drier. Because of the relative inefficiency of many hopper dryers it is best to refer to them as hopper heaters. Such hopper heaters should be used to keep a material hot before melt processing. The material may have been dried in another system and the heated hopper simply acts to prevent the regain of moisture and to maintain material temperature.

#### **Hopper Loading**

Although a hopper may be loaded with material manually, it is easier, safer and more efficient to use one of the commonly available alternatives such as a negative pressure (or vacuum) hopper loader. This may be connected to a floor-mounted material bin via a conveying tube through which the applied vacuum draws up the material to the receiver. Once the receiver is filled, the vacuum application is stopped and the material discharges by gravity. Some negative pressure hopper loaders have a reverse flow feature to pressurize the vacuum receiver and break up potential bridging. Some of these hopper loaders, designed to handle dusty materials such as regrind, are known as powder loaders.

The hopper may be loaded using timed loading cycles or filled to a predetermined level by means of a level control which senses a full receiver and switches off the conveying action to save energy. When materials are conveyed in automatic systems, the dangers of dust generation, and dust inhalation, should be recognized and all equipment must be properly ground-ed.

It should be noted that individual machines may also be fed from a centralized supply (a bulk handling loading system). An augur conveyor (flexible worm hopper loader) is used to convey dusty or difficult-flow materials over short distances. Such conveyors can require considerable cleaning between material changes.

#### **Material Measurement and Blending**

The blending of materials is now common, as many machines run on a mixture of virgin material and regrind. Most systems use volumetric blending where the ingredients of a blend are measured by volume and then fed either to a separate blender or to a blender hopper on the machine (in a blender hopper the material may fall through a series of baffles to cause blending). An accuracy of approximately 0.1% is achievable by volumetric measurement.

Alternatively gravimetric blending may be used. With this type of system the ingredients of a blend are measured by weight. For example, a receiving vacuum hopper may be mounted over a blender supply hopper that is mounted on load cells. As material is metered away from the supply hopper, the amount being fed to the extruder can be weighed by difference. Gravimetric measurement can provide a guaranteed accuracy of 0.05% to 0.005%.

# ► THE BARREL

The barrel of an extruder is a hollow chamber in which the screw (or screws) operates **(See Figure 1.)**. If one screw is used the barrel has a cylindrical cross-section: if two screws are used, the barrel has a figure eight (8) cross-section. It is the screw and the barrel, which interact to melt, convey, and generate pressure on a polymeric material within an extruder. It is essential that this be done under controlled conditions since the extruder must produce a uniformly plasticized material, of constant composition, at the required rate.

To achieve this, the barrel must be made very accurately. The total out-ofalignment error, after machining, must be less than one half of the screw/barrel clearance. A barrel is generally rated in terms of its diameter (D) in inches or millimeters.

#### **Barrel Construction**

Because the pressures generated within an extruder can reach very high values (up to 10,000 psi or 70 Mpa), the barrel is constructed to withstand these high pressures without distortion. The barrel, therefore, is made from thick walled alloy steel tubing or pipe and is usually designed to operate safely at twice the estimated operating pressures. Wear and corrosion resistance are often provided by ion nitriding, although a bimetallic barrel is preferred for wear resistance.

The feed throat, cut through the barrel wall, is approximately the same size as the barrel diameter. It is very important that adequate provision is made to cool the barrel in this region, as otherwise the extruder will not feed properly. Provision is made to attach the adapter and die at the discharge end of the barrel.

#### **Barrel Wear**

Frequently, there is an increase in the clearance of the screw/barrel system, especially in the hopper region, over time of operation. The screw and barrel assembly operates in a very aggressive environment that may cause severe wear problems. As material modification becomes more common with abrasive materials, like flame retardants and fillers (such as glass), being added to more plastic materials, wear has become a major problem. As it is preferable to replace a screw than a barrel, the barrel must be harder than the screw.

To improve the wear resistance of the barrel (which is required mostly in the transition zone) it may be modified or lined. Modification may be by processes such as nitriding or ion implantation, but these treatments are not as good as lining (which gives a bimetallic barrel). This produces a barrel that is based on two different metallic materials, in which one metallic material surrounds the other. The inner layer, in contact with the melt, possesses good wear and/or corrosion resistance characteristics and is about 1.5 mm thick (the thickness is approximately 2 mm thick for a grooved barrel). This layer may be based on one of a number of materials or alloys such as a nickel alloy matrix reinforced with particles of 25% iron boride or of tungsten carbide. The inner liner is cast inside a flanged steel backing cylinder of a high quality tensile steel (AISI 4140), which resists crushing and distortion. An outer assembly, generally a cast aluminum housing, contains heating elements, cooling channels and pockets for the temperature sensors. The barrel may be made in sections, the number of which corresponds to the number of barrel zones.

#### **Barrel Heating and Cooling**

Most extruders are electrically heated and use resistance coils, bands, or cuffs that are strapped or bolted around the barrel. Upon demand, initiated by a thermocouple, electrical current is passed through the resistance wire, inside the coil. The resistance produces heat that causes the temperature to rise. If a PID controller is used, as the set point temperature is approached the power is progressively reduced and then finally turned off completely at the set point. For a given machine, the actual barrel settings required to achieve a desired melt temperature will depend on the screw rotational speed, the pressure within the system and the polymer throughput.

As it is very easy to exceed the extrusion set point temperature, many machines are equipped with cooling systems. Air cooling systems are the simplest to install and consist of fans that circulate air around the barrel on demand. These are not as efficient as liquid cooling systems. A popular system is based on a closed loop, heat exchanger that uses distilled water contained inside a sealed coil surrounding the barrel. When the set point is exceeded, the vapor from this water is cooled by water flow so that the distilled water vapor condenses to absorb more heat.

#### **Zoned Heating**

A zone is a part or section of an extruder barrel. For ease of control, the barrel is divided into zones or regions. Zoned heating is the separate heating, and temperature control, of each zone of a machine. The smallest machines usually have three zones while larger machines may have as many as twelve. A temperature sensor and its associated electronics, (such as a microprocessor-based, three term (PID) controller), controls each of these zones.

#### **Barrel Temperature Settings**

These are the temperatures set on the control instruments. It is the melt temperature, however, that is important and any barrel temperatures quoted (in the literature) are only guidelines. When there is no experience in the processing a particular grade of material, it is best to start with the lowest settings recommended. Usually the first zone temperature is set at the lowest value as this helps prevent premature melting and bridging of the resin in the feed throat. The temperatures of the other zones are gradually increased until the die is reached. In extrusion processes the die temperature is sometimes set slightly higher to put a higher gloss on the product.

As it is easier to heat a barrel than to cool it, it makes sense to approach the final operating temperatures slowly to minimize the risk of temperature over-shoot. Setting the temperatures 20°C/30°F below the run settings during initial barrel heating most easily does this.

#### **Barrel Residence Time**

This is the average time during which a material resides within the extruder barrel. It is important because time at temperature controls of the rate of decomposition of plastics. A plastic may be degraded by a short exposure to a high temperature or by a longer exposure to a lower temperature. How long the plastic is in the barrel is therefore important. The actual residence time may be determined experimentally by measuring the time taken for colored plastic to pass through the extruder.

#### **Vented Extruder**

The barrel of a vented extruder contains a vent or port that is used to reduce the water (moisture) and/or volatile content of a plastic material. The melt is decompressed partway along the barrel by reducing the screw root diameter. At this point in the barrel a vent is located to allow the vapor to escape (the vent may be plugged when venting is not required). The vapor-free melt is then conveyed towards the die and re-compressed by increasing the screw root diameter. To ensure that melt does not escape through the vent, a dam or torpedo section is incorporated on the screw just before the vent. As a result of the way that vented machines are constructed (high L/D ratio and variable screw geometry) and operated they produce very good mixing. It must be remembered, however, that the action of heating some plastics in contact with water can cause decomposition, or degradation. In some cases a vent may be used to add something to the barrel. For example, a relatively high loading of filler may be added to a plastic material if it is first softened in the screw/barrel assembly. Thus the filler may be added partway down the barrel via a vent or vents.

#### **Grooved Barrel Extruder**

This is an extruder that contains grooves in the feed section of the barrel. It may also be referred to as an axial grooved barrel extruder as the barrel contains axial grooves. The barrel contains a number of grooves running parallel to the screw that improve the feeding characteristics of the system. The grooved section is of the order of 3 to 4 D in length and tapers out as it progresses along the barrel. Temperature control of the grooved section is important. It is generally held at a temperature of 70°C by intensive circulation of water. To decrease heat generation, a zero compression screw may be used.

By using a grooved feed throat, the output of the extruder is increased (compared to a machine with a plain barrel), but there is an increase in power consumption (approximately 10% more). The output, however, is virtually independent of the back-pressure. The peak pressure within the barrel often occurs at the end of the grooved section. A grooved barrel extruder is said to be driven from the back – as compared to an extruder fitted with a gear pump that is said to be driven from the front.

The use of axial grooves and a barrier screw produces a machine that can uniformly plasticize plastics at high outputs and comparatively low melt temperatures. A lower melt temperature improves the capacity of the downstream cooling equipment and thus allows an increase in the output.

#### ► THE SCREW

The most popular type of extruder is the single-screw type machine. In this extruder it is the screw and the barrel that interact to convey, melt and generate pressure with a plastic material. It is required that this be done in a controlled way, since the die must be supplied with uniformly plasticized material, of constant composition, at a constant and controllable rate. For this reason, many single screw machines are fitted with mixing sections and/or a gear pump.

#### **Flow Mechanism**

The screw consists of a cylinder of steel with a helical channel cut into it. This screw rotates inside the extrusion barrel and conveys the plastic material from the hopper to the die along the screw channel (the open section between the flights of the screw) (Figure 13.).

In the ideal case, as the screw rotates, the material slips on the screw and adheres to the inside the barrel, producing a purely axial (forward) movement of the polymer. If the material sticks to the screw and slips on the barrel then there will be zero output, as the material and the screw rotate together. In practice, the flow behavior lies between these two extremes since the material adheres to both the screw and the barrel.

#### **Extruder Output**

As the polymer moves along the screw a thin film of the material initially forms on the barrel wall. The screw rotation scrapes this thin film of melted polymer away from the barrel wall and it flows down the front face of the



Figure 13. The Basic Extrusion Screw

screw flight. It is then forced (or dragged) along the core of the screw and up the rear face of the flight giving what is called drag flow. Drag flow, which is due to the interaction of the screw and the barrel, produces a circulatory flow in which the melt surrounds un-melted granules. Ideally the melt pool then expands until there are no granules left. The generation of uniform melt is promoted because the material flow is not purely axial. The material also rotates with the screw producing rotary flow and melt leaks over the screw flights (**Figure 14**).

The output from an extruder is the result of drag flow minus the sum of the pressure flow plus the leakage flow. Pressure flow, which opposes output, occurs because of the pressure built up at the die and leakage flow reduces output as melt leaks over the screw flight (Figure 15).



Screw

Figure 14. Polymer Melting on the Extrusion Screw

#### Length and Diameter

Screws are usually rated in terms of their diameter (D), measured in inches or millimeters. In order to allow rotation there must be a clearance between the screw and the barrel. There is, therefore, a difference in radius between the screw and the barrel. To illustrate, a 100 mm diameter extruder may be fitted with a screw of radius 49.9 mm or, 99.8 mm diameter. The screw clearance is the difference between the radii of the screw and the barrel.

To obtain the desired melt properties of uniform high temperature and a stable output the screw length (L) is, generally, relatively long (L is measured from the rear of the hopper to the front of the screw). Screw length is usually expressed as a ratio of its length to the diameter: the L/D ratio. The use of a long screw (an L/D ratio of, say, 28:1) gives a stable output, improves mixing and gives scope for the use of mixing elements.



Figure 15. Flow Profiles in the Extruder

#### Screw Zones

A screw may be divided into zones or regions. Typically a general-purpose screw has three distinct zones: the feed zone, the compression (plasticating) zone, and the metering (pumping) zone. The feed section, which conveys the material from the hopper, starts at the rear of the hopper and usually has a constant depth. In the transition zone, the root diameter of the screw increases as compression is applied. In the metering section the depth becomes constant again. The feed section, is generally about 50% of the length (50% L), the transition section is about 30% L and the metering section is 20% L.

#### **Compression Ratio**

The ratio of the volume of the flight in the feed zone to that in the metering zone gives the compression ratio. This reduction in volume compensates for the reduction in volume of the feed (solid material + air) which occurs as the

plastic melts and fuses together. Compression ratio is related to channel depth, so if the depth is 0.373" at the hopper and 0.125" at the screw tip, then the compression ratio is said to be 3:1. A typical compression ratio is 2.0/1 (or 2.5/1) and the length to diameter ratio (L/D) is about 20:1. The terms used to describe a screw are shown in **Figure 16**.

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When a three-zone screw is used to process a material, a large amount of heat may be generated as a result of the compression ratio. To avoid this heat generation, extruders are now being built that have screws with zero compression, that is, the depth of the screw flight is the same along its entire length. Such screws give very little mixing but they do prevent the generation of excessively high melt temperatures (melt temperature override). Mixing can be improved by the use of mixing sections, fluted sections, or mixing elements located near the screw tip. Such screws are widely used in extrusion blow molding where high melt temperatures must be avoided if high production rates are to be achieved.



L/D = Screw Length/Screw Diameter Compression Ratio = Channel Depth Start/Channel Depth Final

Figure 16. Elements of an Extrusion Screw

#### Helix Angle and Volumetric Efficiency

In extrusion, the helix angle ( $\phi$ ) is the angle between the screw thread and the transverse plane of the screw. For most screws the pitch of the screw equals the screw diameter and  $\phi = 17^{\circ}40'$ . The volumetric efficiency of the screw is dependent on the helix angle. If volumetric output rate is designated Q then, the volumetric efficiency of the screw may be expressed as

#### $Q_{max}/Q_{ideal} = 0.5 \cos^2 \phi$ (Eqn. 18)

Where  $Q_{max}$  is the maximum volumetric output rate,  $Q_{deal}$  is the ideal volumetric output rate and  $\phi$  is the helix angle of the extrusion screw. The volumetric efficiency depends on the helix angle only and in cases where the pitch equals the screw diameter ( $\phi = 17^{\circ}40^{\circ}$ ) then the volumetric efficiency is 45.4%.

#### **Shear Heat**

The energy expended in turning the screw and pumping the material forward is turned into heat. Because the material is being sheared, the heat generated is known as shear heat. The shear heat is not spread uniformly throughout the material, but is greatest where the shear rate is the highest. It can be high enough that it can cause localized overheating and material decomposition, or degradation at the tips of the screw flights.

For a given material, the amount of shear heat depends on the screw speed and on the screw diameter. If possible, the machine size should be matched to the anticipated output. It may be possible to achieve the required output with a small diameter machine running fast or with a larger diameter machine running more slowly. It is generally found that the larger machine will produce a melt of significantly lower temperature and higher outputs are achieved. A higher screw/barrel L/D is another answer, as this will give the same throughput as a lower one, but at a lower screw speed.

#### SCREW TYPES

Over the years many different types of screws have evolved for the processing of thermoplastics materials (**Figure 17**). Some are designed to improve mixing whereas others may be designed to suit a particular material or process. With the advent of cheap computing power, and a greater knowledge of the extrusion process, computer design of extrusion screws is now extensively used.

#### Tapered and Parallel Extruders (Figure 17.)

A very popular type of extruder contains a tapered parallel screw. A tapered parallel screw has a gradually tapering screw root (the channel depth gradually changes) in the compression zone. The feed zone and the metering zone are parallel and of constant depth. Unlike the double parallel screw, the velocity of the un-melted portion of the melting material bed is maintained and there is less risk of polymer stagnation.

The double parallel screw has a "step" that is, a rapid decrease in channel depth (over one screw turn or less), between the parallel feed and metering sections. In the "stepped" screw (a double parallel screw or nylon screw), it is claimed, that the rapid decrease in channel depth acts as a barrier to forward flow of un-melted granules and therefore fixes the melting point at the step.



Tapered Parallel Screw (with Decreasing Helix Angle)



Double Parallel Screw



Screw with Tapered Root Diameter over Full Length

Figure 17. Tapered and Parallel Screws

A tapered screw may be selected for heat sensitive materials (UPVC). This is a screw which has a gradually tapering screw root, that is, the channel depth gradually changes – usually the diameter of the screw root increases steadily from the hopper end to the die end of the extruder so as to gradually compress the material.

#### **Poor Mixing**

A conventional screw does not provide very good mixing. This is because of the way that the material flows, or is transported, along the screw. Since the material towards the center of the flight can easily remain undisturbed, the output from the machine will not be of uniform quality. It will be inhomogeneous as it has a non-uniform shear history (even if nothing else happens, the melt temperature will be non-uniform). Ideally the system should break up agglomerates (provide dispersive mixing) and then produce a random spatial distribution of any dispersed material (provide distributive mixing) (See Screen Packs Section and Mixing Section).

#### High L/D Ratio Screws

There is a tendency towards the use of longer extrusion screws with length to diameter ratios (L/D ratio) exceeding 24/1. Such long assemblies are used where high melt flow rates and excellent melt homogeneity are required, since they will produce the same throughput as a shorter screws but, at lower speeds. This is particularly important when high frictional heat generation must be avoided. The use of a longer screw/barrel assembly gives increased operating flexibility, as there is more opportunity for the use of shearing and mixing elements. Some extrusion screws are now as much as 32 D long.

## **Screw Cooling**

When screw cooling is used, the temperature of part or all of the screw is reduced or maintained, during machine operation, by circulating water from the rear end of the screw through a bore in the center of the screw. Screw cooling is done to resolve feeding problems (to improve operating consistency), to improve mixing and/or to minimize material decomposition. In some cases it may only be necessary to cool the screw in the feed section, while in other cases it may be necessary to bore out the whole length of the screw (almost as far as the screw tip). In the second case, the cooling water freezes a layer of material at the screw tip that effectively increases the compression ratio of the screw. Mixing efficiency is improved, but the output rate goes down and the power consumption goes up. A big disadvantage in both cases is that the actual temperature of the screw is not known. Screw cooling should be turned off before shutdown to minimize material adhering to the screw.

#### **Dedicated Screw**

A dedicated screw is a screw that is designed to suit one type of material, for instance, nylon (PA 66) or polyvinyl chloride (PVC). A general-purpose (GP) screw is designed to suit as wide a range of plastics as possible and is not the ideal answer for the processing of any specific material. For example, a screw designed for a semi-crystalline, thermoplastics material, such as PA 66, must provide a greater heat input than one designed for an amorphous, thermoplastic material. If a machine will be dedicated to one plastic for a long time, then it is worthwhile considering the purchase of a specially designed, or dedicated, screw.

## **Barrier Design Screw**

This type of screw (Figure 18.) contains a barrier to hold back un-melted material. The flights of a conventional screw are often filled with a mixture of solid and molten plastic. It is difficult for the screw to grip the solid resin



Figure 18. Barrier Design Screws

since it floats or 'swims' in a pool of melt. Barrier design screws are used to improve the ability of the machine to produce melt, and to give more uniform melt. The barrier screw has two separate flights that are separated by the flight land. These two separate flights may only run for part of the screw length. For example, it may run for 13 diameters (13 D), in place of the compression zone, on a 24 D screw. As the resin melts it is transferred from one flight to the other over the narrow land. Thus the screw separates the melt pool and solid bed. This produces improved output per rpm and a lowering of melt temperature.

#### Zero Compression Screw

A large amount of heat will be generated, when a conventional screw is used to extrude a plastic material, because of the compression ratio on the screw. To avoid this heat generation, machines are now built which have screws where the depth of the screw flight is the same along its entire length (zero compression)(**Figure 19**). Such screws give very little mixing, but this can be improved by the use of mixing sections located near the screw tip. Such zero compression screws are commonly used with barrels that have longitudinal grooves in the feed section. These grooved barrel assemblies are now considered essential for the processing of high molecular weight PE, and PP, at high throughput rates. These assemblies give consistent outputs even if the diehead resistance alters due to the use of parison programming in blow molding operations. For blow molding machines, such barrels are available in diameters of up to 90 mm (3.5") with L/Ds of 20/1 or greater (**See The Barrel Section**).

#### **Vented Systems**

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Vented extruders were developed in order to allow gas or vapor to be removed from a molten polymer. In some cases this gas is simply air, but it may also be a vapor given off by the plastics compound (water vapor, residual monomer, or gaseous degradation products). If this vapor is prevented from escaping it will be compressed by the incoming melt and carried to the die,



Figure 19. Zero Compression Screw (Variable Pitch)

where it will expand to produce bubbles in the product. To remove the gas before it reaches the die, the melt is decompressed partway along the extrusion barrel by reducing the screw root diameter partway along its length. A vent or port, through which the gas escapes, is located at this point in the barrel (**Figure 20.**) (The vent may be plugged when venting is not required). The vapor-free melt is then re-compressed, by increasing the screw root diameter, and conveyed to the die. To ensure that melt does not escape through the vent, a dam or torpedo section may be incorporated on the screw just before the vent



Figure 20. Standard Two Stage Screw for Venting

#### Wear Resistant Screws

Screw and barrel assemblies frequently operate in very aggressive environments that can cause severe wear problems. If the clearance between the flight and the barrel increases, then the pumping efficiency of the screw will decrease and temperature differences in the screw channel will increase. Usually the screw is made from a good quality tensile steel such as AISI 4140, which resists torque and distortion, and is oil quenched and tempered or hardened by induction heating. It is easier to replace a screw than a barrel. Thus the barrel must be harder than the screw.

The wear resistance of the screw may be improved in various ways. If the screw is deep nitrided to 67 Rockwell C, this will improve wear and give protection against chemical attack. It will also stop plastic from adhering, and then decomposing, on the screw. The screw need not be of the

same composition over its entire length, as those parts that are subject to the severest use can be given extra protection. For example, the screw may be constructed from 4140 heat-treated steel and the flights may be surfaced with a wear resistant alloy such as Colmonoy 56. Ion nitrided screws are also used, as are hard chromium plated screws. If the thermoplastic material evolves corrosive gases, as in the case of UPVC, then a stainless steel screw (AISI 316) may be specified.

#### **Computer Software**

Computer simulation software is available, which allows many different designs of screws and changes in operating conditions to be tested quickly, without the high cost of machining and running extrusion trials. Software is available which deals with solids transport, material melting, and melt pumping performance within a single screw extruder: These programs predict the presence of un-melted material and variations of temperature, pressure and energy along the screw. Such predictions can be used for screw design and to determine the effect of changes in material properties, screw speed and barrel temperature profile on the performance of the system.

# SCREW MIXING SECTIONS

Unless the screw in the single screw extruder is fitted with special mixing sections, it is unlikely that it will do a very good job of mixing. It is essential that mixing is done under controlled conditions, as production of uniformly plasticized material, of constant composition, delivered at a constant and controllable rate, is essential.

## **Uniform Feed**

The extruder must be fed with a uniform feedstock if a uniform product is to be obtained. This means that all of the ingredients must be accurately dispensed and well dispersed throughout the plastic material before the feedstock reaches the screw. For most materials this simply means emptying known weights of material into a conical blender and then adding a color masterbatch. A simple tumbling operation is then performed. This may be done continuously, on the machine, if the output is sufficient. For PVC, high-speed dryblending is usually used and the uniformly blended materials are then fed to the screw of the extruder for melt mixing.

#### **Poor Melt Mixing**

A conventional screw (for example, a three zone screw) does not give very good melt mixing because of the way that the material is transported along the screw. The material in the center of the flight can easily remain undisturbed, while the material at the edges is circulated and repeatedly sheared. This means that the output from the machine will not be of uniform quality, that is, it will be inhomogeneous, as it has a non-uniform shear history. Even if no mixing is required, the melt temperature will be non-uniform and this non-uniformity can cause product distortion and alterations in output due to viscosity variations.

#### **Cooling and Mixing**

The viscosity of plastic materials is higher at lower temperatures. In fact, for some materials, a slight variation in the melt temperature will have a very

large effect on the viscosity. If the melt viscosity is low, then agglomerates can escape the mixing action. Improved dispersive mixing is obtained if the melt temperature can be kept low and uniform. This means that barrel cooling must be very efficient and heat transfer must be good: particularly in those regions where dispersive mixing is being performed. A great deal of shear heat is generated in those regions.

#### Improving Melt Mixing

A simple way to improve mixing in a single screw extruder is to increase the density of the screen pack by adding fine screens (See the section on Screen **Packs**). However, the use of mixing sections, offers a more efficient way of improving mixing. Barrier design screws improve the ability of the extruder to produce a more uniform melt, as the screw has two separate flights separated by the flight land. As the resin melts, it is transferred from one flight to the other. That is, the screw separates the melt from the solid bed and produces an improved output per rpm and lower melt temperature. Mixing sections may also use this principle. For example the Egan mixing section uses barrier flights. Longer screws give improved mixing, but, perhaps, more importantly, they give scope for the use of mixing sections.

# **Dispersive and Distributive Mixing**

Ideally the extrusion system should reduce additive agglomerates such as pigment or impact modifier agglomerates. That is, it should provide dispersive mixing (Figure 21a.). Once the solid agglomerates have been broken up, then uniform distribution (random spatial orientation) of the dispersed additive is required. This is known as distributive mixing. The two mixing processes are not totally separated. When dispersive mixing is performed, there always is some distributive mixing, (Figure 21b.) but the converse is not always true. One could have a uniform distribution of agglomerated pigment particles. If, in a subsequent process, stresses acting on the agglomerates become too high, the agglomerates may break up, producing color streaks.





Figure 21a. Dispersive Mixing



Figure 21b. Distributive Mixing

## **Mixing Section Requirements**

A system should first provide dispersive mixing and then produce distributive mixing. These two mixing processes may be repeated more than once. Ideally, any mixing section should:

- Not cause a pressure drop and, ideally, should have a positive pumping action.
- Not possess dead spots or material hang-up regions. Flow through the section should be streamlined.
- Completely wipe the barrel surface.
- Be easy to strip, clean and re-assemble.
- Be cheap to purchase, install and maintain.

# **Dispersive Mixing Sections**

A large number of dispersive mixing sections are now available **(Figure 22a.)**. They may be classified into four main groups (1) shear or blister rings, (2) fluted mixers (such as the Egan, Maddock or Zorro mixing sections), (3) cross barrier mixers (such as the EVK and the straight cross-channel barrier mixing sections) and, (4) planetary-gear (PG) extruder mixers.

When judged by the criteria outlined above, the planetary gear extruder (PGE) mixers are probably the best all round single screw machines. The excellent heat transfer obtained, as a result of the large surface areas in contact with the melt, make them well suited for processing heat sensitive materials, such as UPVC. Good distributive and dispersive mixing are obtained. However, it should be possible to obtain slightly better dispersive mixing by the use of relatively simple, fluted mixing sections, such as the Egan, Maddock or Zorro mixing sections,



Spiral Barrier

Figure 22a. Dispersive Mixing Sections

## **Distributive Mixing Sections**

Many distributive mixing sections are now available. They may be classified into four main groups (1) slotted flight mixers, (2) pin mixers, (3) cavity mixers and (4) variable depth mixers (Figure 22b.).

If judged by the criteria outlined above, slotted flight mixers are probably the best The Dulmage mixing section (Dow) is probably the best known. It has several, shallow-angled, multi-flighted sections separated by tangential grooves. The section is formed by machining several flights over approximately 3 D in length and then making three cuts completely across (around) the flights. Because these grooves do not overlap, the barrel surface is not completely wiped by the rotating screw. A Saxton mixing section (DuPont) overcomes this problem by using helical grooves. It gives good distributive mixing with a low pressure drop as the mixing section has some forward conveying action.



Pin Mixer



Dulmage Mixer

Figure 22b. Distributive Mixing Sections

#### **Mixing Pins and Sections**

Pins, which protrude from the root of the screw into the plastic, are sometimes used to improve mixing, by breaking up laminar flow. The screw may also be fitted with mixing sections or elements. Typically, for a blow molding machine running a high molecular weight PE (HMWPE), the zero compression screw used is lengthened so that a fluted barrier section may be fitted, followed by a section which contains mixing pins. The fluted barrier section (also known as a Maddock mixing section or element) is a cylindrical shearing section, of approximately 2 to 3 D in length, which forms the screw tip. It has inlet passages from which melt can only reach the outlet passages via the land of the mixing section. Thus the melt must pass over a narrow gap before it can escape. This produces dispersive mixing (Figures 22a. & 22b.). However, at high output rates, this type of mixing section often cannot homogenize high molecular weight melts satisfactorily. So, use is also made of a mixing pin section after the Maddock mixing section. This arrangement also allows the melt to become thermally homogenized after it has been sheared. Thus it provides distributive mixing. Other arrangements are possible. For example, two mixing pin sections may sandwich a Maddock mixing section (see Dispersive Mixing Sections).



Figure 23. Static Mixer

#### Static Mixers

In addition to using the extruder screw to mix the polymer, one can use a "static" or "motionless" mixer (Figure 23.). These mixers typically consist of a number of metal "blades" or "ribbons", inserted in an adapter pipe between the end of the extruder and the die. As it flows through this section, the extrudate is continuously divided and re-combined by the blades or ribbons to thoroughly mix its components.

## Vented Machines and Mixing

It has been found that vented machines give very good mixing because of the way they are constructed (high L/D ratio and variable screw geometry) and operated.

# **Material Changes**

During the material handling, shaping and post-extrusion processes there should be little or no change in a plastic material. Any change is usually undesirable. Some of the changes that can occur are:

- Water contamination caused by the material absorbing water or by condensation. This will affect the material flow.
- Volatiles loss when the material is contaminated with volatile materials like water. During melt processing the volatile material (e.g., water) will turn to vapor and on exiting from the die will cause foaming or surface marking.
- Oxidation, that occurs when plastics are heated in contact with oxygen. The first sign of this may be a change in color that will be followed by changes in other properties.
- Degradation or decomposition will occur if plastics are overheated, even if there is no air present. Dangerous gases may be produced.
- Surface property changes such dust contamination can easily cause product rejection. It is easy to generate static electricity on plastics and this attracts dust, or dirt, very quickly.

# <sup>92</sup> Thermal Considerations

The large amounts of heat, carried from the extrusion process by thermoplastics, causes post extrusion heat removal problems. Heat removal is made difficult by the very low thermal conductivity of the thermoplastic material. This means that diffusion of heat from the center of the product to the outside and along the length of a product is slow and difficult. Thus, local temperature variations are extremely difficult to eliminate. Shear heating (induced when the molten material passes through a restricted gap or section, or the long chain polymer molecules create friction as they pass over each other) may cause some areas to become hotter than others. This may alter the flow properties of the material. Different rates of cooling will then occur in different areas of the extrudate. The stresses introduced within the product, as a result of different heat removal rates, may affect its properties.

# BREAKER PLATES, SCREEN PACKS, AND GEAR PUMPS

In order to get good quality melt from an extruder, it is important that the material be uniformly heated/melted and well mixed. Proper melting and mixing require that the correct screw be used and sufficient pressure (or back-pressure) in the barrel to obtain mixing and thermal uniformity. Screen packs, supported by a breaker plate, are often used to build up pressure within a conventional machine.

#### **Breaker Plate**

A breaker plate is a disc, or plate, that has a series of uniform holes (approximately 3 mm/0.125") machined through the thickness which are aligned in the flow direction (Figure 24.). As the holes are there to permit the plastic material to flow they should offer minimum restriction. The holes should be polished in the line of flow and the entrances, and exits, should be tapered. The breaker plate fits in the die-end of the barrel and is used to support one or more fine metal screens (the screen pack). These screens remove contamination from the melt and build up pressure within the system, thus improving mixing.

#### Screens

A screen may be a metal plate with holes of a specified size and shape or a woven metal sieve with a specified number of holes per unit area. A screen may be classified by mesh number and by open area. The mesh number is used to classify a screen. The higher the mesh-number, the finer the screen and the better the screen pack is at contamination removal. Open area is the percentage of the total area of a screen that is open. It does not change significantly going from a coarse screen (40 mesh) to a fine screen (200 mesh). For example, the open area of a 40 mesh screen may be 38% while that of a 200 mesh screen is 34%. The fine screen, however, will block more quickly.

Sintered powder and metal fiber screens have been used for gel removal in polyolefin extrusion and polymer fiber production.

#### **Screen Pack**

Combining more than one screen or gauze makes a screen pack, also called a filter pack. A screen pack may be made from an assembly of a 40 mesh, 60 mesh, 100 mesh, 60 mesh and 40-mesh gauze, that is, 40/60/100/60/40. The finer mesh gauze removes the fine contamination while the coarser mesh materials are there to provide support and to minimize fine mesh blocking.



#### Figure 24. Breaker Plate and Screen Pack

Therefore, a screen pack supported by the breaker plate, is used not only to remove contamination from the melt, but also to build up pressure within a conventional extruder.

Two pressure transducers, one on either side of the screen pack, are used to indicate when the screen pack is becoming blocked and needs to be changed. The signals from the transducers may also be used to increase the screw speed to keep the output steady as the screen pack becomes blocked.

#### **Ready Made Packs**

It is possible to buy commercial screen packs with a binding made of a soft metal. This soft metal edge helps the pack to make a good seal in the head assembly and minimizes gauze movement. One should be careful to ensure that the screen pack is used in the correct orientation, that is, with the fine gauze supported by the coarser gauze.

#### **Screen Changers**

In addition to breaker plates, which are built into the extruder barrel or adapter, the are also screen changers, which are devices that allow one to change screens without disassembling the extrusion line. There are three basic types of screen changers (Figure 25.):

- 1. Manual screen changers
- 2. Hydraulic screen changers
- 3. Continuous screen changers

The manual screen changer is the simplest of least costly of the three. It consists of a slide plate with two cavities, each designed to accept a breaker plate with screen packs. The spacing of these cavities is such that one breaker plate will be in line with the extruder flow, while the other is clear of the machine. This allows for the insertion of a clean breaker plate and screen pack combination. When it is time to make a screen change, the extruder pressure is reduced to zero and the operator moves the clean breaker

plate/screen pack assembly into the line by moving a handle attached to the side plate. The dirty breaker plate an screen pack are then removed and replaced with clean units for the next change.

The hydraulic screen changer consists of a slide-plate with two cavities to accept breaker plates with screen packs. They are spaced so that one is in-line with the extruder flow and the other is in a position clear of the machine, which allows insertion of a clean breaker plate and screen pack. The slide plate is attached to a hydraulic cylinder, which moves it back and forth to remove a clogged screen pack and insert a clean one. The shifting procedure typically takes less than one second and causes only minimal interruption of the extrudate flow. The major advantage of the hydraulic screen changer is that the screen pack can be change with the extruder running at full speed. However, it may cause an interruption in the finished extrudate due to the introduction of air from the clean breaker plate/screen changer combination.

The continuous screen changer is designed to insert clean screens without disturbing the extrudate flow. Of the various designs available the most common are the dual bolt or dual slide-plate, continuous screen, and the rotary disc. The dual bolt or dual slide-plate design uses two bolts or slide-plates that hold the screens. During a screen change, one bolt or slide-plate is removed from the line by diverting the flow through the remaining bolt or slide-plate. Continuous type screen changers use a roll of filter screen that is continuously moved across the flow of melt in the extruder to provide new filtration area. The rotary disc type uses a wheel with multiple breaker plates. This system slowly indexes a clean screen pack into the extrudate flow, while the old, clogged, screen pack is indexed out.





Hydraulic Screen Changer

Manual Screen Changer

Figure 25. Screen Changers

#### **Disadvantages of Screen Packs**

A simple way to improve mixing in a single screw extruder is to increase the density of the screen pack by using finer screens. However, the improvement in mixing may not justify the loss in output, increases in material residence time and residence time distribution, melt temperatures and the chances of stagnation and degradation. There will also be an increased risk of variability as the screen pack will suffer more chances of becoming blocked as the extrusion run continues. The use of screw mixing sections offers a more efficient way of improving mixing. The use of longer screws gives improved mixing, but perhaps, more importantly, gives more scope for the use of mixing elements.

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# The Adapter

The heated adapter holds the breaker plate assembly and directs the melt flow to the extruder die. Streamlined flow must be attained to minimize pressure losses and reduce material stagnation. If cross-head extrusion is being performed, then the adapter must turn the melt through the required angle (for example,  $90^{\circ}$ ).

The extruder head is hinged for access so that the screw and the breaker plate can be removed for cleaning when required. When closed, the head must be sealed against the barrel so that material cannot escape, even at maximum extrusion pressure (Figure 26.).



Figure 26. Breaker Plate and Screen Pack Clamp Assembly

# **Rupture Disc**

A rupture disc is a hollow, threaded plug with a thin metal diaphragm welded to the plug housing. The diaphragm is exposed to the melt on one side. If the melt pressure exceeds the strength of the diaphragm it will burst and reduce the extrusion pressure by venting material from the machine. Such a plug is generally located between the screw tip and the breaker plate.

# Gear Pumps

A gear pump (See Figure 3.) is frequently installed between the breaker plate or screen pack (or changer) and the die (Figure 27.). Its function is to build pressure and maintain precise flow of material to the die. As mentioned in Section 1 the gear pump is a very simple form of the twin-screw extruder, in which the screws are replaced by intermeshing gears that accurately pump the extrudate. The precise control of pressure and flow, provided by the gear pump, allows one to reduce the gauge variation in the extrudate and to minimize the amount of material required to produce a given product. By building the the required die pressure more efficiently, a gear pump will also reduce the load on the extruder, resulting in lower extrudate temperature and increased extruder output. 95



Figure 27. Extrusion Gear Pump System

# ► SCREW DRIVE SYSTEM

An extruder drive system consists of an electric motor, gearbox, linkage, and thrust bearings. An appropriate power supply is also required. The electric motor (usually a DC motor) is mounted in the base of the machine and is connected via a system of pulleys to a reduction gearbox. This arrangement reduces the (screw) speed and increases the torque. A flexible coupling connects the gearbox to the screw. The backwards thrust generated by the screw in pumping the material through the die is absorbed by a thrust bearing assembly. The total drive system is designed to support the screw in the barrel and to efficiently rotate it at a selected speed.

#### AC and DC

The drive motor can be either AC or DC. A common type of AC motor is the cage induction motor that is very popular because it is simple in construction, rugged and has low maintenance requirements. It is recommended for all applications (such as driving hydraulic pumps) where a single speed is acceptable. However, an extruder requires a drive system that gives step-less speed control with minimum speed variation in response to torque requirements.

The big advantage of a direct current (DC) motor is that its speed can be readily varied so that the speed can be corrected very quickly if necessary. Such a motor will also run under conditions that would stall an AC motor. However, DC power does not lend itself to efficient transmission and distribution over more than a short distance (couple of hundred yards/meters at most). For this reason electricity is nearly always generated and distributed as alternating current (AC). Electricity is transmitted over long distances at high voltages (V) to take advantage of the lower current (I) needed (as power P = VI). This means that smaller diameter wires are needed and power losses are low as  $P = I^{2}R$  (where R = resistance). AC voltage can be readily changed, by means of a transformer and may be converted by means of a rectifier into DC.

#### **DC Motor**

A direct current (DC) motor is the most popular type of motor for the modern extruder. The motor speed is regulated by solid state systems based on thyristors. Such a drive may operate on 460 V three phase AC with a fixed field of 300 V, a variable armature of 0 to 500 V, and a base speed of 1750 rpm. As the armature voltage is reduced the speed decrease linearly, while the full load torque capacity is maintained. A plot of power available versus speed produces a straight line through the origin. Thus, a motor speed of 1500 rpm gives a screw speed of 90 rpm while at a motor speed of 500 rpm the screw speed will be 30 rpm. At either speed, the full load torque is maintained.

Motors are usually found in a packaged drive (with a solid state, electronic rectifier and a speed controller) ready for connection to an AC supply. The transformer and rectifier are designed as a unit and employ solid state, power devices that can be turned off and on for voltage and current control. Starting speed, running speed and torque can be very closely regulated to give efficient operation and screw protection. This is necessary, as the weakest point in the drive system is the screw because the screw root is thinnest (weakest) in the hopper area and can be snapped by over-loading.

#### **AC Flux Vector Motor**

AC Vector drives are a relatively new method of controlling AC induction motors. The first commercial applications of AC Vector drives only became possible after high speed micro processor electronics were developed. These drives control the field orientation of the induction motor by synthesizing the motor flux current and the torque current, permitting control from full speed to nearly zero shaft speed with full torque. The high speed electronics are necessary because the control scheme requires that the motor be mathematically modeled and updated continuously.

As with all variable speed AC drives, Vector controllers vary the frequency to the induction motor to control the speed. Usually, a pulse width modulated (PWM) drive takes the incoming AC line voltage, where a diode bridge converts it to a stable DC supply. This DC voltage is then fed to the inverter section - usually an insulated gate bipolar transistor (IGBT). The IGBTs regulate the voltage and frequency to the motor and produce a sine like wave output that can vary from 2 to 400hz. The drives are called PWM because the sine wave is actually the sum of a series of pulses that a shaped and timed to "look" like a true seamless sine wave.

Two variations of Vector drives have evolved. The first uses a sensor, usually an encoder, to provide phase feedback. This type of Vector drives can provide nearly full torque down to zero shaft speed. The second type of Vector drive is sensor-less (no encoder!) and only recently became possible. This family of Vector drives employs the relatively new high speed digital signal processors (DSP) electronics to permit even faster processing of the complex motor simulation algorithms required for sensor-less control of induction motors. The DSP's also permit for "on the fly" adjustments to the motor simulations to compensate for changes to the induction motor due to temperature and age. This type of Vector drive requires a minimum signal to maintain control of the motor, thus control down to zero shaft speed is not possible. Control down to very slow shaft speeds, even 2 to 4 Hz, are still attainable though, and because most applications do not require control down to zero shaft speed, the sensor-less vector drives are gaining in popularity.

The control accuracy of the vector drives is approaching and in some application surpassing that of DC motor and drives. This fact coupled with the efficiency advantages of AC motors has led to the increased use of AC Vector drives.

#### Gearbox

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If the motor speed is 1500 rpm and the required screw speed is 90 rpm then a reduction gearbox with a ratio of 16.67:1 is required. Often the exact gearbox ratio required is not available. Then different sized pulleys, connected by a system of belts, are used to make the final adjustments. For example, the speed of the motor may be reduced, at the input to the gearbox, by using a larger pulley on the gearbox shaft and a smaller pulley on the drive shaft. A belt drive provides some safety against sudden overloads, but power losses can be as high as 10%. All belts and pulleys must be guarded. A change of pulleys can be useful if the extruder is used for another application whose power requirements are different. Pulleys whose size can be altered during running (by using expanding pulleys) can provide a non-electrical method for changing screw speed.

Because extruder motors deliver approximately constant torque over their entire speed range, the power available increases linearly with speed. For maximum power therefore, the motor should be run at its top rated speed and the power transmission capability of the gearbox must be matched to the maximum motor power. If herringbone style gears are used (usually in a two stage process) the efficiency of the gearbox is high (96 to 98%). The gearbox is connected to the screw through a flexible coupling. The flexible coupling accommodates any slight errors in alignment that exist between the output shaft of the gearbox and the thrust assembly and helps to isolate thrust loads (generated by the screw) from the gearbox. Regular maintenance of the gearbox is essential.

#### **Screw Support Bearings**

The shank of the screw is fitted into a driving sleeve in the bearing housing. Normally a single slotted key is used, but a splined fitting is better at distributing the torque around the shaft. The bearing must support the screw horizontally, so that it does not contact the barrel wall. It must transmit the motor torque to the screw and withstand the considerable thrust loads generated by the screw. For example, a 4 inch (100 mm) diameter extruder will have a back thrust of 28 tons (30.8 tonnes) if the pressure at the end of the screw is 4000 psi (276 bar). Although ball bearings allow high rotation speeds, their load bearing capacity is not as high as roller bearings and so these are employed to withstand the high thrust loads. Programmed lubrication of the screw support bearings is important.

Bearings do wear out and damaged bearings will affect the performance of the extruder by producing increased motor current and reduced output (because of increased wear of the screw and barrel). Bearings are sold with a predicted life under prescribed loading conditions.

#### Screw Surface Speed and RPM

In extrusion screw speed means screw rotational speed which is usually measured in revolutions per minute (RPM or sec<sup>-1</sup>). However, it is the screw surface speed that determines the amount of shear heat generated. The faster the screw is rotated, the higher is the temperature of the melt (due to the amount of shear being increased). This means that to obtain the same amount of shear, the screw on a larger machine should be rotated more slowly than the screw on a smaller machine. This will keep the shear rate down and prevent localized overheating. The speed of rotation will also differ from material to material. UPVC is limited to a maximum, screw surface speed of 0.25 ms<sup>-1</sup> (15 m/min or, 50 ft/min), whereas with other more shear resistant materials, higher speeds can be employed. To convert from rpm to ms<sup>-1</sup>, multiply the rpm by the screw diameter in mm and then by 0.000.052.4.

Screw Surface Speed =  $ms^{-1} = 0.0000524$  [RPM x D<sub>s</sub>(mm)] (Eqn. 19)

# ► MOTOR SIZE AND THRUST BEARING LIFE

The drive system is designed to support the screw in the barrel and to rotate it at a selected speed. Once the speed is set it must remain constant, despite changes in the load on the screw or in the temperature of the drive system. It is very important that the screw speed selected is held constant during operation as it controls the amount of material fed to the die, the shear history of that material, and the melt temperature. The torque supplied must overcome the resistance of the screw to turning, even at low speeds, such as those seen during start-up. It is the torque and speed desired which determine the power requirements of the system.

#### **Torque and Horsepower**

Torque is a measure of rotary force or the turning effort of a motor. It is a product of the length of a lever arm and the force applied to the end of the lever arm that is measured in Newton meters (Nm).

In North America, motors are rated in horsepower whereas in Europe, motors are rated in kilowatts (kW). Horsepower (HP or hp) is a unit for measuring the rate of work which is equivalent to 550 ft lbs/s or 0.746 kW.

#### HP = 6.28 x rpm x torque/33,000 = rpm x torque/5255. (Eqn. 20)

This means that in order to obtain the same HP from a slow speed motor, more torque must be developed. As torque is what produces stress on the mechanical parts of a motor, a slow speed requires a motor that is larger than a high-speed motor. A 1 hp motor running at 1750 rpm is approximately twice the size of a 1 hp motor that runs at 3500 rpm.

#### **Motor Size**

The size of the motor required to drive a screw can be estimated from the experience based "rule of thumb" that approximately 10 lbs/hr of polymer can be extruded per horsepower (HP) on a single screw machine. Some typical power requirements for various sizes of single screw extruders (24/1 L/D ratio) are given below. This assumes a smooth barrel, no vents and an L/D ratio of 24/1. If a grooved barrel or a vent is used, then extra power will be required.

Diameter		Power		
(in.)	(mm)	(kW)	(HP)	
1.5	38	12	16	
2.5	50	40	50	
3.5	90	85	115	
4.5	100	130	175	
6.0	150	230	310	
8.0	200	400	530	

The motor must be sized to match the workload and this depends not only upon the diameter of the extruder, but also on the application. For example, with a 6" (150 mm) extruder, the motor power can vary from 160 kW to 280 kW according to what is being processed, as shown below.

100	Application		Polymer (lb/hr)	Output (kg/hr)	Power (kW)	/er ) (HP)
	Sheet	PMMA	1260	573	160	215
	Pipe coating	HDPE	1150	523	230	310
	Blown film	LDPE	2000	909	280	375
	Multi-filaments	PP	1750	795	280	375

#### Life of a Thrust Bearing

The B10 life of a thrust bearing is the length of time in hours when 10 out of 100 bearings are expected to fail if run continuously under the specified conditions. Many manufacturers quote B10 life assuming that the extruder will run at 100 rpm with a 5000 psi (345 bar) head pressure. If the extruder is run faster or at a higher head pressure, there is a reduction in the expected bearing life. The following tables indicate the B10 multiplying factor for a range of conditions other than the standard conditions. For example, if a ball thrust bearing has a predicted life of 100,000 hours at 100 rpm, then its expected life at a screw speed of 125 rpm and head pressure of 7,000 psi (483 bar) is only 30,000 hours. (0.3 x 100,000). (Note: 100,000 hours is about 12 years of 7 day per week 24 hour per day operation.)

Multiplying Factor for Thrust Ball Bearings					
Head	ad Pressure Screw Speed (RP			beed (RPN	1)
psi	bar	75	100	125	150
3000	207	6.45	4.84	3.87	3.23
4000	276	2.72	2.04	1.63	1.36
5000	345	1.33	1.00	0.80	0.67
6000	414	0.80	0.60	0.48	0.40
7000	483	0.51	0.38	0.30	0.25
8000	552	0.35	0.26	0.21	0.17

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# SECTION 5: THE DIE AND POST EXTRUSION EQUIPMENT

- > DIE DESIGN CRITERIA
- MATERIALS OF DIE CONSTRUCTION
- ► DIE HEATING
- FLAT FILM AND SHEET DIES
- ► BLOWN FILM DIES
- ► PIPE DIES
- > WIRE AND CABLE COVERING DIES
- ► PROFILE DIES
- ➤ COOLING
- ► EXTRUDATE TAKE UP
- CUTTING AND STACKING

# > DIE DESIGN CRITERIA

The die is the assembly, located at the end of an extruder, which contains an orifice used to shape a plastics melt (**Figure 1.**). Basically the die is a block of metal containing an internal flow channel that can be divided into three parts: 1. The adapter section that connects with the output channel of the extruder

- 2. The distribution section that spreads the melt into the correct shape
- 3 The land, which maintains a constant cross section to the flow channel until it reaches the die exit lips

# General Design Criteria

When designing or evaluating dies there are certain general criteria that can be applied. The prime requirement is to produce the correct extrudate shape or cross-section. This often involves producing an extrudate of constant thickness (film, sheet, pipe, cable coating etc.). To do this the die must:

- Provide a uniform flow rate of material through its exit gap
- Maintain a uniform melt temperature over the whole of the exit gap
- Produce melt that has the same shear history for all parts of the exit gap

Failure to meet any one of these criteria may result in poor thickness distribution in the extrudate. Regions of the extrudate that are hotter than the bulk will deform more readily and thus become thinner than average. The viscoelastic nature of molten plastic means that it can "remember" how much it has been deformed. This "memory" is short term (generally, a few seconds at processing temperatures), but the shear history within this time frame may alter the stiffness of the melt and its die swell behavior.

## **Polymer Degradation**

Degradation within the die must be avoided. This requires smooth flow channels without step changes in size. Steps in the flow channel provide places where the melt can hold up for long periods and degrade. Specks of black **102** degraded polymer are then carried into the melt stream and hence to the extrudate. Any leakage into the die that provides a source for air to enter will also cause degradation of the melt.

The residence time of the melt in the die also will determine how much degradation will occur. The average time in the die is the ratio of the weight of molten polymer held in the die to the output rate. For example, a 15 inch (380 mm) pipe die holds about 200 lb (91 kg) of molten unplasticized polyvinyl chloride (UPVC). If the output rate is 1100 lb/hr (18.3 lb/minute or 8.3 kg/min) then the average residence time in the die is 200/18.3 = 10.9 minutes (91/8.3 = 10.9 min). The weight of material in the die can be determined by experiment or by calculation. In the latter case the volume of the die multiplied by the density of the molten polymer (in the case of PVC, its molten density is about 1020 kg/m<sup>3</sup> (1.02 g/cm<sup>3</sup>) as compared to a solid density of 1400 kg/m<sup>3</sup> (1.4 g/cm<sup>3</sup>) at room temperature). Minimizing the size of the internal die channels thus reduces the possibility of degradation.

(Note that the above calculation only gives average residence time in the die. Material in the center of the flow channel travels through the die faster than material next to the walls of the die; this material takes much longer to pass through).

#### **Material Changes**

Polymer grade changes are frequently made during production and the time for the changeover depends on how rapidly the composition of the extrudate changes to the new material. The die design affects this rate of change. Dies with short residence times, and no areas where polymer can 'hangup', are preferred. Because of the nature of the flow pattern in the die channels, the new material will appear first at the center of the extrudate and the old material will remain the longest in the outer skin layer.

#### **Surface Finish**

The surface finish of the extrudate is affected by the die design. For the best surface finish, the land should be reasonably long and the lead in angle to the land should be low (Note: If the lead in angle is too low, the length of the die is increased. This is usually not an advantage). Blocking, or partial blocking, of flow channels by contamination or degraded melt, which leads to the formation of die lines, can occur if the flow channels are too narrow. For this reason the minimum dimension of a channel should usually not be less than 0.03 inch (0.762 mm).

#### **Back Pressure**

Back-pressure, from the resistance to flow through the die, is an important consideration. If the pressure needed to force material through the die, at the correct output rate, is too high, it will:

- Tend to stretch and distort die bolts
- Cause leakage from joints
- Increase the power requirements of the extruder possibly causing it to stall
- Increase the working of and amount of shear heat generated in the resin
- Increase wear of the screw and extruder

On the other hand too low a pressure can give:

- Poor thickness distribution
- Weld line effects in the extrudate
- Poor mixing of the material in the extruder
Calculations of the estimated pressure drops in a die are very useful in design and evaluation procedures. A simple method to do this is given in the section **Estimation of Pressure Drops in Extrusion Dies**.

#### **Die Maintenance**

Die maintenance is another important consideration. The ideal die is low in size and weight, made from very few components, easily lifted, disassembled and easily reassembled. Simplicity is a virtue here as well as use of designs that allow the die to be rebuilt in one way only. The use of both conventional and wire electro-discharge techniques enable complex extruder dies to be machined in one or two component assemblies. This prevents problems of leakage during production and damage during disassembly and assembly.

## ► MATERIALS OF DIE CONSTRUCTION

The properties required of a die material are as follows:

- High modulus to prevent the die channels from deforming under pressure or under its own weight
- Toughness and robustness to withstand constant use
- Wear and corrosion resistance to give a long production life.
- Good machinability characteristics so that a high surface finish may be maintained throughout the working life of the die (this is especially important near the die exit. For this reason the die land, or sections of the die lands are made interchangeable).
- High thermal conductivity and low density to provide a uniformity of temperature

#### Low Carbon Steels (Mild Steel)

Untreated low carbon steels are easily machined, but are far too soft for extrusion dies due to their inability to be through hardened. In their natural soft state the wear characteristics are poor and the die will not last. Processors who need to manufacture simply shaped extrudates at low output rates with relatively low production requirements often use mild steel.

A considerable number of prototype dies are made from low carbon steels. To improve its wear resistance and dimensional stability the surface of the steel is frequently ion implanted or titanium nitrided.

#### **Pre-toughened Steels**

The main advantage of quenched and tempered, or pre-toughened, steels, is that they can be used to manufacture dies that include intricate shapes. Its inherent hardness is sufficient (42 Rockwell C) to resist deformation during use and, therefore, no subsequent heat treatment is needed. Elimination of this second heat treatment means the die does not need to be re-machined to remove deformation incurred from the first heat treatment process. Therefore, savings in die manufacturing times and costs are realized.

If the internal surface of the die requires a higher hardness, then surface treatments such as titanium nitriding, ion implantation, flame hardening and chromium plating are used. Since many dies are manufactured by electro-discharge machining (EDM), care should be taken in selecting the correct type of pre-toughened steel. Sulfur containing types are not recommended when using EDM. The designation given to the pre-toughened steel is AISI P20, or DIN 40 CrMnMo<sub>7</sub>.

## <sup>104</sup> Brass

This material, though poor in wear, is easily machined and does not require heat treatment. It is, therefore, sometimes used to make prototype dies to evaluate their performance, before going to the expense of building the final die. Changes to the flow channels are relatively easy to make and the die will last long enough for trials to be carried out. However, certain polymer melts are attacked by the constituents of brass which initiate degradation of the polymer in the molten state. This causes a reduction in the physical properties of the resultant extrudate. The polymers most reactive with brass are PP, PE and ABS.

## **Case Hardening Steels**

These steels, also called low carbon or mild steels, (with a carbon content of less than 0.2%) are heat treated after the die shapes have been manufactured. The die parts are heated to a temperature of  $1,470^{\circ}$ F to  $1,650^{\circ}$ F ( $800^{\circ}$ C to  $900^{\circ}$ C) and then quenched. High hardening temperatures and fast quenching give harder steel surfaces, but with a greater dimensional change after hardening.

The surface structure of these case hardened steels will differ from the structure in their interior due to different thermal histories. The softer interior is tougher than the hard skin. The hardened skin, depending upon the grade of steel, can reach as high as 62 Rockwell C. For extruder dies, an oil quench bath at about 390°F (199°C) gives the most suitable combination of hardness and minimal dimensional change. Carburizing of the steel to increase the carbon content of the surface will produce an increase in surface hardness. The thickness of the hardened skin ranges from 0.008 to 0.0394 inch (0.2 to 1.00 mm).

The major drawback to case hardened steel is the change in dimensions upon heat treatment. This is especially problematic for wide sheet or complex profile dies. Modification of the die sometimes requires a further re-hardening operation since the machining operation may remove the hardened skin. Hence the surface hardening and quenching processes must be carried out correctly to give the proper properties to the steel and the required die dimensions.

## **Nitrided Steels**

These materials contain additives such as chromium or molybdenum that form hard nitrides when heated to 1100°F (600°C) in an atmosphere of ammonia. Quench cooling is unnecessary and distortion of the die is less likely. Excellent surface finishes can be achieved when using these types of steels.

## **Corrosion Resistant Steels**

For dies used to process polyvinyl chloride (PVC), steels with a content of up to 16% chromium will give excellent corrosion resistance. This type of steel is able to hold temperature during processing (it has a low thermal conductivity), is readily machined to complex shapes and heat treated to relatively high hardness values (54 Rockwell C) while possessing a very high surface finish and minimal dimensional changes.

This material is inherently corrosion resistant and hence the production life of a die is often increased three fold when compared to other types of steels. The two corrosion resistant steels that are most commonly used are classified as AISI 414 and AISI 420. AISI 420 steel can contain either 13% or 16% chromium. The higher the amount of chromium the more corrosion resistant the steel becomes. Both types of steel are supplied in a pretoughened condition (similar to the hardness of the P20 type steel). However, the lower the percentage of chromium the higher the hardness value that can be achieved. For PVC dies, the 16% chromium grade is preferred, whereas for general use the 13% grade tends to be selected.

#### Anodized Aluminum

Anodized aluminum alloy is now used for extrusion dies due to its ability to resist wear, its overall dimensional stability and the fact that it can be polished to a mirror finish. Special grades of anodized aluminum, that need to be machined by the EDM process, have been produced for extruder dies. Such grades now provide the die maker with the opportunity to obtain a high gloss surface finish of the internal flow channels of the die.

#### **Chromium Plating**

During the plating of a die, a layer of chromium about of 100 microns in thickness is deposited electrolytically. The electrodes must be shaped for the die to give a uniformly thick layer. The chromium protects against corrosive attack from PVC decomposition products. Re-plating is necessary in time, due to wear and chipping of the chrome laminate; particularly at the entrance edges of the flow channels/die lands. Chrome plating, although still used, has been largely replaced by a process called titanium nitriding (TiN). This is due to the non-stick properties associated with TiN and the use of corrosion resistant steels.

## ► DIE HEATING

The temperature of the die should not be used to control the melt temperature of the polymer, but should present the melt with a channel whose walls are at the same temperature as the molten material (melt). Electrical heating elements, with thermocouples as sensors and PID (three-term) controllers (with cascade feedback control) are used. In some cases, where a glossy finish on the extrudate is required, the die temperature may, in fact, be hotter than the extrudate itself.

## Zoning

The die should be divided into as many zones as necessary to maintain the desired temperatures across the die, each having a temperature feed back device and control function. This allows the overall temperature profile of the die to be set globally or individually. Control of the temperature can then be programmed to be global, in zones, or as individual portions (areas) of the die. This provides better temperature control especially during heating of the cold die and during production, particularly when a heating element fails.

## **Cartridge Heaters**

These tubular heaters consist of a nickel chrome resistance element wound onto a magnesium oxide former and contained in a brass or stainless steel outer sheath. The heating element is electrically insulated from the sheath by magnesium oxide powder or paste. Positioning of the heating elements inside of the die gives them protection and places the heat source close to the channel. However, care must be taken not to place the heaters too close to the channel. Apart from mechanical considerations, there is a non-uniform tem**106** perature distribution in the immediate vicinity of each element. The temperature, however, becomes more uniform as the distance from the cartridge heater increases.

When fitting the heaters into the die, there should be a close fit (maximum gap 0.012 inch (0.3 mm) between the cartridge sheath and the die material. Poor contact gives rise to cold spots next to the gap and hot spots on the other part of the heater, leading to reduced life. To prevent premature failure, the cartridge heater is often covered with a polytetraflouroethylene (PTFE) paste so that all the inherent air gaps are filled with the paste. Tapered outer sheaths are preferred nowadays due to the ease with which a failed cartridge heater can be extracted from the die body.

#### **Band and Plate Heaters**

These heaters are clamped onto the outside of the die body and provide a cheaper heating system than internal heaters. Because of their position they are prone to damage and less efficient in the use of power than internal heaters. They should be used with deeply imbedded thermocouples and PID type controllers. When fitting, care must be taken to get good thermal contact between the heater and the surface of the die over the whole area of the heater. Any contamination will lead to a hot spot on the heater and a reduction in heater life. If a number of band heaters are used their clamping bolts should be staggered in position as a neat line will lead to a cold band on the die.

## **Cast Heaters**

Aluminum alloy cast heaters with internal sheathed heating elements can be manufactured to give a close fit to the outside of the die. Though more expensive than band heaters, their life expectancy is considerably greater, they give better temperature uniformity and the heat energy density or watt density, can be higher (up to 50 watts per square inch, or 7.73 watts per square cm).

## **Sizing of Heating Elements**

The selection of the correct power rating for heating elements is very important to ensure that the die heats up to temperature in a reasonable time and that different parts of the die and extrusion system heat up to temperature uniformly. This reduces the risks involved in heating a system when it contains solidified polymer.

One method is to calculate the actual power requirements for each individual extrusion die. The weight of metal to be heated by each separately controlled heating zone, is first determined and the target time to reach temperature is selected. The heater power is calculated from knowledge of the specific heat of the steel and the temperature rise required. For example, suppose a die part to be heated weighs 350 Lbs. (159 kilos) and is required to raise its temperature from 70°F to 400°F (20°C to 204°C) in 30 minutes. To calculate the heater power, multiply the weight by the temperature rise by the specific heat of steel and divide by the time in seconds. The specific heat of steel is 119 Joules (J)/lb/°F (470 J/kg.K).

#### Heater Power = [Wt. x C<sub>sp</sub>]/ t (sec) = (350 x 330 x 119)/30 x 60 = 7,640 watts (Eqn. 21.)

This calculation ignores any heat losses from the surface of the die. To allow for such losses, a 10 kW heater would be recommended.

# FLAT FILM AND SHEET DIES

The main characteristic of these dies is the presence of a manifold that distributes the melt across the width of the die (Figure 28.). Melt flows out from the manifold through a slit channel. These dies are normally made in two halves. In symmetrical dies the flow channel is cut equally from each half of the die. Asymmetric dies have one half with a flat plane surface and the flow channel cut in the other half. Asymmetric dies are cheaper, but exhibit an inferior flow distribution across their surfaces.





Figure 28. Flat Film and Sheet Dies ("T", Coathanger)

#### **'T' and Fishtail Designs**

The simplest manifold is in the 'T' die. The melt enters the center of a cylindrical channel, of uniform cross section, which goes from one side of the die to the other. This is the manifold. Its axis is at right angles to the machine direction. The material escapes from the manifold through a slit running the length of the manifold. Because the size of the manifold cross-section is much greater than the slit, the pressure drop along the manifold is very small and there is nearly uniform output from the manifold across the entire width of the die. 107

There are problems with the 'T' manifold design resulting from 'hang-up' of polymer at the ends of the manifold and a small but finite pressure drop across the manifold. Fishtail designs are better in that the manifold is angled and opens gradually like a fish tail. Though suffering less from polymer 'hang-up', fish-tail designs do not give a perfect flow distribution over the die width for a system where the polymer or the production conditions are changing **(Figure 28.)**.

#### **Coat-Hanger Dies**

The most satisfactory design is the coat-hanger manifold. It is based on the 'T' manifold, but it slopes towards the die exit at the edges of the die, like the shape of a coat-hanger. Usually the slit downstream of the coat-hanger manifold has a constant channel thickness. In a correctly designed coat-hanger die, the flow rate per unit width is constant from the exit of the slit. This means that the flow rate in the manifold should decrease linearly from the center to the edge of the die. The pressure drop between the entrance to the manifold and the end of the slit should be equal for all flow paths.

Ideally the shear rates in the manifold should be the same along the whole width of the manifold. Dies designed to fulfill these requirements have a manifold whose radius decreases as the 1/3 power of the distance from the center and a slit whose length in the machine direction varies as the 2/3 power of the distance from the center.

#### **Die Adjustment**

Following the manifold, the melt passes under a choker bar. The height of the flow channel under the choker bar can be adjusted by moving the choker bar normal to the flow channel. Tension and compression bolts are provided for this purpose at about 1 inch (25.4 mm) intervals. This gives some adjustment to the overall die resistance and can correct differences between one side of the die and the other. These bolts may be adjusted automatically by means of built in heaters and associated controllers on some machines.

The use of heat pipes help to longitudinally distribute the excess heat in the center of the die to the outer edges. Monitoring of this temperature dissipation and using the thermal die resistance control technique can suitably optimize the corresponding output rates across the die length.

In the common "Flex-Lip" flat die design, one side of the exit lips is adjustable. There are a number of closely spaced adjusting bolts that can be used to alter the die gap at the exit. Since each part of the extrudate width can be separately adjusted with a bolt, the system allows for a more even exit pressure across the die, and as a result, the production of close tolerance sheeting.

The width of an extrudate can be reduced, by up to a third, by blocking the edges of the die exit gap with deckels. However, the use of these deckels creates a dead-space on each side of the die where material will stagnate and eventually degrade. Thus deckeling can only be used with very heat stable polymers at low temperatures. They will also alter the flow balance for the die and so thickness tolerances will be more difficult to maintain.

## ► BLOWN FILM DIES

There are three basic die designs used in the manufacture of blown film. These are the sidefed, bottomfed and spiral mandrel types. All three designs aim to produce a uniform melt around the circumference of the die exit lips. A

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uniform melt temperature, shear history, and flow rate are required to achieve this. A typical blown film die is shown in **Figure 29**.



Figure 29. Blown Film Die

#### **Outer Ring**

All three basic die designs can be fitted with an adjustable outer ring that forms the outer boundary of the die exit gap. It can be moved relative to the inner mandrel to correct for variations in the thickness distribution around the circumference of the film (that is, in the transverse direction). However, movement of the outer ring alters the die gap on both sides of the die. So, if the thickness of one side of the die is increased, that on the opposite side will be reduced. The outer ring must be bolted to the body of the die, at the correct torque, to allow movement when the gap is adjusted, while preventing leakage.

#### Side-Fed Film Dies

The oldest design is the side-fed type. Melt is directed at the side of the mandrel and flows around it before joining on the side opposite the feed. Because of this, there is an inherent difference between the two sides of the die. Careful design of the thickness of the flow channel and the provision of flow restrictors on the feed side of the die will produce a reasonably uniform flow. However, any change in operating conditions, the plastic, or the melt viscosity of the material being used will invalidate the design balance. Hence, the film produced will become non-uniform.

# <sup>110</sup> Bottom-Fed Film Dies

A more axially symmetric design is found in the bottom-fed type of die in which the melt is directed at the center of the base of the mandrel. Streamlined spider arms are used to support the mandrel in the die body. The flow of the melt around the spider arms will produce a disturbance in the flow path at each spider arm producing potential flaw in the film. One advantage of the bottom-fed design is that the flow channels in the die have a low total volume. This leads to short residence times and the design is used extensively for manufacturing soft PVC films and other easily degraded polymers.

## Spiral Mandrel Film Dies

The most common blown film die design is the spiral mandrel type. The melt stream is divided into four or more equal flows, each of which passes through its own port at the base of the mandrel. Each port connects with a flow channel cut into the mandrel that takes the melt in a spiral path up the outer surface of the mandrel. The depth of the spiral channel decreases as it spirals up the mandrel, while, at the same time, the gap between the mandrel and the outer die body increases. This design geometry aims to allow plastic melt from a port to flow increasingly upwards in the gap between the mandrel and outer die body as the melt moves away from the port. In a correctly designed die, the rate of flow in the spiral channel decreases uniformly to give an even transfer of flow to the upward direction. Melt from each port is spread evenly over about three-quarters of the die circumference. Since there are a number of ports, typically six or eight in the best dies, the combined flows give a very even output around the circumference of the die exit gap.

## Blown Film Die Land Design

The are a number of factors to be considered in the design of the land section of a blown film die.

- Sufficient back-pressure must be generated to ensure good performance of the die distribution section
- The film must be drawn down to its final thickness after it has left the die
- The wider the die land the greater the necessary draw-down
- The die gap must be large enough to prevent blockage by any contaminants in the melt
- It is necessary to operate in the land area at a melt shear stress below that at which melt fracture will occur.

Widening the land gap will reduce the shear stress in the land. Increasing melt temperature or reducing output has the same effect. The critical stress above which melt fracture occurs depends on the plastic material.

For LDPE a die gap between 0.03 and 0.04 inch (0.762 to 1.016 mm) has proved successful even at output rates up to 20 lb./hour/inch (3.6 kg/hour/cm) of circumference. The length of the land is usually 10 to 15 times its gap width. With this design, the draw down in the machine direction is about 12 for a 100 gauge (40 micron) film at a blow up ratio of 2.

## LLDPE Land Design

The melt rheology of LLDPE leads to a different blown film die land design. Because LLDPE is more Newtonian than LDPE the shear stresses found in the die at high shear rates are higher than those seen for LDPE. It has been found that above a critical land shear stress of 15 psi. (1 bar), melt fracture will occur and give rise to a rough appearance in the film. The source of this melt fracture is thought to originate from slip at the walls of the land. There are at least three ways of reducing the melt fracture tendency and hence increasing possible output rates for LLDPE films.

The die gap is increased to reduce the shear stress. The minimum gap required for a range of melt temperatures and outputs is shown in the following table:

Output Rate	200°C/392°F		220°C/428°F		250°C/482°F	
-	(in.)	(mm)	(in.)	(mm)	(in.)	(mm)
5.6 lb/hr/in.	0.104	2.64	0.086	2.18	0.089	2.26
11.2 lb/hr/in.	0.152	3.86	0.122	3.10	0.099	2.51
22.4 lb/hr/in.	0.213	5.14	0.170	4.32	0.134	3.40

If the die design is such that high pressure drops over the land are needed to ensure the correct functioning of the distribution section, then the land is restricted in width upstream of the exit and opened out to the above figures immediately before the exit.

The material of the walls of the die land is chosen to minimize the slip that produces melt fracture. It has been found that alphabrass once 'run in' allows higher output without melt fracture. The running in period is to allow wear to modify the surface of the land.

Additives, such as fluor-elatomers, can be incorporated into the polymer. These 'anti-melt fracture' materials are deposited on the die surface and reduce melt fracture. Unless they are replenished their effectiveness diminishes. Grades of LLDPE are available with these types of additive included. Alternatively, master-batches with a high concentration of the additives may be blended with the virgin resin in the extruder hopper or used from time to time, in concentrated form, to replenish any coating that has worn away.

#### PIPE DIES

Standard pipe dies are made for pipes with diameters from 0.5 to 30 in. (12.7 to 762 mm). Most of these dies feed the melt to the base of a mandrel from which it spreads out, around the mandrel **(Figure 30.)**. The mandrel is supported in position by up to 24 spider arms. These supports are streamlined to give the least disturbance to flow. Nevertheless, the melt must rejoin after the spiders. To assist fusion and prevent weld lines in the pipe, the channel cross



112 section is reduced in the next section. There is a reduction in both diameter and channel width. In the final land section of the die (in all except the largest dies) there is an outer ring, which is adjustable relative to the mandrel, to correct for variations in pipe wall thickness. The length of the land varies from 10 to 30 times the pipe wall thickness. Dies for polyolefins tend to be longer between the spider section and the land. They are often designed with zones to compress and decompress the melt before it enters the land.

## Sizing of Spider Arms

The spider arms must be strong enough to withstand the shearing forces exerted by the high pressures upstream of the mandrel. For example, if the die mandrel has a diameter of 12 inches (304.8 mm) at the die exit and the pressure at the entrance to the die is 4000 psi (276 bar):

The total load on the mandrel =  $4000 \times \pi \times 6^2$  in. = 452,448 Lbs. (276 x  $\pi \times 15.24^2$  = 201,412 kg).

If there are eight spider arms, the shear force experienced by each arm is 56,500 lbs. (25,496 kg). Assuming that the maximum shear stress that should be applied to the steel spiders is 60,000 psi (4,138 bar), then the cross section of each spider must be about 1  $in^2$  (6.45 cm<sup>2</sup>). Increasing the number of spider arms obviously allows for the reduction of the size of each individual arm.

#### **Alternatives to Spider Arms**

A number of alternatives to spider arms have been tried and used successfully in dies for polyolefins. They include a screen pack die in which the mandrel is supported by a metal screen containing many small holes through which the polymer melt passes. The holes are about 0.02 to 0.04 inches (0.5 to 1.0 mm) in diameter and spaced 0.05 to 0.07 inches (1.25 to 1.78 mm) apart. The screen is cylindrical in shape and fits onto the back of the mandrel. The dies are more compact than spider arm designs as there is no need for a large compression after the spider section. They have been used mainly for large diameter polyolefin pipes. Spiral mandrels, similar to those used in blown film dies, have also been successful.

#### **Die Size Range**

By using adapters and replaceable components, the rear section of a die, which includes the spider support, can be used to produce a range of pipes of differing diameter and wall thickness. This approach is obviously cheaper than buying a complete new die for each size. In the case of polyolefin production, the following table shows how four basic dies can cover a range of pipes of 0.4 in. to 28 in. (10 to 700 mm) in outside diameter.

Die	Pipe Diameter		Wall Th	Max. Output		
	(in.)	(mm)	(in.)	(mm)	(lb/hr)	(kg/hr)
1	0.4-2.5	10-63.5	0.08-0.15	2-3.8	450	203
2	0.8-6.3	20-160	0.08-0.25	2-6.4	1,000	451
3	3.5-16	89-406	0.10-1.1	2.5-27.9	1,300	587
4	9.0-28	229-711	0.35-1.4	8.9-35.6	2,000	903

## **Multiple Outlets**

Dies can be purchased with more than one exit, so two or more pipes can be manufactured simultaneously. For PVC dies with two outlets are used, while

for polyolefins triple heads are available. These dies make more use of the capacity of an extruder in situations where small diameter pipe has to be produced on an extrusion line generally used for large diameters.

#### **Automatic Centering**

To control the pipe dimensions automatically, the pipe wall thickness must be continuously monitored. This is usually done by means of an ultrasonic measurement in, or just after, the cooling bath. A water layer between the pipe and the ultrasound probe is required for accurate measurements. Changing the haul-off speed can reduce departures from the targeted average pipe wall thickness. Departures from wall thickness uniformity, around the circumference of the pipe, require adjustments to the die. Two methods that have been used are a thermal method and one based on motorized rings. In the thermal design, the heating of the land section of the die is split into 8 to 12 zones around its periphery. These zones are thermally separated by air gaps. The temperature of each zone is separately controlled in the range from 355°F to 390°F (180°C to 199°C) to adjust the pipe thickness distribution. In the alternative method, the pipe ring is positioned relative to the mandrel by means of slightly eccentric rings. Rotation of these rings shifts and alters the die gap distribution. By driving the rings through a gearing system, any departures from wall thickness uniformity can be reduced by automatic adjustment of the die gap.

#### > WIRE AND CABLE COVERING DIES

Wire and cable covering dies are crosshead type coating dies, in which the melt enters from the side. The melt is then wrapped around a mandrel through the center of which the conductor is fed (Figure 31.).



Figure 31. Multi-Layer Wire and Cable Die

# <sup>114</sup> Pressure and Tubing Dies

In a pressure coating die the melt and conductor are brought together inside the die. The conductor travels through the die land with the melt surrounding it. To prevent back leakage, the gap between conductor and mandrel is no greater than 0.002 inch. (0.05 mm). With line speeds up to 4,000 ft/min (1,219 m/min), wear of the mandrel is likely and special hardened inserts are used where the mandrel contacts the conductor.

For larger conductors and cable coating, it is not possible to prevent back leakage of melt if it joins the conductor under pressure. Therefore, these act as tubing type dies in which the melt emerges from the die as a concentric tube around the conductor, as it emerges from the center of the mandrel. Vacuum is applied to the rear of the die and as the extrudate is pulled away from the die, the coating is drawn onto the conductor.

Land lengths in coating dies are usually 0.2 to 2 coating diameters in the case of plasticized PVC and 2 to 5 diameters in the case of polyethylene (PE).

#### **Distribution System in Coating Dies**

These side-fed dies usually have a heart-shaped compensating plate in the side of the die where the melt enters that prevents the melt from going straight towards the die exit. The melt has to flow around the plate which converts a single feed into twin feeds from opposite sides of the die. The design of these plates has been largely by trial and error. Computer modeling of the flow passages around the mandrel is now common practice. The depth of the flow channel, throughput of material and the resultant shear rate and shear stress values are readily determined for a particular polymer and coating thickness. Such computer aided engineering (CAE) software packages have now replaced experience and intuition in the design of coating dies.

In small dies, the melt can be split into two flow channels. The melt then flows around the die (through 90°) to converge from opposite sides; each stream onto its own heart shaped compensating plate. Another system uses a manifold similar to that found in flat sheet dies. In the wire coating case the manifold is wrapped round the mandrel.

#### **Pipe and Hose Coating**

Circular cross-section substrates, such as steel piping or fabric fire hoses, can be coated with plastic melt using dies whose design principles are similar to those discussed above. The distributor can be a side-fed, heart-shaped compensator. Spiral mandrel designs have also been used for larger diameters.

## ► PROFILE DIES

This is the name given to the dies used for the production of extrudates with complex and often non-symmetrical crosssections. Because of the lack of symmetry, obtaining the correct crosssection can be difficult.

## **Differential Flow Resistance**

Variations in flow resistance in a die cross-section can result in different flow velocities in different parts of the extrudate. This will cause the extrudate to bend as it leaves the die. The flow velocity can be equalized over the profile by the use of a variable land length over the profile or by the use of restricting plates in the flow channel, where there is a tendency for too rapid a flow.

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Differential die swell can also distort the extrudate. Die swell is directly related to the shear stress in the land. As the shear stress increases, so does the die swell. These complicating factors have made the design of profile dies a skill based on experience and trial and error, rather than on an exact engineering methodology. The use of CAE, however, has enabled the processor to design complex profile dies with consistent accuracy.

#### **Sectioned Dies**

Many profile dies are split into sections. The die is sliced in a direction perpendicular to its major axis. It is then possible to change sections to produce slightly different profiles or to alter sections in the process of die development. In designing a sectioned profile die, it should be remembered that the flow channel should change size gradually and that supporting struts (spider arms) should be streamlined.

## ≻ COOLING

Commercial plastics are poor conductors of heat. In addition, they have high specific heats and frequently have poor thermal stability. This means that they will resist absorbing thermal energy and once it is absorbed they will resist giving it up. And prolonged exposure to this absorbed heat may cause degradation. In addition, heat must be removed from a formed thermoplastic in order for the part to retain its shape. Because of the poor heat transfer properties of thermoplastics, the heat removal step may control the rate of production. In general, semi-crystalline polymers require more heat to raise them to their processing temperatures than amorphous resins. This extra heat, which is required to melt the polymer crystals, must also be removed when the part is cooled.

## **Extrudate Cooling**

The heat contained in a molten extrudate is either lost to the surrounding air or to the cooling system/haul-off. Polymer extrudates are most commonly cooled by air or water. It is best to cool all sides of the product, however, this may not always be feasible. It may be necessary, especially for thick extrudates, to cool different parts at different rates. This is especially true for semicrystalline or filled materials that may distort if cooled unevenly. The aim should be to cool the extrudate as quickly as possible while ensuring that defects such as poor surface appearance, changes in physical properties, etc., are avoided. For example, with thick walled products, hot water may be used initially to prevent the outside of the extrudate from cooling too quickly.

## **Heat Calculations**

The amount of heat contained in a polymer melt can be calculated if the output rate, the specific heat of the polymer, and the melt temperature are known. From the output rate we can obtain the mass/ time and calculate the heat content or enthalpy from:

# Heat Content = $(Mass)_{Polymer} X (Specific Heat)_{Polymer} X [(Melt Temp)_{Polymer} - (Final Temp)] (Eqn. 18)$

The heat contents calculated for several polymers, using a typical processing temperature for the melt and 20°C (68°F) as the final temperature, are shown in **Table 15** (the differences between amorphous and semi-crys-

116 talline should be noted). Since the specific heat changes with temperature the average value, over the temperature range, is used (specific heat has units of cal/g°C, J/Kg°K, or Btu/lb°F). If the heat losses to the surrounding can be estimated, the heat content can then be used to calculate the amount of water required to cool the polymer extrudate.

Heat Contents of Some Thermoplastic Materials							
Material Abbrev.	Temperature Melt/Mold		Difference	Specific Heat	Heat to be Removed		
	(°C)	(°C)	(°C)	(Jkg <sup>-1</sup> K <sup>-1</sup> )	(Jg⁻¹)		
FEP	350	200	150	1600	240		
PES	360	150	210	1150	242		
CA	210	50	160	1700	272		
CAB	210	50	160	1700	272		
CP	210	50	160	1700	272		
PEEK	370	165	205	1340	275		
PET	240	60	180	1570	283		
PETP(C)	275	135	140	2180	305		
PEEL	220	50	170	1800	306		
POM	205	90	115	3000	345		
SAN	240	60	180	1968	354		
BDS	220	35	185	1968	364		
PC	300	90	210	1750	368		
ABS	240	60	180	2050	369		
PMMA	260	60	200	1900	380		
PPS	320	135	185	2080	385		
PS	220	20	200	1970	394		
ASA/AAS	260	60	200	2010	402		
HIPS	240	20	220	1970	433		
PP0	280	80	200	2120	434		
PSU	360	100	260	1675	436		
PETP(A)	265	20	245	1970	483		
PA 11/12	260	60	200	2440	488		
PA 6	250	80	170	3060	520		
LDPE	210	30	180	3180	572		
PA 66	280	80	200	3075	615		
PP	260	20	240	2790	670		
HDPE	240	20	220	3640	801		

TADIE

(Where (A) is amorphous and (C) is crystalline)

#### ➤ EXTRUDATE TAKE UP

The extrudate must be pulled from the die and converted to a suitable form for removal from the extrusion line, packaging, and subsequent sale. If the extrudate is flexible, it is commonly collected on a reel or cut to length and stacked.

#### Nip Haul-Off

For film and sheet, haul off is provided by a pair of nip rolls which grip the extrudate across its width. For a constant rate of extrusion, the rate at which the extrudate is drawn through the haul off determines its average thickness. This means that the nip rolls must be driven at a controllable constant speed and with no slippage of the extrudate. It is common to have one polished steel roll and the other rubber coated. The hardness of the rubber roll should be checked periodically as it may degrade with exposure to the atmosphere.

It is important that haul off rolls and other rolls in the take up system are all parallel to each other and parallel to the die face. It is easiest to make all rolls horizontal as this can be checked with a spirit level. In the case of blown film, the center of the nip rolls should be above the center of the die and should be checked with a plumb line. To assure that sets of rolls are parallel to each other, check that the distance between them is the same on both edges of the roll using measuring rods with a micrometer, or vernier adjustment.

#### Caterpillar Haul-Off

For pipes and some profiles, where the extrudate is rigid, a caterpillar haul-off is the most suitable. The simplest type consists of two rubber belts that rotate, one above the other, and grip the extrudate between a long parallel section of the two belts. Since the pulling forces can be quite high, the length of the caterpillar track should be sufficient to generate the pulling force without marking the extrudate. More than two tracks can be used in a single haul-off. Some manufacturers offer systems with up to 12 caterpillar tracks. The tracks are driven with a DC geared motor with a tachometer generator and thyristor control. The soft elements of the caterpillar are generally a shallow V-shaped in order to grip the extrudate. The following table shows typical properties for a range of caterpillar haul-offs.

Pipe Diameter	Speed Range		Max. F For	Gripping Length		
(in.)	(mm)	(ft/min)	(m/min)	(tons)	(in.)	(cm.)
0.5-3.5	12.7-43.8	4-100	1.2-30.5	0.4	30	76
1.25-12	31.8-305	2.5-50	0.76-15.25	1.0	48	122
2.5-12	63.5-305	2-40	0.6-12.19	1.5	48	122
3.5-16	89-406	1.3-25	0.4-76.2	2.0	60	152
3.5-25	89-635	0.3-20	0.1-63.5	2.5	60	152

## Other Haul-Off Systems

Belt drives can be used for small bore pipe and tubing above about 100 ft/min (30.48 m/min) where the caterpillar system becomes unsuitable. For wire and cable, the coated wire is looped round a capstan drum whose speed is main-tained to provide the haul-off.

#### **Reel Production**

There are two basic types of reeling systems; center fed, in which the wind up is driven by its central shaft and surface winding, where the rotation is generated by friction at the surface of the reel being formed. A surface winder is generally cheaper than a center winding system.

#### **Defects in Reel Production**

Problems become apparent in the inner layers of a reel as it grows. The two most common problems are telescoping and buckling. Both are due to compression of the inner layers by the outside layers. Compression decreases the diameter of the inner layers and the tension in them. If the tension becomes negative, then buckling occurs producing a characteristic star shaped mark on 118 the end of the reel. The loss of tension also reduces the friction between the layers and, if the reel is turned on its end, the layers will slip over each other to telescope the reel. Using larger cores reduces both of these problems as does reducing the tension in the extrudate as the reel diameter increases.

Surface winding would be expected to give winding under constant tension. However, if there is slippage between the layer of film on the lay on roll and the top layer on the reel, the maximum tension will be set by friction and not by the line tension. As the reel grows and its weight increases, this maximum tension will increase, leading to a winding tension pattern which is the reverse of what is required. Mounting the growing reel on a curved support, so that the force between reel and lay on the roll remains constant, even as the reel grows, reduces this adverse effect.

In center winding it is easy to wind under constant torque. Since the torque is the product of film tension and roll radius, as the radius increases the film tension decreases and so buckling and telescoping are less common. However, there is a tendency for outer layers to be loose. Various programmed winding systems are available to give a winding loading between constant tension and constant torque.

#### ► CUTTING AND STACKING

In sheet production the extrudate is cut across its width using circular saws or shears. For right-angled smooth cuts, it is essential to select the correct saw speed and blade for the polymer used and the thickness of the sheet. After cutting, the sheet is lifted by suction cups and stacked. In profile, cutting saws can be either under floor saws, where the cut goes underneath to the top, or immersion saws, that operate in the reverse direction. Small diameter pipes can be cut (up to a wall thickness of 0.75 in. (19 mm) for polyolefin pipes and 0.215 in. (5.5 mm) for UPVC pipes). Large diameter pipes are sawn with a planetary saw. Machinery is also available for chamfering the ends of pipes. Re-heating the ends of the pipe to soften them and then forming them over a correctly shaped mandrel, bells the pipe ends. The operation takes place on a moving system so that continuous line production is not interrupt-ed. Usually the pipe is transferred from the cutting and heating station to an adjacent position for the forming and re-solidification of the belled end.

Saws should be equipped with protective guards and extraction equipment to remove both dust and chips.

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# SECTION 6: EXTRUSION OPERATIONS

≻ START UP

PROCESS MONITORING

- > PROCESS CONTROL
- SHUT DOWN PROCEDURES
- ► PURGING
- STRIPPING AND CLEANING
- ➤ REPROCESSING
- MACHINE SETTINGS AND RUNNING CHART

## ≻ START UP

Start up refers to those procedures involved in getting a machine into full production. It is very important to have intelligent start up procedures, as adherence to such procedures will minimize dangers to the operator and damage to the equipment.

#### **Check the Heating System**

Before commencing operations, one should first check that the thermocouples are in their specified positions, of the correct type and are connected to the appropriate instruments in the control panel. If there is a thermoplastic material in the hopper, its feed gate should be closed. Make sure that the drive motor is off and that the speed is at the lowest setting possible. See that the cooling water is on and flowing at the correct rate through the hopper throat and the barrel cooling system. Turn off any cooling water to the screw.

Turn on the heating system and set the temperature controllers to a very low value (for example,  $50^{\circ}$ C). After this temperature is reached and held, by the control system, one can then begin heating up the machine for production to begin. The actual procedures may differ if the machine contains polymer or, if it is empty.

## Warming Up an Empty Machine

Check the records or obtain experienced advice on what machine settings are needed for the job at hand. Turn the main power switches on. Program the heat input to avoid an overshoot (melt temperature over-ride or MTO) while heating up the extruder in a reasonably short time. This is usually accomplished by using temperature controllers fitted with a three term (PID) control. As it is easier to heat a barrel than to cool it down, it makes sense to approach the final operating temperatures slowly to minimize the risk of a temperature over-shoot (unless it is known that the system will not over-shoot). This is most easily done by setting the temperatures about 20°C/30°F below the running settings, during initial barrel heat-up. Once the system has stabilized at the preliminary settings then heating to the desired running temperatures is carried out.

Such a procedure is necessary with some materials, such as unplasticized polyvinyl chloride (UPVC), where an overshoot can cause seri-

120 ous degradation before processing commences. Once the machine is at the set temperatures it should be allowed to equilibrate before any material is introduced into the barrel. Keep this time as short as reasonably possible. Otherwise, residual material present in the screw flights may burn when heated in the presence of air.

## Warming Up a Full Machine

When a full machine is being warmed up, it is important to ensure that it is heated in a way that avoids decomposition of the polymeric material. Decomposition can produce gases that, under pressure, can cause serious accidents by blowing hot material from the die. All temperatures should be set below the melting temperature of the particular material (for example, to 135°C for LDPE). Allow the machine to reach and equilibrate at these temperatures. Next raise the temperature of the die to above the melting temperature of the resin. Then raise the temperature of the front zone and the rear zone to above the melting temperature. Finally, working towards the center, raise the temperatures for a short time before purging. Only proceed if the purged material looks satisfactory, i.e., sufficiently melted, but not excessively hot.

## **Barrel Temperature Settings**

These are the temperatures set on the control instruments essential to reaching a desired melt temperature. Once an extruder is up and running, most of the heat input required for extrusion is mechanical heat supplied by the screw and drive system. The heaters are used only to warm up the machine and to "fine tune" the system during operation. One should remember that it is the melt temperature that is important and any barrel temperatures quoted in the literature are only guidelines.

When there is no experience with the processing of a particular grade of material, one should start with the lowest recommended settings. Unless the records show otherwise, use a flat temperature profile (each zone set at the same value). Recommended temperatures are 100°C/200°F above the glass transition temperature ( $T_{\sigma}$ ) for an amorphous thermoplastic material and 50°C/70°F above the melting temperature ( $T_{m}$ ) of a crystalline material. One should always remember that the melt temperature is almost always higher than the highest barrel set-point temperature.

## Equilibration

Once the machine is at the set temperatures, it should be allowed to equilibrate for about 20 minutes before material is introduced into the barrel. This time can be used to check that the die is clean and that all parts are operational. Review the production order for color and quantity and check that all necessary tools and equipment are in position. Check that ancillary equipment, such as the hopper or feed system, is clean and is functioning as required.

## Initial Purging

Check the records to determine which screw is needed for the job at hand. Ensure that the correct screw is in the machine and that it is installed properly. Once the machine has equilibrated at the running temperatures, start the screw rotating slowly and then introduce some material by hand into the hopper. Do not fill up the screw throat, otherwise the material may not melt completely and the un-melted granules may block the die and destroy any pressure transducers in the die and barrel. If everything appears satisfactory (for example, no melt frothing, spitting or motor overload) slowly increase the amount of material on the screw until it is covered with material. Then fill the hopper with material.

Check the melt temperature with a melt probe and also check the general appearance of the melt. Proceed only if this purged material looks satisfactory, the melt temperature is as specified and the motor amperage is not excessive.

#### Starting Up

Before starting up, a lead piece (produced in a previous run) or a piece of string is threaded through the cooling system and then through the haul off. The operator should wear suitable gloves. It is also useful to have an arrow drawn at the end of each roll that shows the direction of roll rotation and the path taken by the extrudate through the system. The haul off may then be moved to a convenient position in front of the die and the extruder run at a low speed.

When the material is extruded from the die it is attached to the lead piece by tying or by melt adhesion. The operator steadily pulls the piece through the system. The nips are often left open so that any joints or irregularities may pass. This drawing, or pulling, requires skill and it is essential that the operator is not distracted as he is guiding the extrudate over obstructions, around hot rolls etc. Usually the extrudate is cooled as it is being drawn away, as this gives the product strength. Once the extrudate has passed through the haul off drive is started and the speed adjusted before the nips are closed (save the lead piece for the next start up). Ensure that the haul off cooling system is fully operational and adjust its position to the running position relative to the die. Gradually increase both the screw speed and the haul off speed, providing that the machine operating parameters are within previously stated limits. Adjust the speeds to give approximately the correct dimensions and then adjust the die to produce the required dimensions. Any adjustments must be made slowly as this usually saves time.

#### **Safety Considerations**

One of the most dangerous times during processing is at start up. This is because material being heated in the machine may decompose and spit from the die. The operator is focused on getting the machine running satisfactorily and this involves close contact with machinery. So, great care should be taken at start up. In particular, no one should be allowed to stand in front of the die/nozzle and the hopper lid should be firmly in place, so that the screw cannot be seen (and therefore touched). No unauthorized person should be in the processing area.

#### **Temperature Problems**

If the use of a flat temperature profile causes:

- **Premature melting and bridging** of the material (resin) in the feed throat: gradually decrease the feed zone temperature
- High die pressure fluctuations: raise the feed zone temperature
- Melt temperature fluctuations: raise the transition zone temperature
- The barrel temperature to exceed a set point: slowly raise the set point temperature of that zone (raising the transition zone temperature can, how ever, reduce viscous heat generation and may cause incomplete melting)
- A loss of product gloss: raise the die exit temperature

# PROCESS MONITORING

During extrusion both process-based monitoring and product-based monitoring are used to achieve product improvements. Process-based monitoring watches production process conditions such as melt temperature and pressure. Product-based monitoring follows properties of the product, such as, clarity and thickness.

#### Pressure Measurement

Pressure measurement is now most commonly done with a pressure transducer, or sensor, which senses pressure or pressure changes (Figures 32a. & b.). Several different types of electrical pressure transducer are used, however, the most common type is the strain gauge pressure transducer. Because of its robust nature, accuracy and repeatability, ease of use and universal character, strain gauge pressure transducers are the most frequently used instrument for pressure measurement in extrusion processes.

It is usually melt pressure, such as the pressure within an extrusion die or barrel that this transducer is required to measure. The transducer is made in the form of a probe with its tip flush mounted to the inner diameter of the extruder barrel or die. Since, the high temperatures at the transducer tip can complicate temperature compensation, the measuring diaphragm and strain gauge are located away from the tip. The pressure is transmitted from the transducer tip to the measuring diaphragm, by means of an incompressible fluid such as mercury (in a filled capillary), to strain gauges in a Wheatstone bridge arrangement bonded to the measuring diaphragm. The electrical output from this gauge assembly is directly proportional to the pressure on the sensing diaphragm or tip. Cold start-ups and/or rough handling of the transducer will damage the tip relatively easily.



Figure 32a. Pressure Transducers



Figure 32b. Dynisco's Pressure Gauges, Transducers and Transmitters

#### **Important Pressure Measurement Locations**

There are several locations on the extruder where pressure measurements should be made. Along the barrel, measurements help determine screw performance and design; before and after the screen pack, measurements warn of potential high pressure situations and avoid flow restrictions between the screw and the die; at the inlet and outlet of the gear pump, pressure measurement ensures a constant melt flow to optimize pump performance and safety; and pressure measurement at the die maintains stable output and reduces scrap and material waste. At every location, pressure measurement helps ensure safety of equipment and personnel and increases machine output and efficiency.

#### **Temperature Measurement**

For reasons of cost and convenience, the thermocouple (TC) is the most common temperature-measuring element (**Figures 33a. & b.**). Basically, a thermocouple consists of two dissimilar wires that are joined to form a thermo-junction. A thermo-electric electromotive force (EMF) is generated at the junction of unlike metals if one thermo-junction (one end of the assembly) is made hotter that the other. The magnitude of the EMF depends on the metals used and on the temperature difference of the junctions. For a given combination, the more the one junction is heated, the greater will be the electrical signal. If one end is kept at a stable reference temperature (by cold junction compensation) then the thermocouple may be calibrated, so that a simple and accurate measurement of temperature can be made. The measurement may be displayed as°C or °F in analog or digital form. Usually, a thermocouple (TC) has the tip protected by a rigid metal sheath and a flexible metal covering protects the connecting wires.



"J" - Type Thermocouple

Figure 33a. Thermocouple



Figure 33b. Dynisco's Melt Thermocouple

The melt temperature is traditionally measured by pumping the material into the air (an air shot technique) and then immersing the tip of the TC into the melt. It may also be measured on-line by the use of a melt temperature thermocouple. Ideally, an immersion thermocouple should be employed unless the use of a surface mounted TC can be justified. It is recommended that the tip of the immersion thermocouple should be immersed approximately 0.25 in/6 mm into barrel/nozzle (so that it does not pick up the temperature of the metal). The sensing tip of such a TC should also be insulated from the body of the sensor. The same adapter as used for a pressure transducer may be used to house an immersion thermocouple. Commercial infra-red temperature transducers (sensors) are now available and their use promises to dramatically improve the extrusion process. Infrared line scanning thermometers may be used to measure the temperature and the temperature distribution across the width of extruded sheet to improve temperature uniformity.

#### **Thickness Measurement**

The thickness of sheet is often measured using a beta radiation gauge ( $\beta$  ray gauge). For a given formulation  $\beta$ -ray or particle absorption is directly proportional to mass per unit area. If the extrudate composition is fixed, then the sensor output can be set to continuously read product thickness. To do this, a low energy radioactive source, which emits beta particles, is mounted close to the moving sheet and the transmitted radiation is detected by an ionization chamber. The amount of beta particle transmission varies with the thickness and composition of the material through which it passes. If the composition is constant, the degree of ionization, and hence the conductivity of the chamber, will vary with the thickness. The changes in the conductivity can be converted to give a read-out of thickness or weight per unit area and/or used to actuate a control action.

The read-out from such a gauge gives the gravimetric thickness value or nominal average thickness. Gravimetric thickness is obtained from the weight a sample of known area and its density by dividing the weight by the area and the density. For an non-embossed sheet, the gravimetric thickness is the same as the geometric thickness. For an embossed sheet, the gravimetric thickness is not the same and is more relevant.

Because of concerns about radioactive emissions, X-rays are being used in place of beta rays. X-rays are more controllable and the X-ray beam can be tightly collimated, which reduces the radiation profile risk. Disposal of the radioactive isotope is also a problem that is eliminated.

#### Speed Measurement

In extrusion, the screw speed is the screw rotational speed that is usually measured in revolutions per minute (RPM). However, it should be remembered that it is the screw surface speed that is important. Speed must be measured and displayed very accurately as, screw speed controls how much material is pumped, how well it is mixed, shear history, melt temperature and melt temperature variations. Screw speed must, therefore, be set and read accurately and held to the set value. The processing machine must have an accurate display of screw speed (for example, a digital display) and the screw drive system must be powerful enough to keep the speed constant.

The speed of the screw may be feedback controlled using a tachogenerator, driven by the motor shaft, which generates a voltage that is proportional to the motor speed. This voltage is used as the feedback signal to control the motor speed. Screw speed may be measured directly by means of a transducer and a gear wheel. The gear wheel is mounted centrally on the screw so that when the screw is rotated, the teeth of the wheel pass the transducer. As each tooth passes the sensor, this creates a change in the magnetic field (a pulse) which is sensed by the transducer. When the number of pulses is counted per unit of time, the screw speed is obtained.

#### Width Measurement

Several different, non-contacting systems are used to measure the width or size of an extrudate as it is being produced (on-line). A single laser beam can measure pipe and tubing diameters. Two lasers, mounted at right angles, have been used to measure the size and eccentricity of round extrudates such as pipe, tubing, rod or cable. The collimated laser light casts a shadow on a detector that can determine the size to 0.002 mm (if the size is outside previously set limits, the haul off speed or the screw speed may be automatically adjusted). Pipe wall thickness, and uniformity, may also be determined by ultrasonic measurements so that low gauge and more uniform pipe can be

**126** manufactured. Monofilament diameter can be continuously monitored with a laser. Several strands can be monitored intermittently using a scanning or traversing laser.

When sheet is being produced its width can be determined by infrared measurements. The sheet is hotter than the roll on which it lies and an infrared detector can be used to sense the two edges of the sheet. Lasers are also used for sheet width measurement. Lay flat film width may be determined by ultrasonic measurements. The results of such measurements are used to control bubble size and air exchange in the bubble.

The cross-sectional area of irregularly shaped profiles can be measured with a pneumatic area sensor: this uses the Bernoulli effect to make online measurements that correlate with the cross-sectional area of the profile.

#### **Production Condition Recording**

This is the systematic recording of all production conditions, so that it is known how a component, or product, was produced. It must not be forgotten that the object of production is to produce component/product of the required quality, at a specified cost, within a specified time, and when required. To do this it is essential to keep accurate records. On many machines, fitted with microprocessor control, this can be done at the press of a button. Where this is not possible then, an appropriate record sheet should be completed at periodic intervals. Production samples should also be retained for future reference.

The importance of careful and accurate recording of process settings cannot be over-emphasized. Not only is it useful to have a full and accurate record for machine re-setting, but such records are useful, as well, for product liability reasons or to fulfill ISO 9000 requirements. A microprocessor-based machine will record all relevant data, print it out if required and then, instantly reset the machine when that particular job is re-run. Such instant re-setting saves a great deal of time and gives more accurate settings than that possible manually.

#### **Product Monitoring**

The machine operator may make checks on product quality or a control system or a separate system such as a robot-type device can make them. Verification of product quality at the point of manufacture can be documented in statistical quality control (SQC) records. To do this automatically, the system designer must identify what needs to be monitored and then equip the machine with appropriate transducers. The outputs from these transducers are monitored and if the signal from one or more is different from previously set limits, then various quality control (QC) actions may be taken. For example, an alarm may be sounded so that the product may be diverted to an inspection area. Microprocessor systems can ultimately provide total control of the extrusion line if the causes and effects of changes in machine and process parameters are correlated with machine adjustments.

Where robots are used for product removal, a robot measuring system (for instance a system based on the measurement of different dimensions) may be used to judge, or assess, the extrudate. The information that the robot measuring system generates may then be transmitted to the microprocessor control system and used for QC charts. On-line QC, based on product measurements, is therefore relatively easy and can be done for every piece of extrudate produced. If the product is judged to be defective, then it can be diverted for re-assessment or re-granulation and re-use.

## PROCESS CONTROL

Processing operations in manufacturing must be performed under conditions controlled by documented work instructions that clearly define the manufacturing process, the conditions employed, the variations allowed in production, the inspection procedures and the quality expected.

#### **Temperature Control**

In the extrusion shop, or factory, the thermocouple (TC) is the most common temperature-measuring element. When a thermocouple is used to feed a temperature control instrument with information, the well for the TC should be sunk as deeply into the barrel wall, as safety will allow. The base of the well should accurately match the shape of the TC tip. The TC may be spring loaded, so that when inserted into the hole, and twisted, it is locked in place under a controlled pressure. The TC will then sense the temperature of the metal close to the polymer. If a three-term control system (PID) is used, the barrel temperature may be controlled to within  $\pm1^{\circ}C/1.8^{\circ}F$ .

The temperature control circuit, which uses information from the TC, is commonly kept separate from the remainder of the control system, so that in the event of a stoppage the heat supply to the barrel assembly is unaffected.

#### **Thickness Control**

The control of thickness during production is very important. When extruding thermoplastic materials it is usually the rate of cooling that limits the rate of production. Accurate thickness control, therefore, saves not only on material use, but also gives faster cooling as the product can be held to the bottom of the allowed tolerance band.

Thickness variations in the extrudate (be it, sheet, cast or blown film) can have two basically different causes. 1. Gauge variations parallel to the extrusion direction (along the sheet). These are mainly due to variations in screw speed (caused by changes in material viscosity), haul-off speed changes, temperature variations and inhomogeneities of the raw material. 2. Gauge variations perpendicular to the extrusion direction (across the sheet), which are primarily due to a non-uniform die gap. The die gap and/or the die temperatures can be adjusted. If sheet is being extruded, it is often preferable to leave the die gap constant and to adjust the spreader bar.

Automatic profile control is possible using a system that automatically adjusts itself to control the size of an emerging profile. The die may have a number of heating/cooling units around its circumference, so that melt viscosity can be varied, to give the required output at that point, without adjusting the die gap. The thickness is measured and the temperatures adjusted accordingly. Such a system will improve the stability of the bubble and the output. Changes in conductivity, from a beta radiation gauge, can be used to adjust the motors that control roll speed during sheet extrusion.

## **Drive Control System**

A high-speed motor generally drives an extruder screw via a gearbox and pulleys. Such an arrangement, of a high-speed motor driving through a reduction gearbox, gives considerable speed stability (because of rotational inertia). For many extrusion applications, however, the speed must be held very precisely, in order to achieve a precise output, despite variations in extruder demand. Therefore, the drive system will also contain a regulator that compares input 128 signals from sensors (current, speed and torque) with reference inputs (manually set or computer derived). Corrective signals are then sent to the transformer-rectifier unit and thyristor controls with tacho-generator feedback hold the speed of a motor to approximately 1% from zero to full load. That is, at 1750 rpm the speed is held to ±17.5 rpm. If a digital, rotary pulse encoder together with a microprocessor is used, the speed may be held to ±1.75 rpm at 1750 rpm. As a result of this accuracy, DC and AC Flux Vector motors are widely used as extruder drives.

In some specialized systems, a pressure transducer will feed a signal into a process controller which, in turn, provide feedback control of the screw speed. In this way, a specific pressure can be maintained by varying the screw RPM and thus maintain a steady extrudate output. This system is usually used in the case where steady die output is necessary, and a melt pump cannot be used.

#### Automatic Pressure Control (Figure 34.)

Accurate temperature control can have profound effects on the quality and yield of extruded products. In the last forty years, temperature control has progressed through the original percentage timers, to Proportional, Integral and Derivative (PID) control as adapted by Ziegler and Nichols, to various self-tuning PID control algorithms both with and without the additional simulation of expert operator intervention called fuzzy logic.

More recently similar principles of automatic control have become available for the control of extruder pressure. The speed of the extruder screw is regulated to maintain a preset pressure at the die. PI and PID controllers, utilize algorithms more suited to pressure control than Ziegler -Nichols. Automatic tuning has simplified the installation and set up of controllers and control systems. As an example, the Dynisco ATC770 process controller utilizes two automatic tuning algorithms (Figure 34.). Together they are known as the SMART function. The TUNE algorithm is invoked in the Manual mode of operation, and is a "one-shot" algorithm, which will determine the P&I. parameters based on a change in the manual output. The unique ADAPTIVE tuning algorithm is based on the Dominant Poles Theory, and is a direct method based on the analysis of the input signal rather than the error signal. This algorithm is invoked in the Automatic mode of operation, and as the name implies, it is a full-time algorithm that adapts to changes in the extrusion process. Either or both algorithms may be shut off if manual tuning is desirable.

In addition to the obvious control of the input to the motor drive, today's controllers offer a number of beneficial auxiliary functions and options - all done without operator intervention.

Two or three auxiliary setpoints can be used in a scheme of alarms and interlocks. The use of auxiliary setpoints can prevent starting of the extruder prior to operating conditions having been reached. A setpoint may be set to indicate that it is time for a screen change, thereby avoiding dangerous pressure build-up. An absolute shutdown pressure setpoint may be set and latched to ensure safety of equipment and operator. An analog output directly proportional to the pressure is also available, and digital communications is a frequently supplied option.

Parameter information may be sent to another device in either analog or digital form. The controller can be a redundant device in a DCS and can report its activity to an SPC system.

The processor who chooses to automate can realize numerous benefits:

- Dramatic increase in safety in the workplace
- Monitor machine and screw condition
- Protect machinery from inadvertent damage
- · New controls often enhance the performance of old machines
- Relatively unskilled personnel can operate machines
- Improve yield and maintain consistency of product
- · Reduce reject product and maximize return on raw materials
- Allows duplication of conditions from run to run
- Allows one operator to supervise several machines
- Automatic tuning simplifies setup when changing from one product to another



Figure 34. Dynisco's ATC770 Pressure/Process Controller

#### **Automatic Quality Control**

Quality control may also be performed automatically by the production machine. Because of the power of the microprocessor used for the control system, it is now relatively easy to incorporate features that verify product quality during manufacturing, using the output from measuring sensors. Verification of product quality at the point of manufacture can then be documented in statistical quality control (SQC) records.

To monitor a process on-line, the production machine must be equipped with appropriate transducers and measuring equipment. These may include a melt thermocouple (in the die), pressure transducers (on either side of the breaker plate) and a (product) thickness measuring sensor. During production the output is automatically gathered from these transducers and displayed on a visual display unit (VDU). Production trends can then be easily spotted. If the changes exceed preset limits, the product is then judged to be out of specification. Such product may be rejected completely, or diverted for inspection and subsequent disposal decisions. Changes in the product can be then identified and quantified if the changes are capable of being statistically analyzed. Such analysis is not necessarily difficult, since the use of relatively simple statistics can yield a surprising amount of useful information. This information may be used to adjust the process in order to eliminate defects in the product, by altering die settings, extrusion speed, or some other process parameter.

# <sup>130</sup> ≻ SHUT DOWN PROCEDURES

It is most important to adopt a sensible shut down procedure as it can save a great deal of time and money. If, for example, the resin is prevented from burning then there will not be so much purging required on re-heating and the cost of a complete shut down and machine clean out may be saved.

#### After Processing a Heat-Stable Material

If a thermally stable plastic, such as polystyrene (PS) or polypropylene (PP), is being processed then, for a temporary (overnight) stop, it is usually only necessary to perform a few steps:

- 1. Close the slide at the base of the feed hopper
- 2. Turn off the cylinder heaters (leave the die heater on)
- 3. Purge the barrel clean by pumping the screw dry
- 4. When nothing more comes from the die, put any barrel cooling on maximum, stop the screw, and, when the machine is cool, turn everything off The machine is then ready for re-heating when required.

## After Processing a Heat-Sensitive Material

Decomposition, or burning of polymer in the extruder barrel, may cause color changes that will result in the subsequent product being rejected. If this happens a complete shut down and clean out may be necessary. To prevent this, it may be necessary to purge a heat sensitive resin with another, more heat stable, polymer that will withstand subsequent re-heating. If material oxidation is a problem (with, say polyethylene) then it may be best to leave the cylinder full of the material rather than pumping the screw dry before switching off. Do not shut off the cooling water to the feed throat until the temperature of the first zone has fallen below the melting point of the polymer.

## After High Temperature Operation

When high barrel temperatures are used, the shut down procedure should be modified to prevent thermal decomposition of the resin. One should:

- 1. Turn off the cylinder heaters (leave the die heater on)
- 2. Put any barrel cooling on maximum
- Periodically pump resin through the machine while it is cooling, but make sure the barrel temperature remains slightly above the melting point of the resin
- Close the slide at the base of the feed hopper and purge the barrel clean by pumping the screw dry
- 5. When nothing more comes from the die, and when the machine is cool, turn everything off

The machine is then ready for re-heating when required.

## **Display Notices**

Before leaving a machine, prominent notices should be displayed if the electric supply to the machine is left on, if the heaters are on or, if parts of the machine are still hot. Water and air supplies should be turned off. The motors and pumps should be isolated so that they cannot be started accidentally.

## ≻ PURGING

A method of cleaning the extruder screws and barrel, without pulling the screw and disassembling the machine, is through purging. There are several

reasons for purging (cleaning). One may want to change from one grade or color of material to another and/or change from one type of material to another. The net result is the same: material is wasted and production time is lost. Therefore, the object must be to minimize such losses by careful planning of the work being done to minimize the effects of changes.

#### **Minimizing Effects**

One should try to plan production operations so that the work flows in a logical sequence. Light-colored materials should be processed first. Easy flowing (high melt flow) materials should be processed before stiff flowing (low melt flow) materials. If it is necessary when changing from one material to another, use a polymer that processes at a temperature that is intermediate between those of two widely dissimilar materials.

It is very important to keep the materials handling equipment clean and to ensure contamination is not introduced during drying. The screw, barrel and die assembly must be thoroughly inspected to ensure that there are no worn or broken regions where material can stagnate, degrade, and then be released into the fresh polymer stream during production.

#### **Temporary Stops**

During a temporary stoppage the extruder may be periodically purged, by passing the material being processed through the machine at minimum screw speed. Do not allow material to build up on, or around, the die lips. If necessary, and allowable, coat the die lips with a light coat of silicone grease or other release agent.

If the purged material looks discolored then increase the frequency of this purging. During a minor repair, the heaters on the barrel should be set to low values to minimize thermal degradation.

#### **Purging Procedure**

In many cases, when changing from one material to another, the barrel is simply emptied (purged or pumped dry) and the new material is then introduced into the system. In other cases (for example, when changing from polycarbonate (PC) or polyamide (PA)) a faster changeover is obtained if the barrel is purged dry and a purge material is then used. Thermoplastic materials such as polystyrene (PS), high-density polyethylene (HDPE) or cast polymethylmethacrylate (PMMA or acrylic) are widely used as purging materials. Other proprietary compounds are available for purging purposes and these should be used as directed by the manufacturer.

In general, when changing from one material to another, for which the processing temperature is higher, set the barrel temperatures to those appropriate for the new material. If, however, they are lower, maintain the old temperature settings until purging is complete. Then discontinue the material supply to the hopper, empty the hopper, (carefully save the material), thoroughly clean the hopper by brushing and wiping, and replace it on the barrel (If it has been removed for cleaning). Any remaining material in the barrel should now be very soft as it has been heated (heat soaked) during the hopper cleaning. Empty the barrel into the air by rotating the screw. Introduce the new material and run a few pounds through the system as quickly as possible (that is, use the new material for cleaning or scouring). Allow the machine to stand (for approximately 10 minutes) and then rapidly run more of the new material through the barrel. Repeat this procedure until there is no sign of the old material.

If it is intended to shut the system down after using a purge, the

132 shut down procedure may then be followed once the purge material or compound is coming through.

#### **Purge Materials and Compounds**

A purge material is a polymeric resin used for purging. It is usually a high molecular weight (high viscosity) polymeric material that is relatively stable at processing temperatures. Natural, non-flame retardant grades of polymethylmethacrylate (PMMA) or high-density polyethylene (HDPE) are often used as purge materials.

A purge compound, also known as a purge, flushing compound or cleaning compound is a compound specifically designed, or used, to assist purging, or machine cleaning. It may contain large amounts of filler such as pumice. Before such purge compounds are used, it is advisable to remove the die as many purge compounds do not melt, or flow, like ordinary thermoplastic materials, and can damage the die polish or the die outlet.

#### Purge Compound Cleaning

If a proprietary compound is available for purging purposes then the manufacturer's instructions should be followed when purging. However, if they are used correctly, such purge compounds can save considerable production time. Their use will speed up the cleaning of the barrel unit when changing from one material to another, or, from one color to another. However, in some cases, purging will not be enough to remove contamination and stripping and cleaning of the barrel unit will be required.

Some purge compounds are intended for use over a specified temperature range. For example, one purge material may cover the range 180°C to 230°C/358°F to 446°F (which suits PS, PP and PMMA), another 230°C to 250°C/446°F to 483°F (which suits ABS and SAN) and another 250°C to 310°C/483°F to 590°F (which suits PC, PPO/PS and PBT).

## **Safety Considerations**

During purging, the die area should be shielded to protect the operator from being splashed by hot material. The operator must be trained in the purging procedures appropriate to the particular machine and must be aware of the dangers to himself and others. At the processing temperatures employed, a thermoplastic material can be easily degraded to give unpleasant, irritating odors and, if seriously overheated, some materials can produce a large amount of high pressure gas. Such gases should not be inhaled, or ingested, and should be treated as harmful. Gloves, long sleeved coveralls, safety footwear and a heat resistant face-mask should be worn during purging.

Purging must be done so that there is no danger to the operator or to any others in the vicinity. The purged material should be dropped into a bucket of cold water to minimize the formation of fumes and to protect anyone from touching this hot, sticky, dangerous material.

## **Note Purging Conditions**

During purging, the output and pressure should be noted. These values can be used to indicate screw or barrel wear. If the pressure needed to obtain a known output increases, compared to what was measured on a previous trial, then the screw, and/or barrel, may be worn and the maintenance department should be informed.

During purging note if any unusual noises are heard such as might be caused by the screw and/or the screw tip rubbing against the barrel. Note the pressure employed and the output with the new material, to determine if the screw subsequently wears.

#### STRIPPING AND CLEANING

In some cases, when changing from one material to another, purging is not enough and the barrel unit must be dismantled (stripped) so that it may be cleaned. Cleaning the extruder and die is a long and time consuming operation and, since nothing is produced, it is to be avoided if at all possible. It must be remembered that there is a danger that component damage may occur during the cleaning operation. Before commencing cleaning, the resin supplier's literature should also be consulted since plastics behave differently when heated: PVC may decompose to give corrosive acids and PC may stick tightly to the metal of the die or extruder.

#### **Safety Considerations**

The first stage of this operation is purging and the comments made in that section should be noted. The operator must be trained in the purging procedures appropriate to a particular machine and must be aware of the dangers to him and others. When the barrel, is stripped for cleaning, it should be done in an extremely well ventilated area. Because of this consideration, cleaning is probably best done away from the processing area. Gloves, long sleeved coveralls, safety footwear and a heat resistant, facemask should be worn when the barrel and screw are stripped and cleaned. It should always be remembered that the screw and/or die are heavy and difficult, or awkward, to handle. In many cases such components are also hot enough to cause serious burning. It must be remembered that most hot plastics, when molten, will stick to human skin, and cause destructive burns.

#### **Partial Dismantling**

In some cases, good results are obtained if the screw and barrel assembly is only partially stripped and cleaned. The die is removed, or swung to one side, and all accessible parts, including the joint faces, are cleaned. Before the screw cools, remove the screw cooling assembly from the rear of the screw (the rotary union and cooling wand), slacken any locking ring on the drive shaft and carefully extract the screw by either pushing or pulling. A "pusher", inserted through the hollow drive shaft at the rear of the machine, can be used to drive the screw out. This slowly forces the screw out of the barrel and exposes the front of the screw for cleaning. Alternatively, if the screw is to be pulled out, the nose of the screw must be removed (usually a left-hand thread) so that a "puller" can be inserted.

To strip exposed parts of the screw one may remove material by slowly pulling it away using pliers, while the adhering material is at the leathery stage. More material may then be removed by scraping. On non-chromium plated screws a steel wall paper scraper shaped to fit the channel shape may be used. The scraper may be wetted with a little cold water to minimize polymer adhesion. However, if in doubt, use brass hand tools, brass brushes and brass wool to minimize damage to the screw plating.

During the cleaning operation, all exposed parts of the screw and barrel should be inspected for wear and damage. This should be noted on the machine records and, if necessary, the maintenance department informed so that appropriate action may be taken.

# 134 Heater Handling

In many cases, it is better to dismantle and clean the extruder and die. As dismantling is done while the machine is hot, heat resistant gloves and a face shield must be worn. To keep the die as hot as possible, disconnect the heaters from each zone one at a time. Ideally the power supply should be disconnected by the person who is doing the heater band removal. That is, he/she should pull the electrical plug.

Remove the die heaters and thermocouples from a zone. When removing a heater, take special care not to damage it. For example, avoiding excessive flexing and careless handling. One should handle the heaters by the joint flange or terminal box and carefully store the heaters on a flat smooth surface (not the floor). All heaters and thermocouples should be labeled.

#### **Die Cleaning**

While hot, the die assembly should be carefully dismantled, taking special care not to damage metal surfaces that contact the melt. Disconnect the heaters and associated thermocouples and remove any pressure transducers, from the first part of the die that is to be removed. Then clean each piece of the die as it comes to hand. Remove as much material as possible from the die surfaces by slowly pulling it (use pliers) away while it is at the leathery stage. More material can then be removed using brass hand tools, brass brushes and brass wool. These may be wetted with a little cold water to prevent clogging. Before each part is removed, the part removed before it should be cleaned and placed on a clean smooth surface. Treat the die lips with special care as any nicks or scratches will introduce die lines, which are immediately obvious, on the product. When cleaning flat sealing faces ensure that their edges are not rounded off by the cleaning treatment.

Once the components are clean, each part should be inspected for wear and damage and repaired, or replaced, where necessary. As they are cleaned, the component parts of the die should be checked off against an authorized list before being certified as being ready for use.

#### **Re-assembly**

After cleaning, the die should be re-assembled and stored either on the machine or on a bench to minimize the risk of damage. As the die is being reassembled, the alignment between the various parts of the die should be carefully checked. If these components are out of alignment, hang-up and degradation of the extrudate may occur. Also, thoroughly check the sealing faces between the die, the head, and the barrel. Unless there is adequate contact pressure on the flat sealing faces, contamination from the lubricant used on the screw threads may occur. Clean all threads with a wire brush and apply molybdenum disulfide before re-assembly. Do not use oil or grease as these may oxidize and cause a subsequent blockage. If the die is to be stored it may be treated with a rust inhibitor: Storage must be in a clean dry storeroom on a properly constructed rack. If un-plasticized polyvinyl chloride (UPVC) or another halogenated polymer has been processed, then it will probably be necessary to wash the metal with hot water to remove any traces of acid, before the die is protected with a rust inhibitor.

Records must be kept of the work done on the die, where and what is stored, the tools required for maintenance and the tools required for use. Note that some dies remain the property of the customer although the extrusion firm is responsible for their use and care. If pressure transducers are installed then these must be carefully handled. Care should be taken to ensure that the correct mounting holes are used and that they are clean. If this is not done the sensing tip may be damaged, resulting in failure of the probe.

#### **Screw Cleaning**

After purging the machine with a brittle material such as polystyrene (PS) or PMMA, and when no more material can be squeezed from the screw (See **Purging**), remove the head assembly from the extruder. Following the manufacturer 's instructions, start to remove the screw from the barrel. If the screw can be removed in stages, the material adhering to the screw can be carefully removed with a brass scraper as each fresh portion of the screw comes from the barrel. Drop the scrapings into a bucket of cold water. When it is no longer safe to clean the screw in this way, the screw should be completely removed from the machine and placed on and strapped to a previously prepared rack.

The screw may then be cleaned, by scraping with a brass scraper and a brass brush. If the screw was purged with PS, then this relatively brittle material can be easily broken away when cold. Polish the screw with brass wool and/or a brass mesh cloth until the screw is gleaming. Once the screw is clean it should be inspected for wear, and/or damage and repaired where necessary. A light coat of an anti-corrosive spray should then be applied and the screw should be placed into storage on a special rack.

If the screw cannot be removed in stages then, after extracting the screw from the barrel, immediately remove as much material from the screw as possible with a brass scraper. Gently break away as much plastic material as possible when the screw is cold. If necessary, carefully reheat the screw as required. The reheat temperature should be approximately 50°C/100°F below the polymer's processing temperature where the adhering material has a leathery consistency and can be removed by pulling or peeling. After removing as much material as possible, heat cleaning may be required.

#### **Barrel Cleaning**

Prior to cleaning the barrel, make sure that any pressure transducers or exposed thermocouples have been removed while the barrel is hot. Clean the barrel by pushing or pulling a hardwood bung, which is a good fit, through the barrel. Or, one may clean the barrel by pushing or pulling a circular wire-wool brush, 2 mm/0.8 in. larger than the bore diameter, through the barrel several times. Insert the wire brush into the barrel and using a power system rotate the brush while moving the brush back and forth. Periodically clean the circular wire brush and do not push the brush past the feed opening. Then wrap emery cloth (240 to 280 grade) around the brush head and polish the barrel by the same process as above. Finally polish the barrel with a rag wrapped around the brush head until it is clean and gleaming. Then one should remove any particles, broken wires, or fragments, with a vacuum hose, and inspect the barrel for wear.

## **Other Cleaning Methods**

Flame cleaning (with a propane gas torch) should only be used when the polymer is seriously degraded and can be removed in no other way. This is because the die parts may be distorted by uneven heating. Flame cleaning must be gently done, if it is done at all. Use the lowest temperature flame, over as large an area as practical. Do not exceed a temperature of 450°C/842°F on the screw flights, as the hardness and wear resistance will be diminished. After flame cleaning, clean the screw with a soft wire brush and then polish with emery cloth (approximately 240 to 280 grade). Oven cleaning, with an air atmosphere, is also best avoided as it can produce fumes and drips. The polymer may also ignite when the oven door is opened. Uniform heating, and cleaning, by pyrolytic decomposition, may be accomplished, in an oven with a nitrogen atmosphere, at temperatures in the region of 550°C/1022°F. If solvents are used then great care is required, as the solvent may be flammable and/or toxic. Solvent baths should be treated with respect and a written procedure followed. Ultrasonic cleaning in a heated fluidized bed is preferred for screws and dies if the equipment is available.

It should be noted that copper causes rapid decomposition, or degradation, of polypropylene (PP). Therefore, copper cleaning pads should not be used to clean equipment used to process this material.

## ► RECLAIMED MATERIAL

The ability to reclaim out of specification product, produced during the extrusion of thermoplastic materials, is very often a mixed blessing. It can lead to the attitude that the production of offspec product does not matter. 'After all, the extrudate can be ground up to give reclaimed material and used again', is an often heard comment. Even if this is true, it should be appreciated that processing may dramatically change the properties of plastic materials. Even if this change is minimized, the lost production time, the cost of producing the scrapped product, the labor costs and the energy costs are considerable and are best avoided since they are directly reflected in the bottom line.

#### Terms Used

The re-use, by melt processing, of a formed component is only possible with thermoplastic materials. In the thermoplastics industry, a reclaimed thermoplastic material (sometimes called "re-grind") may be defined as a material that has been recovered from scrapped components and/or the feed system used to produce such components. One of the most common additives used with thermoplastic materials is reclaimed material. That is, the feed to the extruder is a mixture of virgin (new) material, reclaimed material, and a solid masterbatch (to impart color). All three components are generally used in granular form. Many machines run on 100% re-claimed material. Terms other than those used above are:

- Recycled plastics material This is a thermoplastic material prepared from discarded articles that have been cleaned and ground.
- **Regrind material** This is material that has been reclaimed by grinding (usually reworked plastics material). The letter R is used to identify this material. For example, reclaimed high-density polyethylene (HDPE) may be identified as HDPER.
- **Reprocessed plastic material** This is a thermoplastic material prepared from industrial scrap by other than the original processor.
- **Reworked plastics material** This is a thermoplastic material prepared from rejected production components that has been reprocessed in a fabricators plant after having been processed in that plant.

In this publication, the term reclaim (or reclaimed material) will be used and unless specified otherwise will refer to reworked plastics material.

#### **Re-granulation**

Re-granulation is the process of reclaiming output, which is usually achieved by feeding unwanted extrudate to a grinder to give re-granulated material. The grinder consists of a cutting chamber that contains rotating knives. The rotating knives reduce the extrudate in size until the cut pieces will pass through a mesh or screen into a collector. They may then be automatically blended at the required ratio with virgin material and fed directly back into the machine hopper.

Such machinery is extremely dangerous, as the rotor assembly is extremely heavy and has considerable momentum. This means that severe injury can be caused even if the rotor is turned by hand.

#### **Re-granulation/Recovery**

If the product does not fit the specification to be sold then, in some establishments, it is referred to as scrap, which implies that it is of little or no value. However, any thermoplastic material is expensive to purchase and turn into product. Even if the material can be fully recovered there is a large amount of money invested in the rejected product and extra energy and labor must then be spent on material recovery.

Materials for re-granulation or recovery may be of several types or categories:

**Type 1** - Edge trim, ends and incorrect product. Edge trim and ends are produced as part of the process and are usually totally recovered by re-granulation It is fed back to the production machine in a definite and prescribed ratio. Incorrect product based on clean, non-degraded resin, but having an imperfection such as the wrong dimensions or an incorrect surface finish, is treated similarly.

**Type 2** - Lightly contaminated product generated through process changes such as color or grade changes. This should be stored separately from Type 1 material and, as with all material that is to be recovered, protected from further contamination. It should only be recovered if the granulator can be subsequently cleaned to handle Type 1 materials.

**Type 3** - Difficult to handle output and/or contaminated product. This may consist of lumps and irregular pieces produced during start up or shut down which may be difficult to feed to the granulator. It may also contain purging, material generated by machine leakage, material removed from heaters etc. As this type of product is difficult to recover and/or dirty, it is often not recovered in the factory but kept separate and disposed of to another industrial concern. **Type 4** - Spilled raw material. Every effort must be made to avoid spilling the original resin. Although it is easy to collect, it is very difficult to clean or recover, and presents a falling hazard for operators. Within the extrusion shop spilled material is a tremendous safety hazard (danger of falls and/or fire). Once spilled, material must be swept, or vacuumed, up immediately so that it may be sold.

#### Safety

Everyone who uses the granulator must be trained in its use. Periodic inspections should be made for loose guards or parts. Safety instructions must be available and must be closely followed. If the machine jams and does not clear in, say, 10 seconds, do not push in more material but switch off. Then wait for the machine to stop, pull out the plug and follow the cleaning instructions. Avoid putting your hand or fingers near the blades as they are usually very sharp and, as the rotor is heavy and easily turned, severe injury can be caused even if the rotor is turned by hand. Use long-handled brushes and a vacuum cleaner to clean the rotor and blade assembly. If you must put your hands anywhere near the blades, then ensure that the rotor is clamped, so that it will not move.

After cleaning, the blades must be carefully inspected so as to

138 ensure that there is nothing present that will contaminate a subsequent batch of material. A trial run should be performed to ensure correct operation and a clean product. Ideally the granulator should be in a separate room from other equipment as it produces a great deal of noise and dust. A dust mask and hearing protection should be worn whenever a granulator is used.

#### **Material Changes**

During the production process there should be little or no change in the plastic material, as any change is usually undesirable. Some of the changes that can occur are:

- Water Contamination This is caused by the material absorbing water or by condensation.
- **Oxidation** This occurs when plastics are heated in contact with oxygen. They will 'oxidize' or combine with the oxygen. The first sign of this is a change in color and then a change in properties.
- Overheating If overheated, even when no air present, plastics may decompose or degrade. Often gases are produced which can be dangerous.
- Dust Contamination It is easy to generate static electricity on plastics, which attracts dust, or dirt, very quickly.

As changes and contamination are more common with reclaimed material than with virgin material, greater care is necessary when dealing with reclaimed thermoplastic.

## **Consistent Addition**

The exact amount of regrind that is suitable will have to be determined experimentally. Once found, then the content must be held as precisely as possible if consistent product is to be obtained. If the feed to the extruder is not consistent, then inconsistent product will be obtained. The differences may not be discernible to the naked eye, but they are large enough to cause rejection of the product, either because of appearance or thickness variation. So, one must ensure that the agreed ratio is adhered to during production.

No matter what ratio is used, the materials fed to the extruder must be clean, dry and consistent. It must be emphasized that to obtain a consistent output, a consistent feed must be used. This is because of the feeding differences experienced with different feed forms.

## **Regrind Care**

Great care should be taken to ensure that reclaimed material (regrind) is clean, dry and of regular particle size. If the regrind is dirty, then die or machine damage may occur and the appearance of the product will suffer. If the material fed to the machine contains unacceptably high moisture levels, the properties of the extrudate will be affected. In the case of a clear material, the clarity may be affected and for all materials (both clear and opaque) the quality of the surface finish may be reduced by streaking or surface imperfections.

If the feed is not of consistent particle size, then the material will not feed in a uniform way and an inconsistent product will be obtained. The differences between extrudate batches may not be discernible to the naked eye, but they may be large enough to cause rejection of the product, because the size of the extrudate is incorrect.

To get the best results from an additive, such as regrind, it must be very well dispersed throughout the basic polymeric material. Melt mixing, using a compounding extruder, is important to the polymer industry, as it gives the good dispersion required. However, such compounding is expen-
sive, so simple, tumble-mixed blends of virgin and reclaim are frequently used. This produces a lower level of dispersion, but will save on costs and give operational flexibility.

### **Regrind Stabilization**

Care should be taken to ensure that the original material contains sufficient stabilizer, so that reclamation is possible without degradation, or a color change, occurring on re-use. It is particularly important with unplasticized polyvinyl chloride (UPVC), to ensure that adequate stabilizer is present in the original material. For any thermoplastic material, it is very important to avoid a long residence time in the extruder barrel, particularly at high melt temperatures, as it will cause material degradation.

### **Product Identification**

Recyclable products or components should be marked with a symbol or abbreviated term. Legends suitable for the generic identification and marking of plastics products are suggested by standards organizations such as the International Standards Organization (see ISO 1043). In the simplest case, the series of letters associated with a plastic material (See Table 1.) are stamped onto a product between inverted (reversed angle) brackets (for example, >ABS<).

ISO has also suggested how to identify fillers, flame-retardant, plasticizers etc. The presence of a flame retardant may be indicated by a molded or printed legend which contains FR, as in >PA 66GF30-FR(52)<. This legend shows that the product is made from a nylon 66 material which contains 30% glass fiber. The FR(52) indicates that the nylon contains red phosphorous (code number 52) as a flame retardant; other flame retardants have their own number.

### MACHINE SETTING AND RUNNING

Process settings refer to details, with regard to the material, the die and the machine settings used to produce an extrudate, which are recorded on the process setting sheet. This is a sheet used for keeping a record of data regarding each run. The importance of careful and accurate recording of process settings cannot be over-emphasized. Not only is it useful to have a full and accurate record for machine re-setting, but such records are useful for product liability reasons and for compliance with ISO 9000 requirements. With date marking of extrudate now easily possible, the precise details of how a particular run was produced can be assembled and kept, easily and cheaply. A micro-processor-based machine will record all relevant data, print it out if required, and instantly reset the machine when that particular job is re-run. Such instant re-setting saves a great deal of time and gives more accurate setting.

It must not be forgotten that the object of production is to produce components/product, of the required quality and quantity, at a specified cost, within a specified time, and when required. To accomplish this, it is essential to keep accurate records of how a particular extrudate was produced. Also, it must not be forgotten that, to be effective, a quality management system must have excellent record keeping. Accurate documentation helps ensure that specified procedures are being followed and that such procedures are yielding the desired results. Records are the objective evidence to demonstrate that components offered for acceptance meet the purchasers requirements and that the suppliers inspection system complies with the requirements of the ASTM, ISO and any other quality standards required. The following is a general outline of what is recommended for recording. This outline will, however, need to be amended to suit a particular application.

		Ext	trusion D	ata Record	1			
Personnel I	Details							
Date			Shift					
Senior oper	ator		Other	operators				
Machine se	tter		Inspe	ctor				
Material De	etails							
Material use	ed		Suppl	ier	Т	rade nar	ne	
Grade			Lot no	).	C	Color		
Max moistu	ire content '	%						
Alternative	material det	ails						
Formulatio	n & Blend I	Details						
Virgin %			Regri	nd %	I	Masterba	atch %	
Other %								
Mixing deta	ils							
Preheat at °	C/°F for hou	Irs.						
Extruder Id	entification				_		_	
Manufactur	ers name		Mode	no.	F	actory II	)	
Screw type/	details							
Adapter typ	е		Factor	ry ID	1	Heater si	ze/type	
Screen pack	k/breaker pl	ate						
Air/vacuum	required							
Special requ	uirements							
Die Identifi	cation		M I -		-		_	
Manufactur	ers name		IVIODE	INO.	H Inila	actory II	J	
No. of zones Heater type & details								
Air/vacuum required								
Appellary 9	Downotroo	m Equina	ant					
Alluliary a	DOMIISTICA	in cquipn	iein					
Air/vacuum	roquirod							
Special regu	uiromonte							
Safety Faui	nmont							
Details	pinoin							
Air/vacuum	required							
Special regu	uirements							
Instructions	to Machin	e Onerato	r					
During the	run watch o	ut for						
Inspect the	extrudate e	verv	minutes	and if it doe	es not r	pass the	specified	tests
then take th	e following	actions			00 H01 p		0000000	
Specified te	sts							
Corrective a	actions							
Machine Se	ettinas							
			Temps.	Set or	Actual	l or Mea	sured at	Time
			°C/°F	Target	1	2 3	4 5	6
Drier						-	-	-
Machine ho	pper							
Material								
Hopper blog	ck							
Cold water	supply							
Barrel -	Rear	Z1						
	Middle	Z2						
	Middle	Z3						
	Front	Z4						

#### **Machine Settings**

J	Temps.	Set or	Actual or	Measured	at Time
	°C/°F	Target	12	34	56
Adapter	Z5				
Die	D1				
Die	D2				
Die	D3				
Die	D4				
Die	D5				
Die	Db				
IVIEIT Molt	IVI I				
Watar bath WP 1	IVIZ				
Water bath - WP 2					
Water bath - WB 2					
Athers					
Water Flow Rate	nals/min or l/s				
Oil cooler	gais/initi or 1/5				
Hopper block					
Cooling	C1				
Cooling	C2				
Cooling	C3				
Cooling	C4				
Cooling	C5				
Pressures	psi or N/m²				
Air					
Water					
Melt - 1					
Melt - 2					
Back pressure					
Air Flow Rate	cu ft/min or l/s				
Drier					
Die					
Cooling	1				
Cooling	2				
Bower	3 amne				
Main ext motor	annha				
Athers					
Times	minutes				
Time in drier	minutos				
Cooling time					
Take out time					
Speeds	m/s or ft/min				
Roll/belt	(R1)				
Roll/belt	(R2)				
Roll/belt	(R3)				
Roll/belt	(R4)				
Roll/belt	(R5)				
Roll/belt	(R6)				
Extrudate speed					
Speeds	rpm				
Screw speed					
Koll/belt	(K1) (D0)				
Koll/belt	(K2)				
KUII/DEIT	(K3) (D4)				
KUII/DEIL Boll/bolt	(H4) (D5)				
KUII/DEIL Boll/bolt	(H3) (D6)				
NUII/DEIL	(חט)				

142 Distances mm or inches Die gap Die to haul off roll Freeze line height Length of water bath-1 Length of water bath-2 Length of water bath-3 Others Weight kg or lbs Total extrudate Trim Product Critical Dimensions mm or inches 1 2 3 4 5 Visual appearance Observations

# SECTION 7: DEFECTS AND DEFECT DETERMINATION

DEFECT DETERMINATION

- MINIMIZING DEFECTS
- > EXTRUSION DEFECTS AND CAUSES

### PRODUCTION PROBLEMS AND FLOW BEHAVIOR

### DEFECT DETERMINATION

Defect determination is a procedure used to determine the origin of a defect. Such a procedure should be logical and systematic if defects are to be eliminated. This procedure is based upon one suggested by John Brown in his book "Injection Molding of Plastic Components" published by McGraw Hill 1979. Defects should be clearly described and all the possible causes for their occurrence should be examined. When the cause has been identified the necessary steps should be taken to eliminate the defect and to prevent its recurrence. Those defects that cause the most rejects should be identified, using Pareto analysis, and eliminated first.

### **Equipment Required**

Before a defect determination exercise is performed the person concerned must be equipped with the appropriate equipment. These include:

- A pen, notebook and sample marker
- Bags or containers to hold the samples
- Weighing scales accurate to 0.01 g
- A knife and small saw
- A magnifying glass
- A portable pyrometer
- Gauges or jigs for sample measurement
- A stopwatch
- Samples of the product, that have been approved as being of the correct quality
- A light-box to check the color of the product under standardized conditions

### Name the Defect

Many defects are given more than one name. Ensure that the terms used to name a defect are unambiguous and are known to all concerned. The alternative names for a defect should be listed and components that exhibit that defect should be available for inspection and comparison.

### **Describe the Defect**

Describe all common defects in the simplest possible terms. A possible cause may be revealed by this description. For example, voids in the extrudate from the presence of moisture may be described as "foaming" or "out-gassing".

## <sup>144</sup> Defect Percentage

The defects, or rejects, from an extrusion run may be named by such terms as: voids in extrudate, contamination, degradation, granules/nibs in the output, gassing, color distribution or poor surface finish on the output. The total (100%) defects, or rejects, are sorted into their named fault categories. The quantity, in each fault category, is then expressed as a percentage of the total rejects (Pareto analysis) in a three column "league table". The most frequently seen defect is placed at the top of the table (1st column) followed by the remaining defects in descending order of their frequency. The percentage of each defect is entered in the second column with the cumulative percentage total shown in the third column.

	% of Total	Cumulative %
1. Voids	35	35
2. Contamination	25	60
3. Degradation	15	75
4. Granules/nibs	10	85
5. Gassing	8	93
6. Poor color distribution	5	98
7. Poor surface finish	2	100

A Pareto diagram may then be drawn of cumulative percentage (vertical axis) against the type of defect (horizontal axis). The major defect is placed on the left-hand side and the least common on the right. Such a diagram often shows that the bulk of the defects are of three or four types. This type of analysis indicates where efforts should be concentrated to eliminate the most defects in the shortest time.

### Procedure

Make a complete record of the machine settings before making any change. Adjust only one setting at a time, choosing the easiest one first. If this change does not eliminate the defect, go back to the original settings after making notes of what was done and taking marked samples of what was produced. Allow sufficient time for the machine to respond to the changes and to come to equilibrium with each change before samples are taken.

### **Defect Cause**

Finding the cause of the defect may be a lengthy process since it requires consideration of material, machine, die and process.

Observe the effect of regrind addition on component properties and on the processing characteristics (compared to the virgin material). If the defect is apparent with different lots of the same manufacturer's resin or with material from a different supplier, this indicates the material is not at fault.

If the defect disappears when the production is shifted to another machine, the source of the problem is most likely the processing conditions used and/or the consistency of production on the original machine.

If the defect disappears when a different person operates the machine, then the fault may be due to the operator involved in the process.

### Effect of the Defect

If the defect renders the component unusable or non-salable it must be rectified. If it is only of minor significance, then it may be unnecessary to try to eliminate it entirely. However, clearance to continue production with the minor fault must be obtained in writing.

### **Defect Responsibility**

Determine where the responsibility for the defect lies. This may only be of academic interest, but if the defect recurs, the operator – as well as the material, machine, die and process – needs to be checked.

### **Avoidance Action**

Take immediate, and appropriate action to avoid the defect. If this is not done, then the reasons for continuing production with the defect should be entered on the production record sheet.

### **Prevent Recurrence**

Take steps to prevent a recurrence of the defect. Make full records of the conditions used when the defect was present and of the conditions used when it was eliminated. Note any repairs and alterations that were made to the die or the machine and any variations in type, grade or quality of material. If rework is used, note the proportion used and the quality. Keep labeled samples of what is being produced – both with and without the defect.

No defect-determination exercise is complete unless all of the above points have been considered. Making defective products, even though they can all be recovered, re-ground and the material used again, is uneconomic and non-productive.

### > MINIMIZING DEFECTS

In many cases, the production of a product that contains defects can be minimized if the operator makes some basic checks before, and/or during, production. For example, he must check that the extruder appears to be functioning correctly and that the machine is set correctly. The material fed to the machine must be carefully checked.

### **Feedstock Checks**

With regard to the feedstock, the operator must check that the:

- · Correct types of material are being used
- Correct grade of materials are being used
- Correct level of materials (such as masterbatch and regrind) are being used
- Material is free of contamination
- · Material is dry and free from an excessive concentration of fines

### **Temperature-Related Settings**

Temperature-related settings or parameters cause many problems. The operator must check that the:

- Thermocouples are not loose in their mounting holes
- Thermocouples are of the correct type
- The feed throat is at the correct temperature
- The hopper is at the correct temperature
- The barrel and die are at the correct temperatures
- The melt is at the correct temperatures (in order to be accurate the melt temperature must be measured with an immersion thermocouple)
- The specified volumes of water are circulating through the feed throat and rolls so they are at the required temperatures

# <sup>146</sup> Speed Settings

Check that the rotational speed of the screw (in rpm) is being correctly measured and displayed, and that the draw down, or haul off, speed of the rolls (in rpm) is being correctly measured and displayed.

### **Die and Ancillary Equipment Checks**

With regard to the die, the operator must check that:

- The die lips are clean and polished
- · The die is properly set
- · Any adjustable parts of the die function smoothly and correctly
- The correct screen pack has been fitted
- The ancillary equipment is properly set and that all the parts function smoothly and correctly
- All parts of the die and the ancillary equipment are at the correct temperature

### **Consult the Setting Sheet**

The operator must also check that the machine is set as specified by the setting sheet and/or, by the supplier of the material. The operator must also check that the pressures, temperatures and speeds are achieved in production and that accurate records are kept. Samples of product must be taken at approved intervals and submitted to quality control.

### **> EXTRUSION DEFECTS AND CAUSES**

Process Problem (Location or Cause)						Pr	ot	ole	em	0r	Ту	pe	of	De	fec	t			
Hopper Throat	12	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18 19	9
Bridged	*	*																	
Cooling faulty		k																*	
Barrel	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	1819	)
Feed zone temp too high																		*	
Feed zone temp too low	*														*				
Barrel temps too high						*						*	*				*		
Barrel temps too low	*	k											*		*			*	
Temp profile wrong	*		*			*					*	*		*			*	*	
Temps fluctuating			*			*													
Cooling required																*			
Venting required				*				*											
Worn	*	k	*			*													
Process Problem						Pr	ot	le	em	or	Ту	pe	of	De	fec	t			
(Location or Cause)																			
Die	12	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	1819	)
Temps too high						*						*	*				*		
Temps too low	*	k											*		*			*	
Temps fluctuating			*			*													
Incorrect design	*		*		*	*		*		*	*	*	*	*	*				
Requires adjustment	*													*	*				
Requires cleaning	*	k				*				*			*	*	*				

Process Problem (Location or Cause)		Problem or Type of Defect																	
Screen Pack Blocked	12 * *	3	4	5	6	7	8	9	10	11	12 *	21	3	14	15 *	16 *	17	18	19
Wrong rating used	*	*	*						*	*		,	r	*					
Screw	12	3	4	5	6	7	8	9	10	11	12	21	3	14	15	16	17	18	19
Worn	* *	*			*														
Cooling required										*	*				*		*		
Incorrect design	*					*			*	*	*				*			*	
Insufficient mixing										*	*	,	r						
Requires cleaning	* *					*								*					
Drive System	12	3	4	5	6	7	8	9	10	11	12	21	3	14	15	16	17	18	19
RPM varying	*	*																	
Drive belts slipping	* *																		
Gearbox oil level low																			*
Gearing incorrect																			*
Gearbox worn																			*
Plastic Material	12	3	4	5	6	7	8	9	10	11	12	21	3	14	15	16	17	18	19
Requires drying	*	*				*													
Polymer quality poor	* *	*	*		*	*	*	*			*								
Wrong grade used	*	*		*	*	*					*								
Extrusion Rate	12	3	4	5	6	7	8	9	10	11	12	21	3	14	15	16	17	18	19
Too high			*		*	*		*	*	*									
Too low					*		*			*								*	
Additives	12	3	4	5	6	7	8	9	10	11	12	21	3	14	15	16	17	18	19
Colorant not premixed										*	*								
Colorant incompatible			*							*	*	,	r						
Reclaim contaminated	*			*	*								r	*					
Problem or Type of Defect																			
1. Low output			1	1.	(	Co	0	r c	list	rib	uti	or	ı p	00	r				
2. Erratic or intermittent outp	ut		1	2.	(	Co	0	r٧	ari	atio	on		Ĩ						
3. Surging output			1	3.	F	٥ <sup>0</sup>	or	S	urfa	ace	fi	ni	sh	or	n th	ie c	outp	out	
4. Voids in extrudate			1	4.	S	Sti	rea	aks	s in	th	e o	วน	tp	ut					

- 5. Contamination
- 6. Degradation
- 7. Varying viscosity
- 8. Granules/nibs in the output
- 9. Gassing

- 15. Excessive barrel pressure
- 16. Temperature over-shoot
- 17. Material leakage
- 18. High motor amperage
- 19. Material adheres to screw

10. Uneven flow

### > PRODUCTION PROBLEMS AND FLOW BEHAVIOR

Since most processes for plastics involve flow, one might expect that rheological concepts would be understood and widely used. In practice, however, quantitative application of rheology is still somewhat limited. This is because of the complex nature of the flow behavior of polymer melts, the complicated flow patterns involved in many shaping operations (for example, the filling of a profile die), and the fact that melt cooling occurs while the melt is still flowing. Nevertheless, an understanding of flow behavior can often help to overcome processing problems. The use of computer programs, which combine rheological and thermal data, is very important in computer-aided design and engineering.

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# <sup>148</sup> Effect of Flow Channel Shape

For a flow channel of a fixed cross-sectional area, greater flow rates occur when the channel is circular than when it is in the form of a slit. It can be shown that the narrower the slit, even though the crosssectional area is the same, the lower the flow rate (under a constant delivery pressure). This difference is relevant in the extrusion of complex shapes, where the extruded crosssection is made up of components of different thickness and shape.

The extrudate cross-section may be in the form of a dumbbell with two almost spherical parts being bridged by a slit. In this case, flow will tend to be faster in the round sections and slower in the bridging slit, leading to undue stretching, and possible tearing, of the central parts of the extrudate. Even worse defects may occur when the extrudate cross-section is like a half dumbbell, or key cross-section, with one circular section attached to a slit. In this case the extrudate will tend to curl up as it leaves the die.

While such designs are best avoided, the problem may be solved by reducing the parallel portion of the die in those regions where the flow is slowest (in the above examples, in the slit zones). Alternatively, one may throttle the flow in the regions where flow is fastest by the use of a pin or mandrel, inserted in the flow channel at the entrance to the die parallel.

### **Unbalanced Flow in Tubular Extrusion**

Tubing is made by extruding polymers through the annular gap between a circular die and a pin or mandrel. It is sometimes found that the inside wall of the tube has a number of transverse ripples that arise from variations in radial flow velocity through the die.

To understand this phenomenon, consider the following. In a simple rod die the flow rates is greatest in the center of the die and zero, or almost zero, at the wall (with simple Newtonian liquids, such as water, the velocity profile is parabolic in shape. The viscoelastic behavior of polymer melts usually makes this profile somewhat flatter). If one extrudes over a very thin pin (of a thickness approximating to that of a thin piece of wire) the velocity profile will be little affected. The flow rate a short distance from the thin pin will be much greater than the flow rate the same distance from the die wall. As the diameter of the pin is increased the difference becomes less. However, the flow rates will be higher nearer to the pin than to the die wall. Since the melt is coming out faster on the inside of the tube this tends to cause buckling and hence rippling on the inside of the tube. One way of reducing this is to fit a restrictor bulge or ring around the pin to slow the flow in this region.

### **Uneven Die Swell**

As a general rule, die swell will increase with shear rate and decrease with the length of the die parallel. If a complex section with varying cross-sections is being extruded, then different shear rates will exist at the walls of the different crosssections. The shear rates are the highest where the cross-section is least. It may be possible to compensate for these differences when carrying out die sizing **(See Sizing of Die Components)**. However, if it is found that at one point the swell is lower than elsewhere, then the die parallel at that cross-section may be reduced to compensate. If this is done, then one should machine the back end of the die parallel and ensure that all changes in cross-section are gradual.

### Die Swell and Parison Sag

In extrusion blow molding, it is common to extrude a parison vertically down-

ward. The wall thickness, and thus the tube diameters, will tend to increase, due to die swell, while at the same time they will also tend to decrease as the parison sags under its own weight. While the swell effect will be independent of parison length, the sag will increase as the tube length increases. It is possible to compensate for this by using a tapered die and pin that can be made to move axially, with respect to each other, to open the die gap during the extrusion of the parison. The parison movement may be programmed either as the result of trial and error experiments, or by use of theoretical data.

### **Melt Fracture and Sharkskin**

It is often found that surface defects on an extrudate will show a regular pattern on the surface. With large diameter extrudates, this is likely to be in the form of roughness transverse to the direction of flow, called sharkskin. With smaller diameter products, it is a helical form called melt fracture. If the problem appears to be melt fracture, then the following possibilities should be considered:

- 1. Reduce output rate (not usually commercially desirable)
- 2. Increase the melt temperature (which will increase cooling times)
- 3. Taper the die entry further
- 4. Taper the 'die parallel' by up to 1°  $\,$
- 5. Use a polymer of lower molecular weight

If the problem is identified as sharkskin, then the following possibilities should be considered:

- 1. Reduce output rate
- Vary the die temperatures. Either raising or lowering of the die body temperature may help, while extra heating of the die exit may be particularly beneficial

### **Steady State Operation**

Unlike injection molding, extrusion is a continuous operation that requires keeping the materials used, and the operating conditions, constant over very long periods. Changes to machine settings or controls should be made only after due consideration. If changes in settings (such as speed or temperature) are considered necessary, then they should be altered one at a time. After each change, a reasonable time should be allowed for the system to equilibrate.

The operator must appreciate that the reaction time of the extruder to changes differs significantly, depending upon what is being altered. For example, a change in pressure will cause a rapid change in extrudate output and power input. However, a change in a barrel temperature setting will take longer to come to equilibrium, because of the thermal mass of the machine, together with associated output and power input variations. Screw speed changes will also cause a rapid change in extrudate output and power input. It will, however, cause a much slower change in the melt and machine temperatures.

# SECTION 8: GUIDES FOR THE FOLLOWING MATERIALS

- > ABS (Acrylonitrile-butadiene-styrene)
- > HIPS (High impact polystyrene)
- ≻ PA 6 (Nylon 6)
- ≻ PA 66 (Nylon 66)
- LDPE (Low density polyethylene)
- LLDPE (Linear low density polyethylene)
- > HDPE (High density polyethylene)
- ≻ PP (Polypropylene)
- > PMMA (Polymethyl methacrylate)
- PPVC (Plasticized polyvinyl chloride)
- > UPVC (Unplasticized polyvinyl chloride)

## ≻ ABS

1. Common Name 2. Abbreviation(s) 3. Alternative Names	Acrylonitrile butadiene styrene ABS
ABS plastics: styrene/copolymer	blends; ABS copolymer; ABS terpolymer
4. Some Suppliers	5.Trade Names or Trade Marks
A Schulman Inc.	Polyman ABS
Ashley Polymers Inc.	Ashlene
BASF	Terluran
Bayer	Novodur
Chi Mei Corp	Polylac
ComAlloy International Corp.	Comalloy ABS
Daicel Polymers	Cevian
Dow Carbide	Magnum
DSM (Dutch State Mines)	Ronfalin
Elf Atochem S.A.	Arrhadur
EniChem	Sinkral
Ferro Corp	Starflam ABS (ABSFR)
General Electric Co see G.E. Pla	astics
G.E. Plastics	Cycolac
G.E. Plastics (Fr.)	Ugikral
Hoechst Daicel Polymers	Cevian
Industrial Resistol	Epolan
Lati	Lastilac
LG Chemicals	LG ABS
LNP Engineering Plastics Monsanto	Thermocomp (ABS-30%GF) Lustran

Monsanto	Lustran Ultra ABS
Polikemi AB	Polyblend (PC/ABS)
RhônePoulenc Chimie	Alcoryl
RTP	RTP 605 (ABS-30%GF)
Snia	Sniasan
Thai Petrochemicals	Porene
Thermofil Inc.	Thermofil ABS
Toray Industries Inc.	Toyolac
ABS Alloys - other Material(s) N	ot Known
Hoechst Daicel Polymers	Novalloy
ABS/PA Alloys	
DSM (Dutch State Mines)	Stapron M
DSM (Dutch State Mines)	Staloy N
ABS/PBT	
Daicel Polymers	Novalloy B
ABS/PC	
Bayer	Bayblend
Daicel Polymers	Novalloy S
Enichem	Koblend
G.E. Plastics	Cycoloy
Lati	Lastilac
DSM (Dutch State Mines)	Stapron C
ABS/PSU	
USS Chemicals	Arylon T
ABS/PVC	
DSM (Dutch State Mines)	Ronfaloy V
6. Material Properties	

ABS plastics were originally made by blending a lightly crosslinked nitrile rubber (NBR) into an styrene-acrylonitrile (SAN) copolymer. These materials are now commonly produced by polymerizing styrene and acrylonitrile, in the presence of polybutadiene (BR), in the latex form. This process results in a product consisting of BR grafted with styrene-acrylonitrile (SAN) copolymer. The grafted BR phase (the rubbery phase) is then melt compounded with rigid (SAN) material to give ABS. Also added at this stage are additives such as stabilizers, lubricants and colorants. The plastic phase, which is usually called the "rigid" phase, is SAN which comprises more than 70% of the total composition. The grafted polybutadiene phase, which has a high rubber content, may also be used as an impact modifier for other plastics such as polyvinyl chloride (PVC).

By varying the monomer ratios, the way in which they are combined, the size (and the amount) of the rubber particles, the cross-link density of the rubber particles, and the molecular weight of the SAN, it is possible to produce a wide range of materials. These variations lead to materials that may differ in their impact strength, ease of flow, color, etc. In general, as the molecular weight of the SAN is increased, the strength and rigidity of the ABS increases. As the rubber content increases the impact increases, but the strength, hardness, heat resistance and rigidity of the ABS decrease. This family of materials can be divided into injection molding grades and extrusion grades. In turn, each of these two major divisions can be sub-divided into medium, high and very high impact grades. There are also other grades such as high heat, plating and flame retardant grades.

In general, ABS is a hard, tough material with good resistance to impact, even at low temperatures. It has low water absorption and is a good electrical insulator. The electrical properties are unaffected by changes in humidity. It is usually available in opaque colors, although translucent material 151

52 and colors are available. The moldings can have a high gloss, are dimensionally stable and give good reproduction of the mold surface. The molded surface is resistant to scuffing, but the material has poor weathering properties. It has superior heat resistance and impact strength compared to TPS. It has a higher flexural modulus than PP, which often permits lower wall thickness, and, as it is an amorphous material, it is faster cycling. It is not as notch sensitive as PC and PA.

The highest gloss levels are achieved with moderate melt temperatures, high mold temperatures, fast filling speeds and moderate packing pressure. For electroplating applications, it is necessary to produce injection moldings with low levels of internal stress. For a given material, the levels of residual stress are governed by the molding conditions employed. In general, during injection molding, the lowest levels of residual (internal) stress are produced by using high melt temperatures, high mold temperatures and slow cooling rates. If relatively small gates are employed, the use of fast injection speeds may result in low levels of internal stress. If large gates are employed, the use of slow injection speeds is usually recommended. Melt and mold temperatures should be as high as possible and the packing pressure moderate. The use of such conditions will also minimize warping.

By blending ABS with other plastics, it is possible to extend the range of use. For example, the use of PC or SMA improves the heat distortion temperature, while the use of PVC improves flame retardancy. These alloys have higher viscosities than standard ABS. Typically the ratio of ABS:PVC is 80:20. By blending ABS with chlorinated polyethylene (CPE), a type of thermoplastic elastomer (TPE) may be produced, which has improved tear and abrasion resistance (compared to the unmodified material). The addition of approximately 10% of a liquid polybutylene (polybutenes are viscous, non-drying liquids) to an ABS, can double its impact strength at both room temperatures and -40EC/-40EF. This type of blend can have melt viscosities and heat deflection temperatures similar to the unmodified material. There may be a modest decrease, however, in tensile and flexural properties.

If, during polymerization, part or all of the styrene is replaced by  $\alpha$ -methylstyrene (AMS), a high heat grade, with increased heat resistance, is produced. Alloys with styrene-maleic anhydride (SMA) also give high heat grades that have a lower melt viscosity at a reasonable cost. Clear ABS grades may be made by using methyl methacrylate (MMA) as a fourth monomer and/or, by blending ABS with PMMA; this improves the transparency, as it helps to match the refractive index of the other materials. When low levels of polymerization emulsifier are used during manufacture, grades are produced that offer up to approximately 80% light transmission and a haze level of 10%. Other properties are similar to those of medium impact, standard ABS materials.

By the incorporation of CPE into SAN, ABS-type materials known as ACS result. These have better flame retardancy, heat resistance, weatherability and resistance to dust deposition than ABS, but they have poorer processing stability. Olefin modified SAN results from the incorporation of olefin elastomers into SAN. They have properties similar to ABS, but have better weathering properties.

Styrene plastics are not naturally flame resistant although their flame resistance can be improved by the use of flame retardant additives, such as a bromine compound and antimony trihydrate (ATH). Octabromo diphenyl oxide (OBDPO) is often suggested for use in ABS, although high levels may be required (for example, 15 parts per hundred of resin (phr) of bromine may be needed). The bromine content of ABS compounds needs to be approximately 50% higher than those based on HIPS. Compounds based

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on 1,2-bis-(2,4,6-tribromo phenoxy) ethane (TBPE) are preferred where reasonable light stability is required, although mixing/dispersion is often poor and the FR compound has relatively poor thermal stability compared to OBDPO. Because of the level of use, and the relatively stiff flow behavior of ABS, FR additives that ease the flow are to be preferred. ABS/PVC blends are flame retardant, but the PVC reduces the processing stability relative to ABS.

#### 7. Flow Properties

ABS materials usually have high viscosities compared to PS. The Vicat softening point of ABS is approximately 103°C/218°F. However, this depends on type of ABS: the higher the impact strength, the lower the Vicat softening point and the stiffer the flow. In general, extrusion grades have much higher viscosities than injection molding grades. Extrusion grades have mult flow index (MFI), or melt flow rate values (MFR), measured at 200°C/392°F and 21.6 kg from 3 to 14 (if measured at 220°C/428°F and 10 kg the range is from 1 to 8). The most viscous grades are for applications where toughness is necessary, such as drainage pipes. For producing sheet for refrigerators, a grade with intermediate MFR is used. The following table shows how viscosity varies for a relatively viscous grade. However, it should be noted that because of the great range of options in the chemical structure of an ABS compound, the range of viscosity values is wide. For example, over the same range of conditions, a heat resistant grade may have viscosities from 8600 to 700 Ns/m<sup>2</sup>.

Shear Rate (s-1)		Viscosity (Pa-s)	
	(420°F/216°C)	(450°F/232°C)	(480°F/249°C)
20	3450	2570	1880
40	2440	1810	1324
100	1540	1140	840
200	1080	810	590
400	770	570	420

#### 8. Resistant to:

ABS is more resistant to organic chemicals (such as carbon tetrachloride) than polystyrene (PS). The chemical resistance of ABS depends upon the styrene-acrylonitrile (SAN) continuous phase. Increasing the level of SAN, increases the level of chemical resistance.

ABS is resistant to staining and to water, alkalis (up to  $66^{\circ}C/150^{\circ}F$ ), acids (not concentrated oxidizing acids), inorganic salt solutions (acid salts, alkaline salts and neutral salt solutions), oils and fats. It is also resistant to the majority of alcohols and hydrocarbons, although some may mar the surface. Concentrated phosphoric and hydrochloric acid have little effect. Most products have good environmental stress cracking (ESC) resistance. It is resistant to most wet and dry gases, but not to wet sulfur dioxide.

#### 9. Not Resistant to:

In general, this type of material has poor organic solvent resistance. They exhibit, poor resistance to aromatic hydrocarbons (such as benzene, toluene and xylene), esters, ethers, ketones, chlorinated hydrocarbons, methylene chloride, ethylene chloride, trichloroethylene and pdichlorobenzene. In general, ABS is acid resistant, but it is not resistant to 65% nitric acid, perchloric acid and greater than 50% sulfuric acid. It is also not resistant to ammonium hydroxide at elevated temperatures (at 66°C/150°F or higher). It is also lacks resistance to amines and hot alcohols.

Because of the butadiene rubber content, HIPS and ABS are very susceptible to light degradation. This commences at the surface and spreads into inner layers, resulting in a rapid loss of properties, such as impact strength. Surface protection is given by a hindered amine light stabilizer (HALS) such as the comparatively low molecular weight HALS bis-(2,2,6,6,tetramethyl-4-piperidyl) sebacate. A UV absorber of the 2-hydroxyphenyl ben**154** zotriazole class protects the deeper layers. Mixed stabilizer systems are often much more efficient than a single stabilizer.

For most applications an antioxidant is required for ABS, as the rubber component of the material is subject to oxidation during drying and during melt processing. Antioxidants used include butylated hydroxytoluene (BHT), octadecyl-3-(3,5-di-t-butyl-4-hydroxy-phenyl)-propionate, 2,2'-methyl-ene-bis-(4-methyl-6-t-butyl-phenol), 2,2'methylene-bis-(4-ethyl-6-t-butyl-phenol), 2,2'-methylene-bis-(4-methyl-6-t-butyl-phenol). These are sterically-hindered phenols which act as chain breaking antioxidants. A preventative antioxidant (such as, dilauryl thiodipropionate) may also be used at a total concentration of AO of up to 0.25%.

#### 10. Material Detection or Identification

With a density of 1.07 g/cm<sub>3</sub>, the solid, non-filled material will sink in water and float in saturated magnesium chloride. Shrinkage is of the order of 0.004 to 0.008 in/in or mm/mm, that is, 0.4%-0.8%. The addition of 20% glass fiber will raise the density to approximately 1.20 g/cm<sup>3</sup> but the shrinkage is reduced to 0.2%-0.4%.

A wide color range is possible if the natural base color is ivory or white, depending upon the type of polymerization process used, the ingredients used, etc. The ratio of A:B:S is generally in the range 20:30:50.

ABS begins to melt at approximately 175°C/350°F, and the melt viscosity steadily drops as the temperature is increased. At approximately 290°C/550°F, it begins to decompose via depolymerization and oxidation reactions. When this material is heated in a flame, it burns with a yellow sooty flame and gives off an acrid alkaline odor that also smells of rubber. It burns easily (ordinary grades) and continues to burn when the flame is removed. Burning drops are formed. A common feature of all styrene plastics is their resistance to aqueous media such as salt solutions, acids of medium concentration and alkalis. Aliphatic hydrocarbons, like heptane and cyclohexane, readily attack PS and TPS but do not affect SAN and ABS. Carbon tetrachloride attacks SAN and ABS only slowly, but quickly attacks PS and TPS. ABS may therefore be distinguished from TPS by its better resistance to carbon tetrachloride. TPS has better resistance to methyl alcohol.

#### 11. Coloring

A range of techniques can be used to color ABS. These include dry coloring, master batches and liquid coloring. Dry coloring has always been considered to be the most difficult technique for ABS; however, this is not so. Dry coloring of ABS is a little trickier than with other common materials because the material exhibits several disadvantages (from a coloring point of view). For example; it absorbs moisture, has a variable base color, is opaque and requires fairly high processing temperatures. Nonetheless, by matching the make and grade of ABS with suitable colorants, and by using the correct material drying and colorant mixing procedures (see HIPS or TPS), very good, consistent results are obtained.

The color concentrates (master batches) used for ABS are of the "universal" or of the ABS-based types. A typical let down ratio (concentration) for the "universal" type is 1% to 2% whereas a ratio of between 3% to 7% is used for the ABS-based masterbatch. Before using a 'universal' masterbatch it is important to ascertain it's compatibility with ABS. Pastel shades can now be readily achieved with ABS due to the availability of polymers with a lighter (that is, whiter) base color.

Liquid colors are used for ABS at a concentration level of between 1% to 1.5%. Concentrations above these figures can sometimes lead to problems, including screw slippage and variable output.

#### 12. Materials Handling

These materials are hygroscopic and will absorb 0.2% to 0.35% water in 24 hours at room temperature. This moisture content does not greatly affect the mechanical properties, but if it is above 0.03% the surface finish of extrudates is seriously affected. Therefore, if the material has absorbed more than 0.03% moisture content, it should be dried before use (in a hot air oven for 2 to 4 hrs at 80°C to 85°C/175°F to 185°F) or in a desiccant drier for 1 to 2 hrs at 80°C/175°F. Strict control of material storage (that is, FI-FO) can help eliminate extensive drying operations. Pre-warming the material may also improve processability.

Dry PC/ABS blends at 100°C/212°F. Reground material will tend to absorb moisture more quickly than virgin and therefore should always be dried before use. Keep the regrind level constant but below 30%.

The thermal stability of ABS is not as good as PS. At high melt temperatures, fumes can be a problem. Ensure that adequate fume extraction or removal is available over the die or the vent on a vented barrel machine. Gravity feed hoppers are adequate, but should be covered to reduce the moisture uptake. Material should not remain on the top layer of the hopper for more than an hour or so. If this is likely to be the case, use either vacuum hoppers or purge the top airspace with a dry gas such as nitrogen. Heated hoppers will also reduce the risk of moisture uptake.

#### 13. Screw and Barrel Design

If single stage screws are used, the barrel should have an L/D ratio of at least 24:1 and a compression ratio of between 2.7 and 3:1. Two stage screws are preferred and a vented barrel. Vacuum venting of the barrel is recommended.

In the following table typical values are given for a 30:1, two stage screw. All dimensions given are in inches and the figures in brackets refer to zone lengths. In this design, the pump ratio (that is, the ratio of the depth of the second stage metering zone to the first stage metering zone) is 1.75. The pump ratio measures approximately the relative conveying capacity of the second stage compared to the first stage. If it is below about 1.5 then flooding of the vent can occur, while if it is above 2 then surging of the output is possible.

Diameter (D in inches)	2.5	3.5	4.5
1st feed depth (6 D)	0.43	0.55	0.60
Compression (6 D)			
1st metering depth (6 D)	0.16	0.20	0.23
Decompression (1 D)			
Vent zone depth (4 D)	0.53	0.68	0.75
Compression (2 D)			
2nd metering depth (5 D)	0.27	0.35	0.40

#### 14. Barrel and Die Temperatures

When manufacturing pipes and profiles, the melt temperatures used are between 230°C to 245°C/445°F to 465°F. For sheet manufacture, the range is from 220°C to 250°C/430°F to 475°F. The table below shows typical temperature settings used to achieve these melt temperature. Note that in sheet manufacture, often the temperatures at the outside of the die are set higher than those inside to encourage flow to the edges of the sheet.

Temperatures	°F	°C
Zone 1	400	205
Zone 2	425	220
Zone 3	440	230
Adapter and die	460	240

Please note that it is the melt temperature that is important. The temperatures given in the table are only suggested, initial settings. Check all temperatures regularly. Accurate temperature control is essential to prevent overheating of the polymer within the die. This is particularly important when processing colored compounds, as shade variation will occur as a result of the overheating; particularly for flame retardant grades of ABS (FR-ABS).

Maximum barrel residence time is dependent upon temperature. For example, at a temperature of 265°C/509°F the maximum residence time in the barrel should not exceed 5 to 6 minutes. When the temperature is 280°C/536°F then the maximum residence time in the barrel should not exceed 2 to 3 minutes. Flame retardant grades can present a degradation problem in the event of a stoppage. If any unforeseen delays occur, then reduce the barrel temperatures to 120°C/248°F and before recommencing production, purge with a standard grade of ABS. Variations in the barrel residence time may cause color changes which may only develop on storage. Some ABS products may appear satisfactory when produced, but may develop brown or yellow streaks on storage. This is thought to be due to the cylinder residence time being too long or to be due to over-heating.

#### 15. Die Design and Construction

Since ABS materials show a tendency to degrade, low melt volume in the die and smooth flow paths are required, as in PVC dies. For sheet production, streamlined coat-hanger dies, operated at pressures between 1500 and 3500 psi, are recommended. All internal surfaces in contact with the melt should be chrome plated. In general, dies with both choker bar and flex-lip adjustment are the best. Heaters should be located in zones, so the sides of the die can be set at a temperature different from that of the center. Die gap is set about the same as the sheet thickness, as there is little draw down which minimizes reversion. Even so, some orientation does occur in the machine direction. The land length should be at least 10 times the sheet thickness and is usually more for sheet below about 0.125 in. Deckels as a means of reducing sheet width are not recommended, as ABS will degrade in the stagnant area behind them. Deckels also disturb the designed flow pattern and lead to thick edges on the sheet. These edges may prevent the center of the sheet from correctly contacting the polishing rolls.

If there is sufficient flexibility in the die, then, for the production of sheet, with a thickness less than the die gap, the edges should be set to give thin sheet that can be trimmed and reused.

#### 16. Downstream Operations

In pipe production, good results are obtained by using vacuum sizing equipment. Pipes should have a temperature of no more than  $75^{\circ}C/170^{\circ}F$  after leaving the cooling bath. If the melt temperature used to produce the pipe is 240°C/464°F, then the cooling bath must be capable of removing 338 Joules (J) of heat for each gram produced. In the temperature range quoted, the specific heat of an unmodified grade of ABS is approximately 2050 J/kgK.

For sheet production, the material is fed from the die horizontally into the nip of a three-roll polishing stack. These rolls are chrome plated, and should have a surface finish of 3 to 6 microns to achieve a good surface finish on the sheet. The diameter of the rolls must be large enough to remove the heat from the sheet. Thus, larger rolls are required as the output rate increases and as the thickness of the sheet increases. As an approximation, the following formulae can be used to estimate roll diameter (inches) from line speed (feet per minute).

- For 0.1 in (100 mil) sheet, roll diameter = 2 x line speed
- For 200 mil sheet, roll diameter = 32 x line speed
- For 300 mil sheet, roll diameter = 5 x line speed

The roll stack can be a downstack (where the sheet travels down) or an up stack. In downstack operations, for good high gloss finish, the sheet surface temperature should be between  $154^{\circ}$ C to  $177^{\circ}$ C/310°F to  $350^{\circ}$ F as it enters the second nip and between  $99^{\circ}$ C to  $121^{\circ}$ C/210°F to  $250^{\circ}$ F when it leaves the bottom roll. Upstacks are often used for thick sheet and when embossing, with the embossing roll in the middle. Temperatures for the rolls are shown in the table in degrees Fahrenheit (°F).

Roll	Downstack	Upstack (for Embossing)
Top roll	175 to 225	180 to 220
Middle roll	150 to 175	200 to 250
Bottom roll	175 to 225	150 to 170

Sheets below about 0.3" in thickness can be shear cut, while thicker sheets are best cut by saw. Stacked sheet is wrapped in PE film to reduce moisture absorption and dirt contamination.

#### 17. Extrusion Capacity

Output depends on the extruder, sheet thickness and cooling system. The table below shows some typical figures for a sheet line.

Extruder Diameter	Output Ib/hr					
	(Sheet <0.05 in)	(0.3 in sheet)				
2.5 in	220 to 440	140 to 280				
3.5 in	480 to 880	310 to 570				
4.5 in	900 to 1600	580 to 1000				
6 in	1800 to 3500	1200 to 2400				

#### 18. Starting Up

ABS presents no special problems. If the line has not been purged but contains ABS, do not leave it at operating temperatures for more than about 20 minutes before starting to extrude, slowly. A low initial screw speed is especially important if static mixing devices are present. Check pressures, motor current and extrudate temperature.

#### 19. Shutting Down

Fire retardant (FR) grades should be purged first with a standard ABS grade. If temperatures are dropped at once, then ABS can be left in the extruder and die. However, if the equipment is to be left at temperature for any length of time, purge with GPPS. Purging with GPPS is also recommended before cleaning. Immerse purged resin into cold water to stop fuming. There are no problems in changing from one grade to another. When changing to ABS from another material, purge first with PS, PMMA, or PE.

#### 20. Reprocessing

When ABS is reclaimed, it is suggested that no more than 30% regrind should be blended with the virgin material. Care should also be taken when adding regrind, as the Vicat softening point (VSP) and impact properties may be lowered, depending upon the percentage added. Heavy-duty grinders are required to prepare ABS regrind. Take care not to introduce any incompatible material, as this will result in delamination and brittleness in the extruded component. Regrind must be dried.

If a flame-retardant (FR) grade is being reprocessed then it must be confirmed that the FR additive can withstand the extra processing involved without degradation and/or loss of flame retardancy.

#### 21. Finishing

ABS can be welded by hot air, heated tool, friction and ultrasonic methods. Methyl ethyl ketone and methylene chloride may be used to solvent weld this type of material. Products may be decorated by printing, embossing, or metallizing, after suitable surface treatments, deglazing for electroplating and static **158** removal for printing. ABS can be readily machined, that is, drilled, milled, turned, and sawn on conventional metalworking machines. The types of cutters used are similar to those used for non-ferrous alloys (for example, brass and bronze). Because of its poor thermal conductivity, it is important to cool, with air or liquid, during the machining operation in order to achieve the desired surface finish and/or accuracy. Extrudates should be carefully handled and/or wrapped as they scratch easily.

#### 22. Other Comments

Transparent grades are usually supplied tinted, as in their natural state a slight milkiness is apparent. Glass reinforced grades are available for extrusion.

#### 23. Typical Products

ABS has useful properties from -40°C to 100°C/-40°F to 212°F. It has a high notched impact strength (can reach 11.5 ft lbs per inch of notch), a heat distortion temperature that can be 100°C/212°F, good stiffness, excellent processability, a high gloss appearance and moderate cost. The properties of ABS can be tailored to emphasize a specific property (like heat resistance or impact strength) to satisfy specific customer requirements. ABS also has good dimensional stability and low creep.

ABS is widely used for pipe and pipe fittings, as these may be readily joined, one to the other, and yet are chemically resistant. The excellent surface finish and toughness of ABS are an advantage. It is used for profile because its excellent surface finish and toughness are advantages in this application. The major product is extruded sheet, which is thermoformed into refrigerator housings, caravan roofs, small boats and other smaller less glamorous items. The appliance market uses ABS for power tools, vacuum cleaners, fans and kitchen appliances. A major growth area is in the business machine and consumer electronics sector, where this material is used in computer housings, word processors and copying machines. Laser-markable grades of ABS are now available which are cheaper than polybutylene terephthalate (PBT) and are more versatile. With PBT only dark colors on a light background can be generated, while with ABS light marking on a dark background is possible. Components which give electromagnetic shielding (EMI), may be made by the incorporation of conductive materials such as carbon fiber, graphite fiber or aluminum flake into the base material.

PC/ABS blends are used where the heat resistance of ABS is not good enough. Transparent grades of ABS offer direct competition to PC where moderate impact properties and clarity are required. ABS/PA 6 blends are used where the fatigue and chemical resistance of ABS is not good enough. The impact resistance of the blend is approximately twice the sum of that for ABS and PA combined. ABS contributes stiffness and good appearance. PA contributes fatigue and chemical resistance.

#### 24. Yield

The following assume a density of 1.05 g/cm<sup>3</sup>

- Sheet of 0.1 in thickness has a surface area of 1.83 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.48 sgm/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area 91.7 sq ft/lb weight
- Film of 50 microns thickness has a surface area of 19.0 sgm²/kg
- Tube of 3/8 in internal diameter (ID) and a 1/16 in wall thickness has a length of 25.6 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 17.5 m/kg
- Pipe of wall thickness 0.1 in and an ID of 4 in has a length of 1.50/lb weight
- Pipe of wall thickness 2 mm and an ID of 100 mm has a length of 1.32  $\,$  m/kg  $\,$

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### ≻ HIPS

#### 1. Common Name

#### 2. Abbreviation(s)

#### High-impact polystyrene HIPS: PS-HI: TPS: IPS

The abbreviation SB (standing for styrene modified by an elastomer based on butadiene) is sometimes used but it is not recommended as it can lead to confusion with other styrene/butadiene plastics such as styrene butadiene block copolymers.

#### 3. Alternative Names

Toughened polystyrene (TPS): rubber modified polystyrene: rubber toughened polystyrene: impact polystyrene (IPS).

#### 4. Some Suppliers

Atochem Bamberger Polymers B.A.S.F. CdF. Chemie Chevron Chemical Dart Dongbu Petrochemicals Dow Carbide Fina Chemicals

Huls Huntsman Chemicals Idemitsu Petrochemical Lati LG Chemical Mobil Chemical Monsanto EniChem Neste Nova Chemicals Shell Shuman Plastics Spartech Sterling Thermofil

#### 5. Trade Names or Trade Marks

Lacorene Bapolan HIPS Polystyrol, Avantra Gedex Gulf HIPS Dart HIPS Solarene Aim. Stvron HIPS Terblends (TIPS based on Finaclear (SB) and Finaprene (radial SBS) Vestvron Huntsman PS Polvstar Lastirol LG HIPS Mobil HIPS Lustrex Fdistir Neste PS Nova Carinex Shuman HIPS SC2 Sternite Thermofil HIPS

#### 6. Material Properties

HIPS is traditionally made by polymerizing styrene in the presence of butadiene rubber. A polybutadiene rubber (BR) is dissolved in styrene, and on polymerization of the styrene (caused by heat and catalysts), the rubber comes out of solution to form fine rubber particles suspended in the plastic phase. These rubber particles are also chemically grafted, or bonded, to the polystyrene matrix. This grafting gives a material with much higher impact strength than PS, but with lower tensile strength, stiffness, hardness and Vicat softening point. The transparency and surface gloss of PS are also lost by the incorporation of the rubber. Originally styrene-butadiene rubber (SBR) was used for making HIPS, but was replaced by BR, for technical reasons, in the late sixties and early seventies.

Normally the resistance of HIPS to degradation by light is poor, and the material has lower resistance to chemicals than general-purpose polystyrene (PS or GPPS). The impact strength increases as the rubber content is increased, but the tensile strength, stiffness, hardness and Vicat softening point fall. A very high impact grade may have a density of only 1.02 g/cm<sup>3</sup>, 60 although, in general, the densities of HIPS and PS are similar. High impact grades may be made from high-cis polybutadiene and the material contains approximately 9% rubber. Such high impact grades may also be made from master batches, if required. Medium impact grades ("ordinary" grades) contain 3% to 7% rubber, often based on low-cis polybutadiene, are used as supplied unless master-batched for coloring purposes.

HIPS is not as good an electrical insulator as PS, but is cheap and easily processed. HIPS exhibits a lower water absorption than acrylonitrilebutadiene-styrene (ABS) (the 24 hr absorption of HIPS is approximately 0.05% to 0.08%, whereas for ABS it is about 0.2% to 0.35%). The properties of these two materials overlap in some ways, although, in general, ABS is harder, stronger and more chemically resistant. High gloss, high impact grades of HIPS are now available. The gloss ratings are comparable to, and in some cases better than, ABS. By the use of appropriate additives, the fire and UV resistance of HIPS may be dramatically improved.

Styrene plastics are not naturally flame resistant, although it can be improved by the use of flame retardant (FR) additives. For example, housings for electrical/electronic equipment, appliances and tools are often made from flame retardant high impact polystyrene (FR-HIPS) and flame retardant acrylonitrile-butadiene-styrene (FR-ABS). Flame retardancy can be imparted by bromine compounds (such as decabromo diphenyl oxide (DBDPO) in the case of HIPS and octabromo diphenyl oxide (OBDPO) in the case of ABS) and antimony trihydrate (ATH)). The bromine content of FR-ABS needs to be approximately 50% higher than that of FR-HIPS.

Transparent grades of a type of rubber toughened polystyrene, which is not generally considered as HIPS, may be made by blending polystyrene (PS) with styrene-butadiene-styrene (SBS) block copolymer and/or with styrene-butadiene (SB) block copolymer. This gives a material that may be referred to as transparent impact polystyrene (TIPS) or as, SBS toughened polystyrene (SBS may also be added to high impact polystyrene so as to give a type of super-high impact polystyrene). By combining the three materials, in different ratios, transparency, cost and impact strength can be optimized. Such blended materials may be referred to as terblends. Such terblends can be extruded into flat sheet and also into blown film. The SBS gives elasticity and toughness in the transparent extruded material.

#### 7. Flow Properties

HIPS can be a very easy flowing material. It tends to flow more easily than GPPS. For extrusion use, the ease of flow is assessed by measuring melt flow rate or index (MFR or MFI) at 200°C/392°F and with a weight of 5 kg. The HIPS extrusion grades have MFR values of between 2 and 10. For the production of sheet, the MFR used is typically between 2 to 4, with the lower value being used for sheet for thin-walled vacuum forming.

It is common to blend HIPS with PS to obtain the required balance of toughness, stiffness and price. To do this, one should blend materials that have equal MFR values, as this will give more consistent dispersion. The table below shows viscosity values for a reasonably stiff extrusion grade.

Shear Rate (s <sup>-1</sup> )	Viscosity (Pa-s)		
	(180°C/355°F)	(200°C/390°F)	(220°C/425°F)
20	4490	3260	2400
40	2830	2050	1510
100	1530	1110	820
200	970	700	520
400	610	440	330

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#### 8. Resistant to:

HIPS is resistant to aqueous solutions of salts, acids and alkalis, if strong oxidizing agents are absent. HIPS is more resistant to aqueous chromic acid and silicone oil than PS.

#### 9. Not Resistant to:

HIPS is not resistant to aromatic and chlorinated hydrocarbons, esters, ethers, aldehydes, ketones and various chlorinated hydrocarbons (for example, methylene chloride, ethylene chloride and trichloroethylene).

Because of the type of rubber used (an unsaturated BR), HIPS and ABS are very susceptible to light degradation. This commences at the surface and spreads into inner layers, resulting in a rapid loss of properties such as impact strength. A hindered amine light stabilizer (HALS) may provide surface protection. These include the comparatively low molecular weight HALS, bis-(2,2,6,6,-tetramethyl-4-piperidyl) sebacate. A UV absorber of the 2-hydroxyphenyl benzotriazole class protects the deeper layers. Mixed stabilizer systems are often much more efficient than a single stabilizer.

For most applications an antioxidant is required for HIPS, as the rubber component of the material is liable to oxidation. These include octadecyl-3-(3,5-di-t-butyl-4-hydroxy-phenyl)-propionate, which is a hydroxyphenyl propionate, and a sterically-hindered phenol which acts as a chain breaking antioxidant. A preventative antioxidant, such as dilauryl thiodipropionate, may also be used at a total concentration of the antioxidant, of up to 0.25%.

Although HIPS is resistant to foodstuffs and beverages, some spices and essential oils can cause swelling and/or molding failure. This plastic is not resistant to ground caraway, cedar-wood oil, ground cloves, ground ginger, and has limited resistance to curry.

#### 10. Material Detection or Identification

Clear moldings are not normally possible with this material, unlike PS, which may be seen in a wide range of transparent colors. Its natural shade is off-white, but the material is not often seen in that color, but is usually pigmented. Shrinkage is of the order of 0.4% to 0.7%. The density is approximately 1.05 g/cm<sup>3</sup>, so the material will slowly sink in water. Because of the rubber content (approximately 3% to 10%), the material is softer and easier to cut than general purpose or unmodified PS. It also shows a lower heat distortion temperature (approximately 15°C lower) than PS. HIPS can withstand being flexed without cracking. However, a characteristic feature of HIPS is that, when it is deformed or bent, it will exhibit a stress whitening effect at the point of deformation. It burns easily, forming burning drops, gives off large amounts of sooty smoke and smells of burning rubber as well as styrene.

A common feature of styrene plastics, such as ABS, BDS, PS, HIPS and SAN is their resistance to aqueous media such as salt solutions, acids (of medium concentration) and alkalis. Aliphatic hydrocarbons, such as heptane and cyclohexane, readily attack PS and HIPS, but do not affect SAN and ABS. Carbon tetrachloride (CCl<sub>4</sub>) attacks SAN and ABS only slowly, but quickly attacks PS and HIPS. Resistance to CCl<sub>4</sub> may be used to distinguish between PS and SAN. In the case of polystyrene, when it is immersed in the CCl<sub>4</sub> it immediately becomes sticky, while SAN is relatively unaffected. Alter-natively, if a few drops of CCl<sub>4</sub> are put into a test tube containing either PS or SAN, with PS the liquid becomes milky, whereas with SAN it remains colorless. The flexibility of BDS separates it from PS and SAN. Styrene materials usually have a low density, HIPS has a density of 1.05 g/cm<sup>3</sup>, and a comparatively low maximum use temperature of about 50°C/122°F.

#### 11. Coloring

As the natural color of HIPS is a semi-opaque off-white, a wide color range is possible. It is often seen as white sheet or as white thermoformed sheet. A

162 range of techniques including dry coloring, master batches and liquid coloring can be used to color HIPS. Dry colors are widely used with HIPS. It is customary to add a wetting agent (binder), like liquid paraffin, to stop separation. Generally, 30 ml (approximately a tablespoon) of wetting agent is added per 25 kg of material and, the mixture tumble blended prior to colorant addition. Colorants may be supplied in unit packs, or in bulk. The amount used depends upon the strength of the dyestuff used or the required intensity of the final color. As the color of the base polymer can vary from supplier to supplier, it is preferable, when color matching, to use the actual material that is to be processed. A change in base color can affect the amount, or proportions, of pigments that are to be used. If there is the possibility of water absorption occurring, then dry the material before adding the colorant. However, this is not always possible. To assist dispersion of colorant in the polymer melt, the barrel temperatures at the hopper zone may be set lower than those at the die to allow mixing to take place within the feed section of the screw.

The master batches used for HIPS are of the "universal" or, of styrene-based types. A typical concentration is 1%, that is, 25 g per 25 kg bag of polymer. Higher concentrations may be necessary, depending upon the denseness of the color required in the finished product. Color matching can be carried out using solid master batches. Liquid color is used as an alternative to solid master batches and is well suited when a long run of one color is required.

#### 12. Materials Handling

HIPS exhibits a lower water absorption than ABS, (the 24 hr absorption of HIPS is approximately 0.05% to 0.08% whereas for ABS it is about 0.2% to 0.35%), and normally does not need to be dried before use. However, moisture forming on the granules through condensation will affect surface finish. If the granules are kept in a cold environment, opening the bags in the warm atmosphere of the extrusion shop can cause condensation as a result of the temperature difference. Therefore, during the colder months, and at times of very high humidity, it is advisable to store the day's requirements in the processing plant or at a temperature similar to that of the processing plant. Moisture pick-up can also be a problem when using a bulk-supply material handling system and should be accounted for in the design of the handling system. If it does prove necessary to dry the material, it should be dried in a hot air oven for 3 hrs at 70°C/158°F (desiccant dry for 1 to 2 hrs at 70°C). Antistatic grades may need drying at 70°C for 2 to 3 hrs. The polymer is supplied in pellet form and, for extrusion, the pellets should not be externally lubricated. Gravity feed hoppers are adequate.

Although the level of residual styrene monomer in styrene plastics is comparatively low, the workplace should still be well ventilated and extruder vents fitted with an exhaust system to avoid the risk of inhaling styrene.

#### 13. Screw and Barrel Design

HIPS can be extruded with single stage screws and un-vented barrels, but the best results are obtained using vented extruders with a barrel L/D ratio of about 30/1. If single stage screws are used they should have a compression ratio around 3:1 and an L/D ratio of 24/1 or more and should, preferably, be fitted with a mixing zone. This is especially important when using blends of HIPS and GPPS or when blending in color. The dimensions of a range of vented extruders are given below.

#### Depth

Doptil		Diamotor	
	(2.5 in./63.5 mm)	(3.5 in./89 mm)	(4.5 in./114 mm)
1 <sup>st</sup> feed depth (5D) Compression (5D)	0.40 in./10.2 mm	0.48 in./12.2 mm	0.54 in./13.7 mm
1 <sup>st</sup> metering (5D)	0.13 in./3.3 mm	0.16 in./4.1 mm	0.18 in./4.6 mm

Depth		Diameter	
	(2.5 in./63.5 mm)	(3.5 in./89 mm)	(4.5 in./114 mm)
Decompression (1D)			
Vent zone (4D)	0.54 in./13.7 mm	0.7 in./17.8 mm	0.80 in./20.3 mm
Compression (2D)			
2 <sup>nd</sup> metering (5D)	0.20 in./5.1 mm	0.24 in./6.1 mm	0.27 in./6.9 mm
Mixing zone (3D)			

#### 14. Barrel and Die Temperatures

The target melt temperature range is 180 to 220°C/360 to 430°F. Temperature settings may vary according to material, speed, and screw design. The following values provide a starting point.

Zone on barrel	Temperature	
°C	°F	
1 160	320	
2 180	355	
3 190	375	
4 195	380	
5 190	375	
Adapter and die 200	390	

HIPS normally has good resistance to heat, although it will tend to yellow if left to 'cook' in the barrel.

#### 15. Die Design and Construction

Dies for profile and sheet are similar to those use for ABS. Coextrusion of sheet containing HIPS has been employed. It is possible for the different materials to be joined before entering a standard die, provided their viscosities are not very different from each other. If one melt has a much lower viscosity, then it will tend to migrate to the outside of the flow channel to minimize the work done in flow. In these cases, dies are designed with separate flow channels for each material. Usually they are provided with separate adjustments for the thickness distribution in each layer.

#### 16. Downstream Operations

Three-roll polishing stacks are used for sheet extrusion. They are temperature controlled by circulating water or oil. The flow channels for the fluid should be close to the roll surface and extend over the whole width of the sheet. If a melt temperature of  $240^{\circ}$ C/464°F is used and the material is required to be cooled to room temperature (say,  $20^{\circ}$ C/68°F), then the maximum amount of heat that needs to be removed for each gram of material processed is 433 Joules (J). Over the temperature range quoted, the specific heat is approximately 1970 J/kgK.

The rolls are either driven by a common drive or with individual DC drives which are linked by thyristor based electronics for synchronization. Changing the roll arrangement can control the degree of wrap-round. In vertical stacks there is 360° of wrap-round. With an upstack, where the material enters between the bottom two rolls, the top surface of the sheet can be highly polished and it is possible to laminate on the underside of the sheet. The reverse is the case in the downstack. If the rolls are set at 45° to the vertical, wrap-round can be either 31.5° or 40.5°. In another arrangement, the feed is into the bottom two rolls give the best view of the material as it enters the first nip and the possibility of varying die-to-nip distance without the problem of sag.

The haul-off consists of two rubber-covered rolls driven by a thyristor controlled DC motor. Before stacking, sheet can be edge trimmed

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4 using smooth rotating circular blades for thicknesses up to 0.1in (2.5 mm) and a saw for thicker products. Again, for transverse cutting the choice is between guillotine and travelling saw. Extraction systems must be provided when saws are used. A cyclone separator and filter is used to allow the swarf to be reused. Note that sheet temperature will affect the choice and performance of the cutting and sawing processes.

Lines can be equipped with automatic thickness monitoring devices which transverse the width of the film on C-frames. Output from these devices can be used to control line speed and to perform automatic die adjustment.

#### **17. Extrusion Capacity**

Typical maximum outputs for sheet are shown in the table below.

Extruder Size		Sheet W	Vidth Range Max		cimum Output	
(in)	(mm)	(in)	(mm)	(lb/hr)	(kg/hr)	
2.5	64	12 to 40	305 to 1016	420	191	
3.5	90	30 to 50	762 to 1270	1100	500	
4.5	114	48 to 78	1219 to 1981	1900	864	
6.0	150	70 to 108	1778 to 2743	3000	1364	
8.0	200	95 to 150	2413 to 3810	3000	1364	

Note: The output from a given sheet line is usually below the figures above because of cooling limitations for the sheet. The thinner the sheet, the easier it is to cool and hence outputs should be higher.

#### 18. Starting Up

HIPS presents no special problems, and the procedures given for ABS should be used.

#### 19. Shutting Down

There is no need to purge with another material. In fact, as HIPS is relatively inert, it is often used as a purge for other materials. That is, this material has relatively good heat stability although it will tend to yellow if left to 'cook' in the barrel. There is a risk of decomposition at temperatures above 260°C/500°F. An extruder can be shut down with HIPS inside it, provided the temperatures are reduced to below 149°C/300°F as quickly as possible. For cleaning, it can be advantageous to purge with GPPS.

#### 20. Reprocessing

Large proportions of regrind can be used, however, it is good practice not to use more than 20% in blends with virgin material.

#### 21. Finishing

HIPS can be machined without difficulty, but keep the surfaces being cut well cooled. It may be joined together by welding by ultrasonic welding techniques and by solvents. The strength of the joint, with PS and PS, and of HIPS with HIPS, is greater than that of PS with HIPS. Processes such as screen-printing and dry offset, may be used to print on it. Components may be decorated by hot foiling and painting (but care must be taken to select the correct type of paint, as some solvent based types have a tendency to attack or stress crack the part where the paint has been applied).

#### 22. Other Comments

The use of a saturated rubber, for example, an EPDM rubber in place of polybutadiene can result in significant improvements in the light and oxidation resistance of HIPS. This material is stabilized with an antioxidant, a UV absorber and a hindered amine light stabilizer (HALS). EPDM modified polystyrene has light resistance, that resembles that of ASA but, it is unfortunately, more expensive than traditional HIPS. One could reduce material costs by using a skin of the light resistant material and a core of HIPS.

#### 23. Typical Products

This material is used where impact resistance is required and where chemical

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resistance is not important. As a result of their lower price, the high glosshigh impact grades of HIPS are now being used for products that were previously made from ABS.

Some profiled products are produced, but the major application is for sheet for subsequent thermoforming. A wide variety of cups and small containers for food are made this way, as the rapid production of thin-walled containers is possible. By coextrusion, the product range can be extended. Combinations include HIPS main layer with a PS gloss layer; HIPS main layer with a PS antistatic layer; layers of different cores of HIPS; regrind HIPS as center layer of a sandwich; multilayer construction for margarine tubs, etc. **24. Yield** 

- Sheet of 0.1 ~ thickness has a surface area of 1.83 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.48 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 91.7 sq ft/lb weight
- . Film of 50 microns thickness has a surface area of 19.0 sq m/kg
- Tube of 3/8 in ID and 1/16 ~ wall thickness has a length of 25.6 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 17.6 m/kg

### ≻ PA 6

- 1. Common Name(s)
- 2. Abbreviation(s)

Nylon 6: polyamide 6

PA 6: PA6: PA.6: PA,6: PA6: Ny6

A nylon copolymer may be identified by the use of an oblique stroke between the numbers. For example, PA 66/6.10, indicates a form of nylon based on PA 66 and PA 6.10 (note the use of only *one* PA). Nylon blends may be identified by the use of an oblique stroke between the abbreviations used for each of the PA materials in the blend: PA 66/PA 6.10, indicates a nylon blend based on two nylon polymers (PA 66 and PA 6.10).

#### 3. Alternative Names

It is also referred to as nylon six or as, poly-( $\omega$ -amino-caproamide) or as, poly-(6-aminocaproic acid) or as, poly-( $\omega$ -aminocaproic acid) or as, poly-caproamide or as, polycaprolactam or as, caprolactam PA 6 or as, poly-(imino-1-oxohexamethylene).

#### 4. Some Suppliers

A Schulman Inc. Adell PlasticsInc. Albis Allied-Signal Plastics (ASP) Ashley Polymers Inc. BASE Baver **BIP Plastics BIP Chemicals** ComAllov Compounding Technology Inc. Custom Resins DSM (Dutch State Mines) DSM (Dutch State Mines) DuPont Elf Atochem **FMS-Grilon** FMS EniChem

#### 5. Trade Names or Trade Marks

Schulamid Nylon 6 Adell B Albis Capron Ashlene Nvlon 6 Ultramid B Durethan B Beetle Nylon 6 Jonilon Nylon 6 Comtuf CTI Nvlon 6 Nvlene Akulon Stapron N (an ABS/PA 6 blend) Zvtel 6 Orgamide Grilon Grilon Nivionplast B

### 166 4. Some Suppliers

Ferro Franz Rauscher Goodfellow Japan Synthetic Rubber (JSR) Kanebo I ati LNP Plastics MA Hanna Mitsubishi Engineering Plastics Nvltech Nyltech Polikemi AB Polymer Corp Polymerland Radicinovacips Radicinovacips RhônePoulenc RTP Sniamid Teiiin Thermofil Inc. Ticona Toray Industries Inc. Toray Industries Inc. Tovobo Ube Industries Wellman Inc. **Recycled (R) Materials** Honeywell BASF Nvltech

### 5. Trade Names or Trade Marks

Star-C PA6 Heramid Nylon 6 Capron Kanebo Nvlon Latamid 6 Thermocomp Nvmax Novamid Sniamid Technyl (PA 6/66 copolymer) Polvamid Nylatron Nylon 6 Isomid Radilon Radiflam Technvl C RTP Nvltech Teijin Nylon ESBRID Celanese Nylon 6 Toray Nylon Amilan Nylon 6 Toyobo Nylon Ube Nylon 6 Wellamid Nylon 6

Nypel (PAR) Ultramid RC (PA-R) Oromid (PA 6R)

#### 6. Material Properties

An amide contains the polar amide (CONH) group. A polyamide (PA) contains many (poly), amide (CONH) groups. As the PA material is based on linear aliphatic materials, or monomers, it may be referred to as a linear aliphatic polyamide. Now, commonly a polyamide is referred to as a nylon and the two most widely used nylons are nylon 6 and nylon 66 (PA 6 & PA 66). Both are linear aliphatic polyamides in which the polar amide groups result in strong inter-chain attractions which, in turn, give the materials toughness, rigidity and heat resistance. A high degree of crystallinity is possible, but is not always attained. Processing conditions can significantly alter the degree of crystallinity, and therefore the properties of nylons. Because of the many polar amide (CONH) groups, PA 6 absorbs a large amount of water and this feature must always be remembered when handling this material. The first nylon (PA) moldings were produced in approximately 1941, but PA only became more generally known as an injection molding material in the nineteen fifties. Most PA is produced for fiber applications, even though the nylon family is still a very widely used type of engineering thermoplastics material (ETP).

If a single number follows the material abbreviation for a PA, preparation from either a w-amino acid or a lactam is indicated. PA 6 is prepared from caprolactam by a ring-opening process. The material may be represented by the chemical formula -[-NH(CH<sub>2</sub>)<sub>s</sub>-CO]<sub>n</sub>-. This type of material may be referred to as an AB polymer (it may be visualized as being made from a monomer that contains one acid group and one amine group, which react to

give the amide group). It is a crystalline material that is tough, resilient and creep resistant (to dynamic loads). It has similar physical, chemical and electrical properties to nylon 66 (PA 66) and is classed and handled in the same way, that is, it is treated as a moisture-sensitive material.

PA 6 has a lower melting point than PA 66 and a wider processing temperature range (greater than, 25°C/77°F). It can give superior weld strengths and a better surface finish than PA 66. It is slightly lighter in color, has higher impact strength and slightly better low temperature properties than PA 66. It has better solvent, grease and detergent resistance than PA 66, but its resistance to dilute mineral acids is poorer. PA 6 and PA 66 are reasonably good electrical insulators at low temperatures and under conditions of low humidity.

After processing, PA 6 absorbs water (more than PA 66), which causes swelling and an increase in toughness (the toughness may be doubled). When designing PA moldings, account must be taken of this moisture absorption, as it affects dimensional tolerances and physical properties. The rate, and amount of water absorption depends on wall thickness, relative humidity, and compound composition. Many properties are shown as functions of moisture content.

A wide range of grades is available, as the material may be extensively modified with particulate or fibrous fillers, plasticizers, and other plastics or elastomers. Glass, in one of its many forms, is common filler for polyamides. Glass is relatively cheap and its use can lead to significant improvements in strength and stiffness. The impact resistance of PA may be improved by the use of other plastics or elastomers. For example, the impact resistance when dry may be improved by the use of approximately, 15% PE, providing the two plastics materials are coupled together. High impact PA, results from the successful incorporation of an appropriate elastomer such as EPDM or SBR. To get low temperature impact strength (-40°C/-40°F), it is necessary to use an elastomer which has a very low glass transition temperature and which is partially compatible with the PA. It is possible to get flame-retardant (FR) grades of PA, which are halogen and phosphorus free and are VO rated at 0.25 mm (0.010 in). A well-dispersed silicone fluid can produce a material that has low frictional losses. The use of nucleating agents can give fast cooling grades, as such agents improve the rate of crystallization.

Although a pure aliphatic PA does not absorb short wavelength light, commercial materials are degraded by light, as they contain impurity groups and/or structural features that contribute towards degradation. Heat stabilizers such as phenolic antioxidants, do help with light stabilization, but for the best results, use a HALS-type stabilizer.

A preventative antioxidant, like a phosphite, may be used to retain color during melt processing when used at a level of up to 0.4%. For long term stabilization, use is made of antioxidants such as copper salts (in combination with a halogen compound and/or a phosphorous compound), aromatic amines (a pphenylene diamine) or a sterically hindered phenol antioxidant. Copper salts and amines can cause discoloration, so hindered phenols are often preferred; one can use butylated hydroxytoluene (BHT), 1,3,5-tris-(5-tbutyl-4-hydroxy-2-methylphenyl-butane, 1,3,5-tris-(3,5-di-t-butyl-4-hydroxybenzyl-mesitylene or, N,N'hexamethylene-bis-3-(3,5-di-t-butyl-4-hydroyphenylpropionamide. The level of use may reach 0.7%.

#### 7. Ease of Flow

PA 6 is an easy flow material that is slightly stiffer than PA 66. It does not set up so sharply. At 280°C/536°F) the viscosity ranges from 45 to 300 Pa-s, depending on the grade. The flow of this material can vary if the melt is too wet, too hot, or subjected to a shearing force that is too high. In all these **168** cases the mechanical properties of the molded component will be considerably reduced when compared to a component which has been correctly processed.

There is a range of grades available with differing viscosities. The high viscosity grades are used for rod, tube, profiles and blown film. Medium viscosity grades are used for cast film and thin walled profiles. Low viscosity grades are used for cable sheathing and are often heat stabilized. Typical viscosity values for different grades are as follows at a shear rate of 100 s<sup>-1</sup> and a temperature of 240°C/465°F.

•	High Viscosity Grade	10,000	Pa-s
•	Medium Viscosity Grade	1,500	Pa-s
•	Low Viscosity Grade	200	Pa-s

The table below shows how viscosity depends on temperature and shear rate for a Bayer grade of nylon 6.

Shear Rate (s <sup>-1</sup> )		Viscosity (Pa-s)	
. ,	(230°C/446°F)	(240°C/464°F)	(250°C/482°F)
20	2870	2460	1800
40	2080	1770	1300
100	1350	1150	840
200	980	830	610
400	710	600	440

#### 8. Resistant to:

Both nylon 6 and nylon 66 have superior abrasion resistance and toughness compared to acetal. They are not as tough as polycarbonate (PC), but have better stress crack and solvent resistance. These nylons are fairly good electrical insulators at low temperatures and when the relative humidity is low. Broadly speaking, PA 6 and PA 66 nylons exhibit similar chemical resistance (PA 66 probably has better overall chemical resistance than PA 6). Nylons resist fungal and bacterial attack. They are resistant to hydrolysis and to most common organic solvents. The linear aliphatic polyamides (such as. PA 46. PA 6, PA 66, PA 610, PA 11 and PA 12) are resistant to glycols, aromatic hydrocarbons, aliphatic hydrocarbons, esters, alkyl halides and ketones. They are unaffected by alkalis and many salt solutions. They have good resistance to oils, greases, fuels, fats, and phosphate esters and to refrigerants. Alcohols may cause some swelling and may dissolve some copolymers. Mixed solvents (for example, chloroform and methanol) will attack nylons particularly at high temperatures. At room temperatures, nylons have good resistance to alkalis (in concentrations of up to 20%). In general, nylons have good resistance to gases such as carbon dioxide, carbon monoxide, and to hydrogen sulfide. Both PA 6 and PA 66 have average resistance to radiation when non-reinforced. A light yellowing may occur on irradiation with light on uncolored components. Some grades can be steam sterilized at 121°C/250°F for 30 minutes.

#### 9. Not Resistant to:

Formic acid, glacial acetic acid, concentrated sulfuric acid, dimethyl formamide, phenol and m-cresol are solvents for all PA materials. In general, the resistance of nylons to chlorinated hydrocarbons is only fair. The nylons show poor resistance to mineral acids (dilute and concentrated) and nitric acid in particular will attack them. Nylons may also show poor resistance to solutions of oxidizing agents and to certain salt solutions, such as potassium thiocyanate, calcium chloride and zinc chloride. These salts will cause stress cracking (salt cracking) in PA 6 and PA 66, though PA 11 and PA 12 are resistant.

Salt cracking occurs in polyamides (PA) such as nylon 6 and nylon

66 as the metal ions destroy inter-molecular hydrogen bonding by forming metal complexes with the amide groups. Cracking occurs because of the stress induced by localized swelling and plasticization. Blending nylon 6 with high carbon nylon (PA 12) and/or with a polyolefin, which contains carboxylic groups, improves resistance to salt cracking. Immersion of a stressed (bent) specimen in zinc chloride is used to assess salt cracking resistance. Lithium chloride, calcium chloride and magnesium chloride do not induce cracking in aqueous solutions, but alcohol/salt combinations will attack nylons (for example, methanol and lithium chloride).

In general, nylons have poor resistance to chlorine (wet and dry) and sulfur dioxide (wet and dry).

#### 10. Material Detection or Identification

With a density of 1.12 g/cm<sup>3</sup> (0.65 oz/in<sup>3</sup>) nylon (solid, non-filled material) will sink in water and float in saturated, magnesium chloride. The natural color of the material ranges from translucent white to translucent beige.

On pyrolysis, polyamides give alkaline vapors. When this type of material is heated in a flame, a small blue flame (with a yellow edge) results and white smoke is emitted. The flame is easily extinguished and there may be a smell of burnt hair. PA 6 is a hard, tough material that may easily be cut with a knife producing cuts with smooth edges. The shrinkage of unfilled PA 6 is lower than that of PA 66 and is of the order of 0.01 to 0.015 in/in (or mm/mm) or 1.0% to 1.5%, but is dependent upon processing and filler addition.

The easiest way of differentiating between different types of nylon materials is by means of melting point determinations. PA 6 has a melting point that is approximately 50°C lower than PA 66. The approximate melting points for the more common polyamides are as follows:

PA 12:	175°C/347°F	PA 6:	220°C/428°F
PA 11:	185°C/365°F	PA 66:	265°C/509kF
PA 610:	223°C/434°F	PA 46:	295°C/563°F

PA materials are widely used in blends or alloys with other materials. To analyze for a PA material in such a blend, it may be decomposed by acid hydrolysis (with concentrated hydrochloric acid). The acid on which the PA is based is then collected and identified by analytical means, such as a melting point determination. The approximate melting points for the more common polyamide acids are as follows:

Aminocaproic acid hydrochloride (from PA 6)	123°C/254°F
Sebacic acid (from PA 610)	133°C/272°F
11-aminodecanoic acid (from PA 11)	145°C/293°F
12-aminolauric acid (from PA 12)	163°C/326°F
Adipic acid (from PA 66 and PA 46)	163°C/326°F

#### 11. Coloring

As the natural color of the material ranges from a translucent white to translucent beige, a wide color range is possible. Both translucent and opaque colors are obtained. PA resins are sold in both compounded colors and as natural material for coloring. They may be colored on the injection molding machine by techniques such as dry coloring, masterbatching and liquid coloring.

In-house coloring can be difficult, as some pigments may introduce problems such as leaching, changes in UV resistance (worsened by titanium dioxide), etc. When dry coloring, the choice and type of pigment is important. Inorganic metal oxides are preferred, but some organic blue and green pigments are used. Before making any addition, check with the manufacturer of the material and/or the colorant supplier.

Dry colorant addition is carried out using the multi-axial tumble blending method. The blending drum needs to be two thirds full before the 170 colorant is added and rotation commenced. The material and colorant should be dried before being mixed and it is essential that both ingredients are not exposed to the atmosphere for prolonged periods. Typically, tumble mix times of 5 to 8 minutes are used. The speed of drum rotation depends upon the type of equipment selected. However, the most important factor is to create sufficient mixing/grinding action in the blender to breakdown agglomerates of the pigment (which are present in most colorants) and to disperse the colorant uniformly throughout the blend. The presence of specks, or streaks, and/or variations in shade on molding is a consequence of poor tumble blend-ing.

Master-batches are regularly used, and are often preferred to dry colorants, as their addition is relatively simple and clean. Typical additions of master-batches are between 1% to 2%. Fully compounded material is often preferred to other coloring methods, as color addition may affect the properties of the end product (particularly with components that are used in outside applications). Compounded material is also used for the manufacture of individual components of an assembly that need to be of an exact color and shade.

Liquid colors are not very popular, as the volume of production does not always warrant the costs involved in handling and accurately dispensing the liquid colorant (into the hopper throat or nozzle, on the extruder).

Surface dyeing is possible, but to achieve color consistency and reproducibility, it is essential to critically control the dye concentration relative to the weight of nylon to be dyed (i.e. molding weight). Typical dye concentrations of between 0.002% to 0.02% are used (the actual percentage depends upon the shade and depth of color required).

#### 12. Materials Handling

As supplied by the manufacturer, the material is usually suitable for processing. If containers are left open, rapid water absorption will occur. If the moisture content is >0.2%, flow and component properties are affected and the component will often show signs of foaming. PA should be dried in a wellventilated, hot air oven for 16 hrs at 80°C/176°F. If the material is very wet (exposed to air for more than 8 hours) vacuum, or desiccant drying is necessary (12 hrs at 105°C/221°F).

Both PA 6 and PA 66 will absorb large amounts of water, approximately 10% of water at saturation. However, PA 6 absorbs more water than PA 66, under the same conditions (2.5% as compared to 1.5%). Both materials must, therefore, be carefully stored in a clean dry space. A first-in, firstout (Fi-Fo) storage system must be enforced and the moisture-proof containers should only be opened when required, after they have reached workshop temperature. Keep hoppers and storage containers closed at all times.

It is preferable to use hopper dryers to eliminate any problem of moisture absorption, however, one should always ensure that the temperature of the dryer does not exceed the stated values, otherwise discoloration of the material may result due to overheating and/or oxidation.

#### 13. Screw and Barrel Design

For nylons, barrels with an L/D ratio of at least 20/1 and bimetallic liners are used. Screws are designed with the traditional three zones (feed, compression and metering) with a square pitch. The sizes below are for use with a relatively high molecular weight film resin. For a 24-D screw, the feed zone is 10 D, the compression zone is 6 D and the metering zone is 8 D.

Diameter	2.5"	3.5"	4.5"
Feed channel depth	0.42"	0.51"	0.56"
Metering zone depth	0.11"	0.13"	0.14"
Compression ratio	3.8"	3.9"	4.0"

Experience suggests that good performance can be achieved with a screw design in which there is a continuous decrease in channel depth from under the feed hopper to the screw tip.

Glass reinforced PA grades cause high wear, but this can be minimized by using ion implantation on screws and barrels or, by using screws with Stellite flights.

#### 14. Barrel and Die Temperatures

	Temperatures	
	(°F)	(°C)
Zone 1	410 - 445	210 - 230
Zone 2	445 - 480	230 - 250
Zone 3	465 - 500	240 - 260
Adapter	480 - 520	250 - 270
Die	465 - 500	240 - 260

Please note that it is the melt temperature that is important. The temperatures given in the table are only suggested, initial settings. Check all temperatures regularly. Accurate temperature control is essential to prevent overheating of the polymer within the die and to maintain a uniform polymer viscosity. Too long a barrel residence time will be shown by a change in color of the material (it darkens - particularly with light colors). If unavoidable delays occur, reduce the temperatures to 200°C/392°F.

#### 15. Die Design and Construction

Because of the low viscosity of the molten nylon, and the high temperatures used, it is important to ensure that there is an even temperature distribution in the die. Heating elements and thermocouples should be positioned with care. When using fire retardant (FR) reinforced grades of PA 6 (which can emit corrosive gases, if overheated) consideration should be given to using steels, commonly known as stainless steels, containing 13% or 16% chromium.

**Cable Sheathing:** It is best to use a coating die design in which the low viscosity melt for cable sheathing emerges as a concentric ring from the torpedo and separate from the wire to be coated. This reduces the risk of the low viscosity nylon 6 (PA 6) melt, at high pressures (say 2000 psi), being forced along the cable producing variations in the insulated wire to be coated.

**Wire Coating:** Because of the reasonably tight tolerance in the conductor diameter, coating can take place under pressure inside the die. The radial clearance of the conductor should be about 0.0015 in. The die orifice diameter should be chosen to give a draw down of 10% to 20% on the thickness of the wire covering.

**Blown Film:** Though bottom-fed designs have been used, the preferred type is the spiral mandrel design. The die exit gap should be between 0.02 in and 0.03 in (0.5 mm - 0.8 mm) in width and the land length between 1.2 in and 2 in (30 - 50 mm).

**Flat Film:** Coat-hanger designs are employed with widths up to 80 in (2 m). Die gap widths and land lengths are similar to those used in the blown film dies described above. To achieve the necessary temperature control across the width of the die, it is best to have a large number of narrow band or cartridge heaters that are separately controlled. One heater every 2 in (50 mm) of die width is ideal.

**Monofilament:** The monofilament die is a flat plate, in which are drilled a series of holes that form the filaments. The filaments are extruded downward into a water bath. A gear pump extruder is frequently fitted at the discharge end of the extruder to ensure that a uniform melt flow is obtained. If there is non-uniform flow, fiber breakage or size variations will easily occur with the low viscosity nylon melt.

Tubing: Small bore tubing (where the diameter is less than 0.25 in\ (0.6 mm))

172 can be produced on a crosshead die similar in design to that used for cable sheathing. Air is injected along the central axis of the torpedo to prevent the tube from collapsing as it leaves the die. For larger tubes, an in-line spider type die is employed. The cross-section of the flow channel in the die is reduced after the spider supports (though the taper angle should not exceed 25°). The ratio of channel cross-section at the spider, to that at the land, should be at least 3:1. For tubing with a wall thickness less than 0.12 in (3 mm), the land length should be about 20 times the wall thickness. For greater wall thickness, the land length should be about 25 times the wall thickness. In contrast to the larger dies used to manufacture PVC pipes, the diameter of the torpedo in these dies is often constant throughout their length.

Tubing may be drawn down after the die, typically by an area ratio of 2:1. However, it is possible for small diameter thin-walled tubing, where the wall thickness is only 0.025 in (0.6 mm), for the die gap to be up to 4 times the wall thickness. Above 0.05 in (1.25 mm), the ratio should not exceed 1.25.

#### 16. Downstream Operations

The flat film or chill roll process is commonly used to produce film, as water quenching cannot be used due to its effect on nylons. To achieve good thickness tolerance, the distance between die and chill roll should be no more than 0.5 in (12 mm). The amount of crystallization in the nylon film depends on the rate of cooling. The faster the cooling, the lower the level of crystallinity. By setting the chill roll temperature between 80°C to 90°C/175°F to 195°F. low levels of crystallinity are achieved. This amorphous film is highly transparent, but has a tendency to blocking. It has excellent deep draw properties and is used for the production of vacuum-formed packs. Increasing the roll temperature to 150°C/300°F gives a cloudier, more crystalline, film with a reduced tendency to block. It is tougher and used for the manufacture of heat sealed pouches.

If the roll temperatures are 80°C/76°F and the melt temperature is 250°C/482°F, then the maximum amount of heat that needs to be removed per gram of output is 520 J. Over the temperature range guoted the specific heat is approximately 3060 J/kgK.

At the roll temperatures suggested, there is a tendency for the melt to stick to the roll. This is cured by using release agents or by having rolls with the correct degree of surface roughness. Rolls are coated with chromium, ground, and then treated electrolytically to give a surface with a maximum roughness of 0.0005 in (14 microns).

In the blown film process, the melt is best drawn vertically upwards from the die and cooled by prechilled air directed parallel to the molten extrudate. Iris diaphragms above the cooling ring help to stabilize the bubble. Because of its stiffness, nylon 6 (PA 6) film can crease as it is collapsed from the round to the lay-flat state. To avoid edge creases, it is necessary to make the angle between the collapsing frames as narrow as possible. This will tend to give center drag creases, as PA 6 has a tendency to stick to the collapsing frame surface. To reduce this tendency, use either closely spaced freely rotating rollers or a low friction surface. Perforated aluminum sheet with a matted surface is successful, especially if cooled from the back.

For cable sheathing, vacuum is applied to the rear of the die where the cable enters. This will draw the coating onto the cable as it leaves the die. Both coated cables and covered wires are water cooled after extrusion, with water temperatures that are about 10°C/50°F.

To retain its circular shape, nylon 6 tubing must be supported; that is, sized during solidification. For small bore tubing (less than 0.25 in (6.2 mm) in diameter) simple brass sizing plates may be used. They are positioned 1 in to 2 in (25mm to 50 mm) from the die exit and form the end of the cooling water bath. Vacuum sizing dies are used for tubing between 0.25 in and

0.625 in (6.2 mm and 15.5 mm) diameter (sometimes for larger diameters as well). The vacuum sizing unit is usually separated from the die by a small air gap. For tubing over 0.5 in (12 mm) in diameter, pressure sizing is used. The sizing die length is typically 4 times the tube diameter. The water-cooled sizing die is bolted to the main die, but thermally insulated from it by a gasket. Because of its stiffness, large diameter nylon tubing cannot be rolled and must be cut into lengths. Therefore, a floating bung is used to maintain air pressure for sizing. This prevents a loss of production through distortion, occurring when tubes are cut to length and upstream pressure falls.

Nylon 6 (PA6) extrudates tend to build up large electrostatic charges during production. Passing the extrudate through a discharge station can dissipate these charges. This can consist of:

- A high voltage discharge of about 600 volts AC
- · Radioactively ionized air generated by a polonium 210 isotope
- A point discharge using fine brass wire brushes

#### **17. Extruder Capacity**

The figures below refer to maximum capabilities.

Extrude	r	Output		
(in)	(mm)	(lb/hr)	(kg/hr)	
1.00	25	22	10	
2.00	50	110	45	
3.00	75	600	270	
4.00	100	900	410	
6.00	150	1900	900	

#### 18. Starting Up

Care must be exercised to prevent any gases given off during heating, from becoming trapped, due to un-melted polymer. At the same time, the resin should not be allowed to melt in the section of the extruder next to the hopper. Start heating from the die and do not raise the feed zone temperature above 200°C/390°F, until you are ready to start screw rotation. Also ensure that there is cooling water flowing through the feed throat. Start at low speed and then raise the feed zone temperature to operating settings. Increase screw speed gradually, while keeping an eye on pressure readings. The purge material must be completely removed from the system before extrusion of product. Signs of water in the resin include bubbles in the extrudate, unusually low pressure readings and unstable extrudate.

#### 19. Shutting Down

PA 6 is easily purged with a more viscous polymer. If in doubt, purge with HDPE or PP. Since nylon sticks to the machine surfaces, it is better to purge before cleaning. The residual polymer may be burnt off in an oven at 400°C/750°F. Salt bath cleaning is not recommended due to the danger of explosions.

PA 6 can be extruded immediately after running PA 66. When going from PA6 to PA66, the temperatures should be increased before the PA 66 is introduced.

#### 20. Reprocessing

When this material is reclaimed, up to 20% may be blended into the virgin material without a serious change in the flow or mechanical properties. The reclaimed material must be dried. In general, the amount of regrind used in a product is related to the end-service requirements of the component. Products that will be subjected to high levels of stress in service, are generally produced using a minimum amount of regrind, while other, less critical products are often produced using higher levels. The use of regrind may cause intermittent feeding problems due to bridging in the hopper. If possible, reclaiming should take place immediately after production, as the extrudate

174 will not have absorbed water and is dry. Dry PA 6 is more brittle than nylon that has absorbed water and is, therefore, easier to reclaim.

#### 21. Finishing

Nylon 6 films may be laminated to polyethylene film, aluminum foil or to paper. A dry laminating process is used, in which an adhesive is roll coated to the film, and dried in a heated chamber to remove solvent. The film is then laminated in the nip between two rolls, where heat and pressure ensure a firm bond. A lower cost process is extrusion coating of nylon films with polyethylene. In this case the nylon 6 film is sometimes primed by an electric discharge treatment. Nylon 6 components have a relatively high coefficient of friction when extruded, and films may block. This can be reduced by coating the film with a fine maize flour powder. If components of high heat stability are required, they can be annealed in a non-oxidizing oil for 20 minutes at 150°C/302°F.

PA 6 can be readily machined by processes such as turning, drilling, or milling (using cutting feeds of 0.05 to 0.40 mm/rev (0.002 to 0.016 in/rev)). PA 6 can be decorated by plating, painting, vacuum metallization and surface dyeing, hot foil stamping, silk-screen printing and laser printing. Prior treatment is not necessary for printing on PA 6, and standard inks may be used in the letter-press and flexographic processes. Welding can be carried out using heat impulse, ultrasonic or high frequency methods. PA 6 surfaces can also be bonded together using a two-component epoxy or cyanoacrylate adhesive. To ensure that a more effective bond is achieved, the surfaces should be roughened prior to applying the adhesive. The toughness of extruded products increases as the level of moisture in the nylon increases.

#### 22. Other Comments

When extruded, PA 6 is dry and picks up water as it is stored in the atmosphere. The dry state as extruded can develop large electrostatic charges, and care should be taken to avoid discharge through operators or fire hazard from sparks.

#### 23. Typical Products

Because of its excellent wear resistance, its good mechanical strength, impact resistance, rigidity and lightness in weight, PA 6 is widely used as a light engineering material. Its uses are similar to those of PA 66, so that these materials compete against each other in the marketplace. Nylons are not as tough as PC, but have better stress cracking and solvent resistance. Glass reinforced impact-modified grades of PA 6 are available. These are good electrical insulators that can withstand reasonably high temperatures without distortion. PA 6 is also used to make a range of blends or alloys. For example, ABS/PA 6 blends are used where the fatigue and chemical resistance of ABS is not good enough for the proposed application. Impact resistance of the blend is approximately twice the sum of that for ABS and PA combined. ABS contributes stiffness and good appearance. PA contributes fatigue and chemical resistance.

PA 6 films are used because of their toughness, good gas barrier properties, and ability to withstand fairly high temperatures. The material is often coextruded with polyethylene with an adhesive layer, such as an ionomer, between them. This is required, as nylons do not bond to polyolefins. Small- bore nylon tubing is also used in applications where its good mechanical properties are needed. These properties also make it useful for profiled runners and protective edges.

#### 24. Yield

Since the density of nylon 6 varies with the amount of crystallinity (which depends on production conditions) the yield figures are given as a range assuming a density range from 1.112 to 1.115 g/cm<sup>3</sup>.
- Sheet of 0.1 in thickness has a surface area of 1.68 to 1.72 sg.ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.435 to 0.446 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 83.8 to 86.0 sq ft/lb weight
- Film of 50 microns thickness has a surface area of 17.4 to 17.8 sg m/kg
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 23.4 to 24.0 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 16.0 to 16.5 m/kg

# ≻ PA 66

1. Common Name(s) Nvlon 66: polvamide 66: polvamide 6.6

2. Abbreviation(s)

PA 66: PA66: PA 6.6: PA 6/6: PA,66: PA66: Ny66

A nylon copolymer may be identified by the use of an obligue stroke between the numbers. For example, PA 66/6.10, indicates a nylon copolymer based on PA 66 and PA 6.10 (note the use of only one PA). Nylon blends may be identified by the use of an oblique stroke between the abbreviations used for each of the PA materials in the blend; PA 66/PA 6.10, indicates a nylon blend based on two nylon polymers, PA 66 and PA 6.10 (note the use of two PA abbreviations).

## 3. Alternative Names

Also known as nylon six-six, PA 66 nylon, PA 66 nylon, 66 nylon, poly-(hexamethylene adipamide), polyhexamethyleneadipamide, polyhexamethylene adipamide, or as poly-[imino-(1,6-dioxohexamethylene)-iminohexamethylene].

4. Some Suppliers	5. Trade Names or Trade Marks
Adell Plastics Inc.	Adell A
Albis	Albis
Honeywell	CAPRON
Asahi Chemical	Leona
Ashley Polymers Inc.	Ashlene Nylon 66
BASF	Ultramid A
Bayer	Durethan A
BIP Plastics	Beetle Nylon 66
BIP Chemicals	Jonilon Nylon 66
ComAlloy	Hiloy, Voloy
Compounding Technology Inc.	CTI Nylon 66
Comtec Polymers	Hylon
DSM (Dutch State Mines)	Akulon
DuPont	Minlon, Zytel 66
EMS-Grilon	Grilon T
EniChem	Nivionplast A
Ferro	Staramide 66
Franz Rauscher	Heramid
Goodfellow	Nylon 6/6
Lati	Latamid 66
LNP Plastics	Thermocomp
Mitsubishi Kasei	Novamid
Nyltech	Sniamid
Nyltech	Technyl (PA 6/66 copolymer)
Polikemi AB	Polyamid
Polymer Corp	Nylatron Nylon 66

176	4. Some Suppliers	5. Trade Names or Trade Marks
	Polymerland	AVP
	Polyplastics	Polypla Nylon
	Radicinovacips	Radilon
	RhônePoulenc	Technyl A
	RTP Co	RTP
	Showa-Denko	Tecnil
	Solutia	Vydyne
	Thermofil Inc.	Thermofil Nylon 66
	Ticona	Celanese
	Toray Industries Inc.	Toray Nylon
	Tubize (RhônePoulenc Chimie)	Fabelnyl
	Ube Industries	Ube Nylon 66
	Wellman Inc.	Wellamid Nylon 66
	Recycled (R) materials	-
	Honeywell	Nypel (PAR)
	BASF	Ultramid RC (PA-R)
	Nyltech	Oromid (PA 66R)
	6. Material Properties	
	The second secon	

The two most widely used nylons are nylon 6 and nylon 66 (PA 6 & PA 66): see PA 6. Both are linear aliphatic polyamides (PA), in which the polar amide groups form strong inter-chain attractions that give the materials toughness, rigidity, and heat resistance. A high degree of crystallinity is possible. It is not always attained, however, because processing conditions can significantly alter the degree of crystallinity (and therefore the properties) of nylons. As a result of the many polar amide (CONH) groups, PA 66 absorbs a large amount of water. This feature must always be remembered when handling this material.

A polyamide (PA) may be prepared by reacting a diamine with a dibasic acid. The homopolymer, 66 nylon, comes from the reaction of hexamethylene diamine and adipic acid. The polyamide (PA) is identified by the number of carbon atoms in both the diamine and in the acid. Of the two numbers involved, the first comes from the diamine and the second from the acid, to give PA 66. This type of nylon may be referred to as an \* AABB polymer (made from two monomers one of which contains two acid groups and one of which contains two amine groups). It is made from nylon 66 salt, which is prepared by the reaction of hexamethylene diamine and adipic acid. The use of the salt produces high molecular weight polymers, because it gives the exact, equal ratios of the two monomers. The polymer is represented as -[-NH-(CH<sub>2</sub>)<sub>6</sub>-NHCO-(CH<sub>2</sub>)<sub>4</sub>-CO-]<sub>n</sub><sup>-</sup>. By increasing the number of monomers (during polymerization) copolymers may be blended, filled and plasticized, to produce a wide range of grades with a broad range of properties.

PA 66 is a semi-crystalline thermoplastic material that has one of the highest melting points of the commercial polyamides. It has very high strength and stiffness, and retains its stiffness with increasing temperature. After processing, it absorbs water (but not as much as PA 6), which causes swelling and an increase in toughness (the toughness may be doubled). When designing PA 66 components, account must be taken of this moisture absorption as it affects the polymers dimensional tolerances and physical properties. For every 1% water absorbed, the size increases by approximately 0.003 in/in. The rate, and amount of water absorption depends on storage time, wall thickness, relative humidity, and compound composition. Many properties are shown as functions of moisture content. At 50% RH and 23°C/73°F the moisture content is 2.5%.

A wide range of grades is available, as the material may be extensively modified with particulate or fibrous fillers, plasticizers, other plastic materials and elastomers. Glass, in one of its many forms, is a popular filler for PA 66, as it is relatively cheap and its use can lead to significant improvements in strength and stiffness. The impact resistance of PA 66 may be improved by the use of other plastics or elastomers. For example, the impact resistance when dry may be improved by the use of, approximately, 15% PE, providing the two plastics materials are coupled together. High impact PA 66 results from the successful incorporation of an appropriate elastomer such as EPDM or SBR. To get low temperature impact strength (at -40°C/-40°F), it is necessary to use an elastomer that has a very low glass transition temperature and which is partially compatible with the PA. It is also possible to get flameretardant (FR) grades of PA which are halogen and phosphorus free, and are VO rated at 0.25 mm (0.010 in). A well-dispersed silicone fluid can result in a material with low frictional losses. The use of nucleating agents can give fast cycling grades, as such agents improve the rate of crystallization. PA 66 components are tough even in thin sections. Both PA 6 and PA 66, have better abrasion resistance and toughness than acetal. Super-tough PA 66 has good resistance to crack initiation and propagation and is stress concentration resistant.

### 7. Ease of Flow

In general, PA 66 is a low viscosity material and flows very easily. The viscosity is very dependent on shear rate and on temperature. The temperature of the melt must, therefore, be controlled very precisely, as altering the melt temperature by approximately 20°C/50°F alters the viscosity by a factor of two. Nylon 66 is available in a range of viscosities. At 275°C/525°F the viscosity at 100 s<sup>-1</sup> may range from 90 to 4000 Pa-s. The choice of grade depends on application, as can be seen in the following table:

#### Grade

## Typical Application(s)

- Very high viscosity
- Large diameter rodRod, tube, strip, profile and tubular film
- High viscosityMedium viscosity
- Cast film & thin strip

 Low viscosity
Wire covering, cable covering and monofilament (Note: This grade is equivalent to a general purpose molding grade) The variation in viscosity for a medium to high viscosity

grade (Zytel 42) is tabulated below.

Shear rate (s <sup>-1</sup> )		Viscosity (Pa-s)	
	(250°C/500°F)	(250°C/525°F)	(250°C/550°F)
20	5130	3930	3030
40	3430	2640	2040
100	2030	1570	1210
200	1370	1060	810
400	920	710	550

## 8. Resistant to:

PA 66 is resistant to most solvents. It is insoluble in esters, ketones, aromatic hydrocarbons, chlorinated hydrocarbons, alkalis, dilute acid solutions and most organic acids. Some organic solvents, like chlorinated hydrocarbons, cause swelling. It has excellent resistance to oils, fuels, greases and fats.

## 9. Not Resistant to:

PA 66 is not resistant to strong acids or oxidizing agents. Formic acid and phenol are solvents. It is reversibly swollen by water, which worsens the electrical properties. Outdoor exposure can cause color fading and embrittlement, unless the material is stabilized. Although a pure aliphatic PA does not absorb light of short wavelengths, commercial materials are degraded by light, as they contain impurities and/or structural features that contribute to degradation. Heat stabilizers (such as a phenolic antioxidant) do help with light stabilization, but for the best results, use a HALS-type stabilizer.

A preventative antioxidant (for instance, a phosphite) may be used to retain color during melt processing, if used at a level of up to 0.4%. For long term stabilization, use is made of antioxidants, such as copper salts (in combination with a halogen compound and/or a phosphorous compound), aromatic amines (a p-phenylene diamine), or a sterically hindered phenol antioxidant. Copper salts and amines can cause discoloration, so hindered phenols are often preferred; one can use butylated hydroxytoluene (BHT), 1,3,5-tris-(5-t-butyl-4-hydroxy-2-methylphenyl-butane, 1,3,5-tris-(3,5-di-tbutyl-4-hydroxybenzyl-mesitylene, or N,N' hexamethylene-bis-3-(3,5-di-tbutyl-4-hydroxyphenyl-propionamide. The level of use may reach 0.7%.

Salt solutions (such as calcium bromide, zinc chloride, and potassium thiocyanate) will cause stress cracking of PA 6 and PA 66. PA 11 and PA 12 are more resistant, although these materials cannot withstand such high temperatures. Lithium chloride, calcium chloride, and magnesium chloride do not induce cracking in aqueous solutions, but alcohol/salt combinations, like methanol and lithium chloride, will attack nylons. Blending PA 66 with a high carbon nylon (PA 12) and/or a polyolefin, which contains carboxylic groups, will improve its resistance to salt cracking. Mixed solvents (for example, chloroform and methanol) will also attack nylons, particularly at high temperatures.

## 10. Material Detection or Identification

With a density of 1.15 g/cm<sup>3</sup>/0.67 oz.in<sup>3</sup>, PA 66 (solid, non-filled material) will sink in water and float in saturated magnesium chloride. The natural color of the material ranges from translucent white to a translucent light brown.

When this nylon 66 is heated in a flame, it melts, darkens, boils and then burns with a small blue flame (yellow edge). White smoke is emitted and the flame is easily extinguished, emitting a celery-type odor. It is a hard, tough, opaque material that can be cut easily with a knife, producing cuts with smooth edges.

The easiest way of classifying nylons is by melting point (MP) determinations. PA 66 has a high sharp MP of  $256^{\circ}C/490^{\circ}F$  (PA 6  $220^{\circ}C/428^{\circ}F$ ). PA 66 is more resistant to chloroform, benzyl alcohol and trichloroethylene than PA 6. PA 6 will dissolve in 4N hydrochloric acid and boiling N, N-dimethylformamide, while PA 66 will not.

## 11. Coloring

As the natural color of the material ranges from translucent white to translucent light brown, a wide color range is possible. Both transparent and opaque colors are available. The darker the base color, the more difficult it is to obtain the correct color match. PA 66 is sold in both compounded colors and as natural material, for coloring on the extruder by techniques such as dry coloring, masterbatching, and liquid coloring.

Fully compounded material is preferred by many, as color addition may affect properties. When dry coloring, the choice and type of pigment is important. Inorganic metal oxides are preferred; however, some organic blue and green pigments are used. Before making a colorant addition, one should check with the manufacturer of the material and/or the colorant supplier.

Dry colorant addition is carried out using the multi-axial tumble blend method. The drum needs to be two thirds full before the colorant is added and rotation commenced. It may also be necessary to purge the blender with an inert gas first and then fill it with freshly-opened material. The material and colorant should be pre-dried before being mixed and it is essential that both ingredients are not exposed to the atmosphere for prolonged

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periods. After blending, the dry-blend should be used immediately, or stored in a moisture-proof container. Typically, mixing times of 5 to 8 minutes are used. The speed of drum rotation depends upon the type of equipment selected. The most important factor is the creation of sufficient mixing/grinding action in the blender. This is necessary to break down agglomerates of the pigment (which are present in most colorants), and to disperse the colorant uniformly throughout the blend. The presence of specks, streaks, and/or, variation in shade on the products are a consequence of poor tumble blending. Master-batches are regularly used. and are often preferred to dry colorants, as their addition is relatively simple and clean. Typical levels of addition of master batches are between 1% to 2%.

Compounded material is also used where components require exactness of color matching and/or, they are to be used in outdoor applications (particularly with the color black). Liquid colors are not as popular as dry coloring and masterbatching, because the volume of production does not always warrant the costs involved in handling and accurately dispensing the liquid colorant into the hopper throat.

When processing colored PA 66, the major difference, compared to PA 6, is the need to reduce the temperature of the feed zone and hopper throat on the barrel assembly. This should be reduced to the lowest possible setting, to minimize the risk of screw slip. Screw slip may occur due to the combination of the higher melt temperature, used to process PA 66, and the type of carrier incorporated in the masterbatch or liquid colorant.

Surface dyeing of PA 66 is possible. However, to achieve color consistency and reproducibility, it is essential to critically control the dye concentration relative to the weight of nylon to be dyed. Typical dye concentrations of between 0.002% to 0.02% are used (the actual percentage depends upon the shade and depth of color required).

#### 12. Materials Handling

As supplied, nylon 66 is suitable for processing. If, however, containers are left open, rapid water absorption will occur. The material should be dried in a well ventilated, hot air oven for 4 to 5 hrs at 85°C/185°F (be careful not to cause oxidation), or in a desiccant drier for 1 to 2 hrs at 105°C/221°F. If the moisture content is greater than 0.2%, the flow properties of the melt are affected. If the material is very wet (for example, it has been exposed to air for more than 8 hrs), vacuum or desiccant drying is necessary (12 hrs at 105°C/221°F). Ensure that a Fi-Fo storage system is adopted and reseal containers immediately after use. Keep hoppers covered and do not overfill. Dry reinforced grades for 16 hrs at 75°C/167°F.

Mechanical handling and material transfer systems can be used if properly designed and operated. A good design employs a rotating coarse screw inside a flexible tube. The discharge end and motor is fitted over the extruder hopper and the feed end into a tilted raw material container. If the material is supplied in flexible bags, then the system can prevent air coming into contact with the nylon. If vacuum loader conveyors are used, then the pump must be switched off except when material is being transferred in batches. Failure to do this will cause air to pass over the resin and allow it to absorb moisture. A closed loop system, in which the air for conveying the nylon granules is recirculated, is an improvement.

#### 13. Screw and Barrel Design

Because of the high melting point of nylon 66, extruder barrels should be at least 20 diameters in length, or preferably 24 D, and they should have a bimetallic structure to reduce wear. Because of the high temperatures and sharp melting point of nylon 66, good temperature control is essential. It is also advisable to fit a pressure transducer at the end of the barrel to monitor melt pressure.

Traditional screw designs are based on a very short compression section of one turn or less. In recent years, this design has gone out of favor, as it is not very efficient. The table below shows designs for both short compression (SC) screws and those with a longer compression section (a standard (Std) or general purpose, design). Compression ratios are between 3.3 and 3.9. The screw designs given for nylon 6 are also suitable for nylon 66. Barrier screws have also been successful. The figures in brackets refer to zone lengths

Screw Diameter (in.) 1.5			2.5		3.	3.5	
Screw Type	SC	Std	SC	Std	SC	Std	
Channel Depth	(in.)						
Feed zone	0.31	0.30	0.42	0.38	0.58	0.4	
	(15D)	(10D)	(15D)	(8D)	(15D)	(8D)	
Compression	(1D)	(6D)	(1D)	(8D)	(1D)	(8D)	
Metering	0.08	0.09	0.12	0.11	0.16	0.12	
	(8D)	(8D)	(8D)	(8D)	(8D)	(8D)	

#### 14. Barrel and Die Temperatures

-	°F	°C
Zone 1 Temperature	510 - 550	265 - 290
Zone 2 Temperature	525 - 545	275 - 285
Zone 3 Temperature	535 - 555	280 - 290
Adapter	535 - 555	280 - 290
Die	520 - 555	270 ñ 290

The barrel residence time depends on the temperature. At a temperature of 280°C/536°F, the maximum residence time in the barrel should not exceed 5 minutes. When the temperature is 320°C/608°F, however, the maximum residence time in the barrel should not exceed 3 minutes. Melt temperatures above 300°C/572°F, and long residence times (more than 15 minutes), should be avoided, as degradation will cause the material to discolor and become brittle. Material must be purged if overheated.

Ideally, the barrel should be divided into at least 4 zones to improve the accuracy of temperature control. It is common to have a lower temperature at the hopper-end of the machine and a higher temperature at the die-end. The temperatures of the zones in between alter fairly uniformly. This is a normal or uphill temperature profile. However, with some materials, for example, a high melting point nylon such as PA 66, the extruder may be started with an uphill profile and then be switched to a downhill profile once extrusion is safely underway. To do this, the line is started with a conventional temperature profile and with a relatively low feed zone setting. Once pressures have been checked and the line is operational, the feed (zone 1) is set hotter than the metering zone, whose temperature can be reduced (the metering zone is the last zone on the barrel). This setting can minimize degradation and help in maintaining viscosity.

#### 15. Die Design and Construction

The principles of die design for nylon 66 are very similar to those for nylon 6 and the section on PA 6 should be consulted. Because of the higher temperatures used for PA 66, temperature control is more critical. In some instances, it is desirable to fit internal heating elements in the die (such as in the torpedo or mandrel). Pressure monitoring in the adapter is recommended. Chrome plating of the inside of the flow passages in the die is desirable. The following specification is suggested: Chrome - 8 RMS micro-inches, Rockwell hardness C-60 to 65, and thickness 0.002 in - 0.003 in.

For blown film, avoid the use of side fed dies. Use a screen pack consisting of a 120 mesh screen sandwiched by two 80 mesh screens.

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## 16. Downstream Operations

Most downstream operations are similar to those for nylon 6. The heat removal requirements are greater because of the higher temperatures used to process this material. For instance, if the rolls are set at 80°C/76°F and the melt temperature is 280°C/536°F, then the maximum amount of heat that needs to be removed per gram of output is 612 J. Over the temperature range quoted, the specific heat is approximately 3075 J/kgK.

Because of its abrupt transition from melt to solid and large amount of shrinkage, nylon 66 rod is difficult to produce at larger diameters, without voids. Attaching a forming die after the main die can reduce voids. The forming die is cooled and holds the rod under pressure as it cools. Further cooling occurs after the die. Slow cooling is necessary in thick rod production. With rapid cooling, the outer layers of the rod will form a hard skin and as the material in the center shrinks, voids will be produced. Slow cooling provides a more even temperature distribution in the cooling process.

For extrusion of monofilament, a gear pump is inserted between the main extruder and the die. A fine filter pack (one based on sand or sintered aluminum) is also used. The holes in the die plate are 2 to 12 times the diameter of the un-drawn filament, and the molten extrudate is thus cooled under tension. Cooling water is maintained at a temperature of about 40°C/100°F. In-line drawing and annealing takes place using a series of rollers and electrically heated chambers. The monofilament may be wound onto bobbins.

## 17. Extruder Plasticizing Capacity

The figures below refer to maximum capacities. In most cases, the limiting factor in an extrusion process is not the extruder, but another part of the process, such as cooling or take-off capacity.

Extruder		Output		
(in)	(mm)	(lb/hr)	(kg/hr)	
1.00	25.0	20	9	
2.00	50.0	100	40	
3.00	75.0	500	220	
4.00	100.0	800	370	
6.00	150.0	1700	800	

## 18. Starting Up

Start up procedures for nylon 66 are similar to those used for nylon 6. Because of the higher temperatures involved, however, even more care must be taken to avoid trapped gases, premature granule melting and blocking of the feed section of the screw. The symptoms of bridging in the feed are surging, loss of output and a reduction in the motor current. Sometimes, the bridge can be removed by raising the feed zone temperature to 315°C/600°F (or higher) and hand feeding pieces of nylon rod. If this fails, then the extruder must be purged and cleaned.

## 19. Shutting Down

If the extruder temperatures are no more than 290°C/555°F, then the screw can be stopped for up to 10 minutes and restarted. To shut down the machine for longer periods, reduce the screw speed to approximately 10 rpm and then lower the temperature settings to 270°C/515°F. When the extruder has cooled to this temperature, purge with PP or HDPE.

An alternative is to purge initially with polyethylene, of melt flow rate 1.0, until the majority of the nylon has been removed. This should be done without changing temperature settings. Remove the die and the adapter and clean them using soft scrapers based on brass or copper. Remove the last traces of polymer by burning it off in an oven at approximately 800°F- 900°F. It is preferable that heating takes place under vacuum or in an inert atmosphere, 182 to reduce the risk of oxidation of the metal surfaces. After the plastic material has been burned away, a sandblast can be used to remove residual carbon. Note: Dies that have been chrome plated should not be subjected to this high temperature treatment, nor should they be sandblasted.

Polymer can be removed from the screw and barrel by means of an acrylic purge compound. This material will not melt, as its molecular weight is in the millions. It should not be confused with grades of acrylic resin used for extrusion or injection molding. These grades are not suitable for purging. The acrylic purge compound is hand fed into the extruder that is run at a fairly high speed. When all of the polymer has been used, run the extruder until dry, remove the screw and clean the screw and barrel by wiping off the purge material.

### 20. Reprocessing

Reclaim PA immediately after production if possible. A desirable method is to granulate off-spec extrudate within 15 seconds and feed it back immediately. When first produced, PA components are dry, as they have not had the chance to absorb water. The dry material is more brittle than material which has absorbed water and is, therefore, easier to reclaim. Remove fines from the reground material by sizing, using a 16-20 mesh sieve or screen. Material that falls through the sieve should be discarded. Do not re-use overheated (more than 300°C/572°F) material.

If PA material is not immediately regranulated, and re-used at the machine, it is recommended that it be kept clean and dry and then pre-dried before use. Careful drying of regrind is essential. The amount of regrind used is related to the component end service requirements. In general, when this material is reclaimed, up to 25% may be blended into the virgin material. However, the level of regrind possible varies very much from application to application. The use of regrind tends to lower the melt viscosity and hence it is used only sparingly in applications where a high viscosity grade of nylon 66 is required.

## 21. Finishing

Machining should be done at high speeds with sharp tools and low feed rates and should use efficient cooling. PA 66 may be solvent welded with aqueous phenol (12%) or a solution of PA 66 in ethanol and calcium chloride. If dimensions are very critical, then annealing, and/or moisture conditioning, may be necessary. Anneal by heating in a non-oxidizing oil for 20 minutes at 170°C/ 338°F. PA 66 may be conditioned to equilibrium water content by immersion in water (maximum temperature 60°C/140°F). To get uniform water distribution, wrap the part in PE film and store. Painting is also possible, as is chrome plating, after etching first with 40% formic acid. Heat foil at 160°C to 190°C/320°F to 374'F. Nylon parts may be dyed (for example, use ICI's Nylomine) at 90°C/194°F for 15 minutes. Epoxides and cyanoacrylates are good adhesives.

## 22. Other Comments

The use of molybdenum disulfide and graphite, dispersed in the original material, improves the wear of PA 66. Glass filled (GF) grades are available for extrusion. Glass filled rods will be stiffer and less affected by changes in temperature than their unfilled counterparts. The affects of the addition of glass fiber to nylon 66 are shown in the following table. Note, however, that the glass filled grades increase the wear rate of an extruder very considerably.

## Properties of Nylon 66 + Glass Fiber Conditioned to 50% RH

			-
0	15	33	50
60	85	115	155
>100	6	5	6
1.15	3.2	5.5	9.6
	<b>0</b> 60 >100 1.15	0 15 60 85 >100 6 1.15 3.2	0     15     33       60     85     115       >100     6     5       1.15     3.2     5.5

The heat distortion temperature (HDT) also rises as the amount of glass fiber increases. The shrinkage of unfilled PA 66 is of the order of 0.010 to 0.020 in/in or mm/mm (1% to 2%), but the addition of glass fiber (GF) will reduce the shrinkage to 0.2% to 1%. However, differences in shrinkage between the transverse and longitudinal directions (that is, along and across the flow) can be as much as five times. Glass bead filed grades, and mineral filled grades, give more isotropic products.

#### 23. Typical Products

PA 6 and PA 66 are the most widely used of the so-called engineering plastics. Like PA 6, PA 66 is best known in the plastics industry as a light engineering material which is mainly processed by injection molding. Often the material is selected because of the low level of friction encountered, without lubrication, together with good oil and grease resistance. PA 66, like PA 6, is often used where the components require impact resistance and impact strength. They are also widely used as glass reinforced materials. These find use in the automotive industry, as fuel resistant parts that must operate in the aggressive environment in and around the engine. Alloys of PA are now being used for computer housings and for automotive panels. These panels may be required to withstand elevated paint bake oven temperatures and then subsequent low temperatures in use.

Nylon 66 is extruded into rod, strip, profiles, film and monofilaments. Its high melting temperature and inertness make it suitable for 'roast in a bag' applications. Special heat stabilized grades are available for this application. Its high temperature stability also makes it suitable for hot fill and sterilization packaging applications.

With PA 66, shrinkage and after-shrinkage decrease the component size, but moisture absorption increases component size. So, after production, the component may increase in size due to water absorption, which may negate the effect of shrinkage. Nucleating agents help reduce after-shrinkage. **24. Yield** 

- Sheet of 0.1 in thickness has a surface area of 1.69 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.439 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 84.5 sq ft/lb weight
- Film of 50 microns thickness 50 microns has a surface area of 17.5 sq m/kg
- Tube of 3/8 in I.D. and 1/16 in wall thickness has a length of 23.6 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 16.2 m/kg

# ≻ LDPE

- 1. Common Name
- 2. Abbreviation(s)

Low density polyethylene LDPE: PE-LD

#### 3. Alternative Names

Polyethylene - low density (PELE): polythene: low density polythene: poly(methylene): high pressure polyethylene: branched polyethylene.

#### 4. Some Suppliers

Borealis Chemicals Borealis Chemicals BP Chemicals/Amoco Carmel Olefins Chevron Dow-Carbide DSM 5. Trade Names or Trade Marks Neste LDPE Statoil LDPE Novex LD Ipithene Chevron PE Dowlex, DFDA Stamylan LD

# 184 4. Some Suppliers

Eastman Chemical Co. Elanac EquiStar Chemical Co Exxon Mobil Huntsman Polimeri Europa Shuman Plastics Slovnaft Ube Industries Westlake

## 5. Trade Names or Trade Marks

Tenite PE Lupolen, Carlona Petrothene Escorene, LCA Rexene PE Unipol Shuman PE Bralen Ube PE Westlake EF

## 6. Material Properties

Polyethylene (PE) plastics are plastic materials that are based on polymers made mainly with the olefin commonly called ethylene. This usually means no less than 85% ethylene and no less than 95% total olefins. Although a PE homopolymer may be represented as -(CH<sub>2</sub>-CH<sub>2</sub>)<sub>n</sub>-, the simplicity of the formula does not convey the tremendous number of variations (or grades) of PE that are available. Such materials may differ in, for example, molecular weight, molecular weight distribution, short chain branching (SCB), long chain branching (LCB) and the presence of catalyst residues. Polymerization using two or more olefin monomers produces copolymers (olefin copolymers). Because of developments in catalyst technology, copolymers are undergoing a period of very rapid growth. Many commercial PE materials are copolymers that contain a small amount of an alpha olefin, which permits control over SCB and therefore product density. This is important, as the mechanical properties of PE materials are very dependent upon density.

When ethylene is heated at high pressures (up to 3000 bar/43,500 psi) and at temperatures of 100°C to 300°C/212°F to 572°F, in the presence of a free radical initiator, the plastic material originally known as polythene or polyethylene (PE) is produced. It was the first PE ever produced, and is now better known as low-density polyethylene (LDPE or PELD). LDPE is a semicrystalline thermoplastic material whose natural color, in thick sections, is a milky white. It has a soft, wax-like feel. Because chain branching (both long and short chain branching are present) interrupts crystallization of the polymer chains, the crystallinity level is low. This means that if the extrudate is kept thin and cooled quickly, then film can appear transparent. The material generally has a relatively low density (typically 0.92 g/cm³), but by varying the polymerization conditions, it is possible to produce commercial materials with densities over the range 0.915 to 0.94 g/cm³.

LDPE is a low-cost material with excellent chemical resistance. It is tough, even at low temperatures, has excellent electrical insulation properties, and is capable of being processed by a wide range of techniques. However, LDPE only has moderate tensile strength, low stiffness, low maximum use temperature, and suffers from creep. Its resistance to light and fire is poor. LDPE has high water vapor and gas (particularly carbon dioxide) permeability. Permeability to organic vapors is lowest for alcohols and increases in the order: acids to aldehydes and ketones, esters, ethers, hydrocarbons and halogenated hydrocarbons (permeability decreases with density). LDPE is also susceptible to environmental stress cracking (ESC).

By copolymerization of ethylene with a higher alpha olefin (HAO), it is possible to produce a range of very low-density materials. Very low-density polyethylene (VLDPE or PEVLD) may also be known as ultra low-density polyethylene (ULDPE or PEULD). Sometimes VLDPE is distinguished from ULDPE on the grounds of density. VLDPE may be considered to be a polyethylene (PE) which has a density of 0.915 to 0.90 g/cm<sup>3</sup> and ULDPE is a PE which has a density below 0.9 g/cm<sup>3</sup> (say 0.88 g/cm<sup>3</sup>). Such very low-density HAO grades, which are hardly crystalline, are rubber-like materials that can be used as an alternative to thermoplastic elastomers and for materials modification (for example, improving the impact strength of PP).

#### 7. Ease of Flow

Both long chain branching (LCB) and short chain branching (SCB) are present in LDPE and, because LCB produces compact molecules, the material flows relatively easily. The ease of flow is rated by what is known as the melt flow index (MFI) or rate (MFR). Both terms refer to the same test. The lower the number, the stiffer the flow since the molecular weight is, generally, higher. At 200°C/392°F a plastic material with an MFR of 20 will have approximately twice the spiral flow length of a plastic with an MFR of 2. Low MFR materials exhibit better environmental stress cracking resistance (ESC), solvent resistance, and higher impact strength. Polymers of different density, but with the same melt flow index, do not necessarily have the same molecular weight.

LDPE is more pseudoplastic (shear thinning) than LLDPE because it has a broader molecular weight distribution. If LDPE of a given melt flow rate (index) is compared with LLDPE material of the same melt flow index, it will be found that the LLDPE material has a higher melt viscosity at processing shear rates.

For blown film production, the MFR ranges from 0.2 to 3.0. The low MFR grades are used for heavy-duty sacks, while the high MFR grades are used for packaging applications where good gloss is required. For extrusion coating less viscous grades, with MFR in the range 4 to 12, are employed. In cable coating, the MFR is generally about 0.2.

The following table shows viscosity values for a MFR 2 film grade resin over a range of shear rates and temperatures.

Shear Rate (s <sup>-1</sup> )	Viscosity (Ns/m²)				
	(171°C/340°F)	(190°C/375°F)	(210°C/410°F)		
20	2480	1910	1460		
40	2190	1330	1040		
100	1030	810	640		
200	660	540	440		
400	430	350	290		

#### 8. Resistant to:

LDPE is resistant to most solvents at room temperature; however, aromatic and chlorinated hydrocarbons will cause swelling. It is relatively unaffected by polar solvents, (alcohols, phenols, esters and ketones); vegetable oils, water, alkalis, most concentrated acids (at room temperature), and ozone (in absence of UV). It has very low water absorption, even after long immersion times (<0.2%. after one year at 20°C/68°F). The addition of carbon black, used to improve weathering, will increase the water absorption. Absorption of other liquids, such as acetone and benzene, will be greater for LDPE than for HDPE. The best chemical resistance is found with HDPE and cross-linked PE.

Broadly speaking, PE materials exhibit similar chemical resistance in the long term, although, in the short term, LDPE will be attacked more quickly than the higher density materials. Overall, HDPE or LLDPE has the best chemical resistance. HDPE resists aromatic and chlorinated hydrocarbons more than LDPE. In general, PE materials resist fungal and bacterial attack. They are resistant to hydrolysis and attack by most common organic solvents at room temperature. They are insoluble in organic solvents at temperatures below 50°C/122°F. At higher temperatures, they are soluble in hydrocarbons and halogenated hydrocarbons. They can be dissolved in hot solvents, such as toluene, xylene, amyl acetate, trichloroethylene, petroleum ether, paraffin, turpentine and lubricating oils. Although there is no solvent for PE at room tem50 perature, they swell in chlorinated hydrocarbons, aliphatic hydrocarbons, aromatic hydrocarbons, some esters and oils.

Polyolefin materials are resistant to acids and to alkalies, but are not to oxidizing acids, such as nitric acid, chlorosulfonic acid, chromic acid, and fuming sulfuric acid. They resist acid, basic and neutral salts. The materials have good resistance to gases such as carbon dioxide, carbon monoxide, hydrogen sulphide and sulfur dioxide (wet and dry), but have poor resistance to chlorine (wet and dry). The water vapor permeability of all types of PE is low. PE materials are permeable to gases and vapors, but LLDPE and HDPE are less permeable to gases and vapors than LDPE. Permeability for organic vapors is least for alcohols and then increases from acids to aldehydes and ketones, to esters, ethers, hydrocarbons and halogenated hydrocarbons (permeability decreases with density). Some high molecular weight grades of HDPE and LLDPE are accepted as being suitable for containers for oil and petroleum based products. They are used for fuel tanks. In some cases the formed containers are chemically modified, by fluorination or sulfonation, which makes them almost impermeable to fuels.

#### 9. Not resistant to:

Polyethylenes are not resistant to fuming nitric acid or fuming sulfuric acid. Halogens and chlorinating agents such as chlorosulfonic acid and phosgene slowly attack them. LDPE is sensitive to environmental stress cracking (ESC), which occurs when a material is subject to strain, or an internal stress, in the presence of polar liquids or the vapors of such liquids. Such ESC is also associated with detergents or silicone fluids, although there are many other environmental stress-cracking agents, such as chloroform, xylene and paraffin. The ESC effect is minimized by reducing the residual stresses in the molding, careful component design, and by using the lowest MFR (the highest molecular weight) grade. At any particular density level, LLDPE has better ESC resistance than LDPE and the higher molecular weight grades have the best resistance.

In many cases the resistance of PO materials to light is satisfactory, but, if it is not, the cheapest way of improving the light resistance is by the addition of carbon black. Ultraviolet (UV) absorbers and screening agents are also used to give protection. Unless so protected, appearance will initially suffer, and then the material will seriously degrade. The most commonly used non-black light stabilizers are those based on ultraviolet absorbers, such as 2hydroxy-4-alkoxybenzophenones and 2-(2'hydroxyphenyl) benzotriazoles), nickel (11) chelates, and polymeric hindered amine light stabilizers (HALS). Mixed stabilizer systems are often much more efficient than a single stabilizer.

As PO materials have comparatively limited resistance to oxygen at elevated temperatures, antioxidants are also used for their protection. Unless they are protected, the electrical properties will suffer. LDPE has limited resistance to oxygen at elevated temperatures, but not as susceptible to oxidation at high temperatures (for example, those experienced during melt processing) as polypropylene (PP). The antioxidants used are similar to those used for PP, but the level of use is lower (~0.05 to 0.1%) because of compatibility problems. 1,3,5-tris-(5-t-butyl-4-hydroxy-2-methylphenyl-butane or bis- (2-hydroxy-5-methyl-3-(1-methylcyclohexyl)-phenyl)-methane are used for product protection, whereas for protection during processing, and for non-toxic applications 2,6 di-t-butyl-4-methylphenol (BHT) is used. Metal deactivators such as N,N'dibenzaloxalyldihydrazide or, N,N'-bis-(3-(3',5'di-t-butyl-4'-hydroxy-phenyl)-propionyl)-hydrazine are also used.

#### 10. Material Detection or Identification

With a density of approximately 0.92 g/cm<sup>3</sup> LDPE (solid, non-filled material) will float in both water and in saturated magnesium chloride. There is no sol-

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vent for the polymer at room temperature, but at higher temperatures (approximately 55°C/131°F), LDPE is soluble in hydrocarbons and chlorinated hydrocarbons, such as xylene and trichloroethylene. Below approximately 60°C/ 140°F, PE is insoluble in all organic solvents, but it does swell in aliphatic, aromatic and chlorinated hydrocarbons. The lower the density the more it swells.

The natural color of the material is a milky white and so a wide color range is possible. When this material is heated in a flame it ignites easily and burns with a yellow-tipped, blue flame, giving off only a little smoke. It forms burning drops and, when the flame is extinguished, gives a smell like candle wax.

The easiest way of differentiating between the different PO materials is by density and melting point. LDPE has a melting point of 110°C to 125°C (230°F to 257°F), LLDPE is 115°C to 128°C (239°F to 262°F), HDPE is 130°C to 135°C (266°F to 275°F) and PP is 165°C to 175°C (329°F to 347°F). When LDPE is heated in the absence of a flame the material will soften and melt to give a clear liquid as the crystal structures are destroyed. The melt is stable, in the absence of air, up to approximately  $300^{\circ}C/572^{\circ}F$  when it decomposes to give low molecular weight hydrocarbons. Cross-linked PE will not melt but will become rubbery at approximately  $115^{\circ}C/239^{\circ}F$ . LDPE can be cut or scratched easily with a knife. The shrinkage of LDPE is of the order of 0.02 to 0.05 in/in (2% to 5%) when the density is 0.910 to 0.925 g/cm<sup>3</sup>. It is in the range of 0.015 to 0.04 in/in (1.5% to 4%) when the density is 0.926 to 0.94 g/cm<sup>3</sup>.

#### 11. Coloring

As the natural color of the material is off-white, a wide color range is possible. This does not include transparent colors in thick sections. It is sold in both compounded colors and as natural material for coloring on the processing machine, by techniques such as dry coloring, masterbatching and liquid coloring. When coloring LDPE, organic dyes should not be used, due to the problem of color leeching or bleeding (often called blush). For most coloring purposes organic and inorganic pigments are preferred. When dry coloring LDPE, colorant levels of up to 1% are generally used. Wetting agents are used with dry colorants, primarily for cleanliness in the processing shop, rather than for aiding the dispersion of the colorant. Universal type master batches are often used with LDPE at concentration levels of 1%. However, to obtain a more uniform color on a molded component, the addition level may need to be increased to 3% to 5%. When color uniformity is particularly important, LDPE-based master batches are often preferred. If opacity of color is required, then inorganic pigments are generally used.

There are no limitations to using liquid colorants with LDPE, however, levels have to be kept to a minimum (< 3%), otherwise screw slip may occur.

When coloring LDPE, its softness and very easy flow characteristics must be remembered. To improve the dispersion of the pigments within the melt, it is necessary to create an adequate mixing/grinding action within the barrel of the machine. In order to achieve this, it is often necessary to reduce the machine temperatures to the lowest values that will still enable components of the required quality to be produced.

#### 12. Materials Handling

LDPE will absorb less than 0.02% water in 24 hrs at room temperature. This means that drying is not normally necessary. If drying is necessary, the resin should be dried in a hot air oven for 3 hrs at  $65^{\circ}$ C/149°F, or in a desiccant dryer for 1 – 1.5 hrs at  $80^{\circ}$ C/176°F.

LDPE is normally sold in the form of pellets. This is the easiest form of the feedstock for use in extrusion. The simplest hopper design is nor-

**188** mally satisfactory. Sometimes LDPE may be obtained as a powder, or regranulated scrap may be added to the feedstock at fairly high concentrations. In these cases, it may be necessary to use a crammer-feeder on the hopper.

### 13. Screw and Barrel Design

LDPE is extruded using a standard screw. A typical design is shown below. **Total Length** 

24	D
Length of Feed Section	6 D
Length of Compression Section	10 D
Length of Metering Section	8 D
Channel Depth in Feed Section	0.1 - 0.15 D
Compression Ratio	3:1 to 4:1
Flight Width	0.1 D
Flight Pitch	P = D (1 D) that is, helix angle 17.7E

Screws designed to the above specification are shown in the following table: the length of each zone is shown in brackets, (i.e. 6 D).

Screw Diameter	2.5" (62 mm)	3.5" (87 mm)	4.5" (112 mm)
Feed Zone Depth (6 D)	0.35" (8.89 mm)	0.50" (12.7 mm)	0.56" (14.2 mm)
Compression (10 D)			
Metering Zone Depth (8 D)	0.09" (2.29 mm)	0.13" (3.30 mm)	0.15" (3.81 mm)

It is quite common for polyethylene screws to have a mixing head or section after the metering section. This zone is 2 to 3 D in length and consists of a number of studs arranged in a pattern around the root of the screw. As the melt passes through the mixing head, it is chopped and sheared by the rotating studs. This considerably increases the mixing and, hence, the uniformity of the melt (Note: The motor power required will be increased on adding a mixing head).

Barrier screws are frequently used with LDPE, as they produce a more even melt temperature. For higher outputs with a given barrel diameter, the barrel is fitted with a grooved feed section. The grooves run parallel to the axis of the extruder and force the solid pellets along the screw. The output from grooved barrel designs depends on the temperature of the grooved zone. For maximum output, the zone is cooled to prevent melting in the grooves. When a grooved feed is employed, then a mixing zone, usually fitted before the metering zone, is also required. Grooved extruders produce higher outputs, but the motor power requirements are higher than those for a comparably sized conventional design. Barrel wear can also be a problem with the grooved feed system.

## 14. Barrel and Die Temperatures

It must be remembered that the barrel and die temperature interact with the screw design, screw speed and material rheology to determine the melt temperature. Target melt temperatures, together with guides for the barrel and die temperatures, are shown, for three applications, in the table below.

		Blown Film	Cable Coating	Extrusion Coating
Melt Temperature	(°C)	170 - 190	190 - 200	320 - 330
	(°F)	335 - 375	375 - 395	610 - 630
<b>Zone Temperatures</b>	: (°C/°F	:)		
Zone 1		170/340	175/350	240/460
Zone 2		180/355	190/375	320/610
Zone 3		185/365	195/385	330/625
Die		185/365	195/385	325/620

## 15. Die Design and Construction

For blown film production, the spiral mandrel design has largely replaced the

side fed die type. This die has at least four ports and more usually about eight. A typical gap width at the die exit is 0.04 in (1 mm).

Damage to the die lips will cause die lines on the product. These are especially noticeable in thin blown film extrudates. Soft metal scrapers, of copper or possibly brass, must, therefore, be used to remove any blockage of the die lips. A specially made scraper, which fits inside the die lips, is useful to ensure they are clean at start up.

LDPE is not a very damaging or easily damaged material. Provided dies are correctly designed, and do not leak, they should run for lengthy periods without the need for stripping and cleaning. Molten LDPE should be removed when the die is hot.

#### 16. Downstream Equipment

As LDPE is a semi-crystalline material, a large amount of heat must be removed after the extrudate has been formed in the die. If it is assumed that the extrudate must be cooled to  $30^{\circ}$ C/86°F from a melt temperature of  $210^{\circ}$ C/  $410^{\circ}$ F, then the maximum amount of heat that needs to be removed for each gram of extrudate is 572 Joules (J). Over the above temperature range the specific heat is approximately 3180 J/kgk.

In blown film production, the molten extrudate is cooled by air, directed nearly parallel to it, from a cooling ring. The cooling ring should give an even air flow and should be kept clear of obstructions and grease. Higher outputs are obtainable with a dual-lip cooling ring. In this design, an iris diaphragm is positioned about 2 ft (approximately 600 mm) above the die and adjusted so that there is a small gap between it and the film. It will also stabilize the bubble and increase output potential. The largest increase in output is obtained by using internal bubble cooling (IBC), as well as the conventional ring. Collapsing can be done on a wooden slatted surface. The angle between the collapsing surfaces should be small. Gusset boards are positioned at the edges to gusset film where this is required. These should be relatively short to avoid too much creasing. If it is to be printed, the film is corona discharge treated. Reeling is usually by surface winding. Film can be slit or reeled as layflat. There has been some work to develop in-line bag making and printing facilities, especially for carrier bag manufacture. Though this saves warehousing and labor costs, a high degree of reliability in all components of the process is necessary to avoid expensive hold ups.

#### 17. Extruder Plasticizing Capacity

Typical maximum outputs from extruders are shown in the tables below. In the table, two output figures are given: the first are for a smooth barrel and conventional screw and the second figures are for a cooled groove feed section and a screw with a mixing zone.

Extruder Size		Output Ib/hr (kg/hr)	
(in)	(mm)	Standard Barrel	<b>Grooved Barrel</b>
1.5	38	100 (45.5)	180 (81.8)
2.5	64	370 (168.2)	550 (250.0)
3.5	89	680 (309.1)	920 (418.2)
4.5	114	1140 (518.2)	1340 (609.1)

#### 18. Starting Up

Sufficient start up time must be allowed for the extruder/die to heat and melt all of the residual polyethylene in the flow channels. This usually takes about an hour. During this time, the feed throat must be water-cooled. It is advisable to limit the temperature setting on the feed zone of the extruder to  $120^{\circ}C/230^{\circ}F$  to prevent the melt from sticking to, and blocking, the feed zone of the extruder screw. When starting, raise the temperature of the feed zone to its running value and start at slow extrusion speed.

In blown film extrusion, the air should be circulated through the cooling ring during the heating up period. It is beneficial to clean the die surface with a soft scraper and then to smear it with a silicon grease, to prevent melt sticking to the die surface during start up.

## 19. Shutting Down

LDPE is a relatively stable material and there is usually no need to purge the equipment. However, it will degrade when exposed to air, and this process is accelerated at high temperatures. Therefore, any shut down procedure should aim to eliminate air from the material and to reduce the time that the polyethvlene is at a high temperature. This can be done by reducing the screw speed to a very low value (to keep the material moving without generating too much waste), while lowering the temperature settings to ~150°C/300°F. Continue extruding in this way for a short while, then switch off completely and reduce temperatures as fast as possible. Leave the LDPE in the extruder. If you are shutting down for cleaning purposes, temperatures cannot be reduced since the material needs to be molten when the die is taken apart and the screw removed from the barrel. In this case, all traces of polymer are removed from the metal surfaces before reassembly.

#### 20. Reprocessing

Up to 25% reclaimed LDPE may be blended with virgin material. If the material has been reclaimed from film, its bulk density tends to be rather high, and there can be problems with feed in conventional hoppers if high quantities of regrind are used. In this case, crammer feeding screws can be fitted to the hopper.

## 21. Finishing

LDPE may not be joined to itself using solvents, since there are no solvents for the material at room temperature. Because of its inert 'non-stick' surface. it cannot be very successfully bonded using adhesives. There has been limited success with contact or hot melt adhesives. If the surface is made polar by flame or electrical discharge, the treated material may be bonded to metals using epoxides or nitrile-phenolic adhesives. Such treatments also improve printability. LDPE is commonly welded using techniques such as hot plate or hot shoe.

Machining of LDPE is difficult because of its soft, resilient nature. Do not apply too much pressure when machining, as the material will distort.

## 22. Other Comments

A common characteristic of all polyolefins is their excellent chemical resistance. However, radiation sterilization can cause chain scission and cross-linking, but in PE the effect of one seems to balance the other. Thus, most types of PE (LDPE and LLDPE) are resistant to moderate doses of radiation, and so may be used in gamma radiation sterilized medical devices. Antioxidants, and/or light stabilizers, may improve the resistance to radiation, but changing the antioxidant (for example, to a phenolic antioxidant), on irradiation can cause color changes in the polymer.

LDPE may be cross-linked using high energy radiation or by the incorporation of peroxides. Carbon black reinforces such materials and also improves light resistance. Internal antistatic agents used for polyolefins (polyethylene and polypropylene), include glycol alkyl esters and betaine-type materials.

## 23. Typical Products

Because of its ease of extrusion and its low cost, LDPE has become established as a general purpose extrusion material. Most of these applications, like film and pipe, do not utilize the excellent electrical insulation properties or the water resistance of the material. Close tolerances are difficult to extrude because of the material's softness, high shrinkage and wide shrinkage range.

Tolerances are also difficult to hold in service because of the high coefficient of thermal expansion and the tendency of this semi-crystalline thermoplastic to creep. Shrinkage is of the order of 0.02 to 0.05 mm/mm (or in/in) (2% to 5%) when the density is 0.910 to 0.925 g/cm<sup>3</sup>. It is of the order of 0.015 to 0.04 mm/mm (in/in) or 1.5% to 4% when the density is 0.926 to 0.94 g/cm<sup>3</sup>. The softness of this plastic may allow attack by insects or animals. It is used for insulation in preference to PVC, when its better low loss characteristics are required for long distance cabling and for high frequency transmission.

Its major application is in film for packaging, where its low cost, flexibility, toughness, heat sealability, and water resistance make it an ideal material. It can be used in bag or pouch form, in composite materials as a coating on board, paper or aluminum foil, or as a laminate or coextrudate with nylon, polypropylene and cellulose films. Though used for shrink-wrap, this application has been superseded by stretch wrapping for economic reasons. **24. Yield** 

- . Sheet of 0.1 in thickness has a surface area of 2.09 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.54 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 105 sq ft/lb weight
- . Film of 50 microns thickness has a surface area of 21.7 sq m/kg
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 29.2 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 20.0 m/kg

## ► LLDPE

1. Common Name 2. Abbreviation(s) 3. Alternative Names 4. Some Suppliers **Borealis Chemicals** Dow Carbide DSM DuPont Elf Atochem Elf Atochem EniChem Exxon Mobil Exxon Mobil Huntsman Nova Phillips Chemical Equistar Chemicals Sabic

Linear low-density polyethylene LLDPE: PE-LLD Linear low: linear low density polythene 5. Trade Names or Trade Marks Neste LLDPE Dow LLDPE, Dowlex, Elite, DFD Stamvlex PE Sclair Lacotene HX Lacqtene LX Flexirene Escorene, Unipol, Exceed (MLLDPE) Rexell Novapol, Sclair Marlex LLDPE Petrothene Ladene

#### 6. Material Properties

LLDPE was developed in the 1950's, but it is only comparatively recently that it has come into prominence. This material is a copolymer made by polymerizing ethylene with a small percentage of a higher olefin, such as octene or butene. Butene-type materials can be made by any of the present available processes and are less expensive, however, octene-type materials have slightly better mechanical properties. When LLDPE materials were first introduced, their lower price drove their use. The standard butene-based product was, in effect, being used as filler for low-density polyethylene **(see LDPE)**. This is no longer the case, since, with suitable catalysts such as the metallocenes, only low pressures and temperatures are required for LLDPE manufacture. Poly**192** merization efficiency is also high and it is possible to produce a material that has a particular molecular weight and a narrow molecular weight distribution.

The co-monomer introduces short chain branches (SCB) into the molecular structure (more than high-density polyethylene - see HDPE). So, it is possible to produce a material, which in some respects is similar to LDPE, but which can be produced without the need for high polymerization pressures. The short chain branches interfere with chain packing, so the crystallinity is not as high as for the linear HDPE. LLDPE therefore, has a lower density than HDPE. The term "linear", in this case, means that there is an absence of long chain branching (LCB). Commercial materials have densities in the range 0.89 to 0.955 g/cm<sup>3</sup> and have a narrower molecular weight distribution than LDPE. They contain a smaller amount of low molecular weight material and more medium molecular weight molecules. The average length of the molecular chain is relatively long. For a given molecular weight, therefore, LLDPE will have better mechanical properties at low temperatures (for example, -40°C/-40°F) and high temperatures, better environmental stress cracking resistance (ESC), and higher melt viscosity than LDPE. LLDPE can be stronger and tougher than LDPE. The shrinkage of LLDPE is of the order of 0.015 to 0.030 in/in (1.5% to 3.0%) and it is possible to obtain lower shrinkage values than those for LDPE and HDPE. LLDPE also exhibits a much lower differential shrinkage than LDPE and so, warping is considerably reduced.

Increasing the co-monomer content increases the flexibility, the impact strength, the tear strength, the ESC resistance, the ease of sealing and the ease of printing. However, the stiffness, yield, creep strength, hardness, Vicat softening point, and the melting point decrease proportionally. Decreasing the density, or the crystallinity, also increases the flexibility, the impact strength, tear strength, the ESC resistance, and the ease of sealing and printing. Similarly, the stiffness, yield and creep strength, the hardness, Vicat softening point and the melting point decrease proportionally.

Higher alpha olefin grades (HAO) of PE, are co- or terpolymers that contain up to 10% of octene, 4-tetramethylpentene-1 or, propylene. It is possible to produce very low-density polyethylene (VLDPE) materials that contain both long chain branching and short chain branching.

#### 7. Ease of Flow

LLDPE is an easy flow material with good heat stability, that is less pseudoplastic than LDPE. When a LDPE plastic of a certain melt flow rate (index), is compared with an LLDPE plastic of the same melt flow index, the LLDPE polymer will have higher viscosities at processing shear rates. MFR values can reach 100 with LLDPE, while with LDPE they can reach 250. (Polymers of different density, but with the same melt flow index, do not have the same molecular weight). To replace a LDPE grade, with an MFR of 7 to 10, one must use a LLDPE of MFR of 20 to 50.

Most LLDPE is used for blown film. The grades used have melt flow rates of between 0.5 and 2.0. Viscosity values for a typical film grade (MFR 1.0) are shown below.

Shear Rate (s <sup>-1</sup> )		Viscosity (Pa-s)	
ζ, γ	(190°C/374°F)	(220°C/428°F)	(250°C/482°F)
20	3460	2520	1820
40	2510	1910	1450
100	1630	1330	1080
200	1050	870	730
400	670	570	490

Another aspect of the flow of LLDPE, which differs from that of LDPE, is in melt extensional flow. This type of flow occurs when a molten

sample is stretched in tension and thins as it This sort of flow occurs after melt has left the die and is being formed into a film in the blown film process. It is easier to stretch LLDPE than LDPE (i.e., it is softer). Also the LLDPE will stretch considerably more than LDPE before breaking. The difference in extensional flow means LLDPE film bubbles tend to be less stable than those formed from LDPE. However, it is possible to draw LLDPE down to thinner films than LDPE.

Melt fracture usually appears in plastic melts at high output rates. The effect shows itself as a roughening of the extrudate surface. It generally occurs when the shear stress in the molten polymer exceeds a critical value (Note: The shear stress is related to the pressure gradient over the die land). In the case of LLDPE, the critical shear stress is about 14 MPa (2000 psi). Thus, because of its high viscosity at processing rates, melt fracture tends to occur in the extrusion of LLDPE. It can be reduced by modifying the die dimensions (see Die Design) or by modifying the surface of the die. A different metal can be used for the die land surface (alpha brass has been found successful) or the steel surface can be modified by coating it with a fluorocarbon material. These materials (polymer processing aids) are deposited on the die metal surfaces by incorporating them in the LLDPE plastic as an additive "external lubricant". The processing aid is best blended into all the LLDPE at a level that maintains the correct die surface to eliminate melt fracture. Alternatively, it can be used from time to time at higher concentrations producing a coating that gradually wears away from the metal surface. At this time another batch of concentrated additive must be used to re-coat the surface.

#### 8. Resistant to:

PE is associated with environmental stress cracking (ESC) which occurs when a material is subject to strain, or an internal stress, in the presence of polar liquids or the vapors of such liquids. Such ESC is also associated with detergents or silicone fluids, although there are many other ESC agents, such as chloroform, xylene and paraffin. LLDPE has better ESC resistance than LDPE. Higher molecular weight grades have the best ESC.

The resistance to a surface-active agent may be used to assess residual stresses in PE components. A component is immersed in a 2% aqueous solution of sodium lauryl sulphate at 40°C/104°F for 40 hr. A component with a low residual stress level will withstand this treatment without cracking, crazing or distortion.

The water vapor permeability of this type of PE, as with all types of PE, is low. PE is permeable to gases and vapors, but LLDPE and HDPE are less permeable to gases and vapors than LDPE. Permeability for organic vapors is least for alcohols and then increases in the order: acids, aldehydes and ketones, esters, ethers, hydrocarbons and halogenated hydrocarbons. (permeability decreases with density). Some grades of LLDPE are suitable for containers for oil and petrol and have been used for fuel tanks. In some cases, the formed containers do not have to be chemically modified to make them almost impermeable to fuels.

LLDPE is both radiation and ethylene oxide (EtO) resistant and can withstand multiple EtO cycles.

#### 9. Not Resistant to:

The light, or UV, resistance of LLDPE is relatively poor and is similar to that of LDPE. The cheapest way of improving this is by the addition of carbon black. Addition of 2% black can improve the weathering resistance by twelve times. If black is to be avoided then, a polymeric hindered amine light stabilizer (HALS) is more efficient than a simple HALS stabilizer. "Blooming" may be observed with the lower molecular weight HALS stabilizer.

LLDPE has limited resistance to oxygen at elevated temperatures.

**194** Antioxidants are used for protection. Unless so protected, the electrical properties will suffer. The antioxidants used with LLDPE are similar to those used with other polyolefins.

## 10. Material Detection or Identification

With a density of 0.90 to 0.94 g/cm<sup>3</sup>, the material (solid, non-filled material) will float in both water and a saturated magnesium chloride solution. There is no solvent for the polymer at room temperature, but at higher temperatures (approximately 55°C/131°F), LLDPE is soluble in hydrocarbons and chlorinated hydrocarbons, such as xylene and trichloroethylene. Below 60°C/140°F PE is insoluble in all organic solvents, but it does swell in aliphatic, aromatic and chlorinated hydrocarbons. The lower the density the more it swells.

The natural color of the material is a translucent white. When this material is heated in a flame it ignites easily, burns with a yellow-tipped blue flame, and gives off little smoke It forms burning drops and when the flame is extinguished, gives off a smell like candle wax. It can be distinguished from other polyolefins by density and melting point. LDPE has a melting point of 110°C to 125°C (230°F to 257°F), LLDPE is 115°C to 128°C (239°F to 262°F), HDPE is 130°C to 135°C (266°F to 275°F) and PP is 165°C to 175°C (329°F to 347°F).

When heated in the absence of a flame, the material will soften and melt to give a clear liquid as the crystal structures are destroyed. It is stable in the absence of air up to approximately  $300^{\circ}C/572^{\circ}F$ , when it decomposes to give low molecular weight hydrocarbons. It can be cut easily with a knife and easily scratched with a knife or fingernail.

#### 11. Coloring

As the natural color of the material is off-white, a wide color range is possible. This does not include transparent colors in thick sections. It is sold in both compounded colors and as natural material, for coloring on the machine by techniques such as solid master batches, liquid color and dry color.

Pigment selection is important as some types of pigments increase the sensitivity to stress cracking and/or warpage. When coloring LLDPE, organic dyes should not be used, due to problems of color leeching or bleeding. For most coloring purposes organic and inorganic pigments are preferred with colorant levels of up to and including 1%.

When dry coloring, adhesion promoters such as paraffin can be used. Adhesion promoters (that is, wetting agents) are used with dry colorants primarily for cleanliness in the processing shop rather than to aid the dispersion of the colorant. It is important to control the mixing time precisely, as too short a mixing time gives poor dispersion and inadequate adhesion. Too long a mixing time may cause pigment compaction. When using carbon black as a dry colorant, care should be taken to prevent it from absorbing any moisture during storage, otherwise the colored polymer will need to be dried before processing.

Universal or LDPE based master batches are often used with LLDPE, at concentration levels of 1%. However, to obtain a more uniform color on the molded component, the addition level may need to be increased to 3% to 5%. When color uniformity and optimum end-use performance are particularly important, LLDPE based master batches are often preferred. If opacity of color is required, then inorganic pigments tend to be used. There are no real limitations to using liquid colorants with LLDPE, however, colorant addition levels should be kept to a minimum to reduce the tendency for screw slip.

LLDPE's higher melt viscosity, compared to LDPE, often promotes better colorant dispersion, particularly if the machine temperatures are reduced to the lowest value that will still enable quality product to be produced. Such low temperatures improve the dispersion by developing a mixing/grinding action within the barrel of the machine.

#### 12. Materials Handling

LLDPE will absorb less than 0.02% water in 24 hrs at room temperature. This means that drying is not normally necessary. If drying is required, it can be dried in a hot air oven for 3 hrs at 65°C/149°F or in a desiccant dryer for 1 - 1.5 hrs at 85°C/185°F.

When used in pellet form, there is no problem with feeding, but difficulties can be experienced when using LLDPE in powder form. In this case, use a vibrating hopper or a secondary feed screw for the hopper

#### 13. Screw and Barrel Design

If LLDPE is run on equipment designed for LDPE, then at the same screw speed LLDPE will need considerably more motor power. This is because the viscosity of LLDPE is higher at the shear rates found in the extruder. The extra energy input results in higher melt temperatures and a greater dependence of melt temperature on screw speed. To provide better temperature control, a number of screw designs, differing from the LDPE screw, have been tested and found successful.

The basic general purpose design (described in the section on LDPE) is modified for LLDPE by deepening both the feed and the metering sections. The power requirements and the melt temperature increase are also reduced by increasing the clearance between the tips of the screw flights and the barrel, in the metering zone.

Improved designs incorporate a Maddock-type shearing section in the metering zone and a deepening of the metering zone towards the end of the screw. A 2.5 in (64 mm) screw based on this design would have the following characteristics.

Depth	Length
0.35"/8.89 mm	7 D
variable	6 D
0.23"/5.84 mm	6 D
	2/3 D
0.35"/8.89 mm	8 D
	<b>Depth</b> 0.35"/8.89 mm variable 0.23"/5.84 mm 0.35"/8.89 mm

Such a screw when run at 150 rpm would produce an output of about 220 lb/hr (100 kg/h). Union Carbide has developed decreasing pitch screws for use with LLDPE and has designs for screw diameters from 3/4 in to 8 in (19 mm to 200 mm). Compression is achieved by reducing the pitch of the screw rather than the channel depth. The design is claimed to give good output with low torque and lower than normal wear. An 18:1 screw of 2.5in (64 mm) diameter has a rated output of 395 lb/h at 100 rpm. In contrast, screws with increasing pitch have also been used successfully. These designs also increase the gap between screw flights and the barrel wall and reduce the flight widths below the customary 0.1 D.

Grooved barrel feed sections can be used with LLDPE. They offer increased output without a great increase in melt temperature, but at the expense of higher motor torque requirements and greater barrel wear. Both beneficial and adverse effects are increased by water cooling of the grooved feed section. If grooved barrels are used, the screw design needs modification. The compression ratio is reduced to less than 2:1 and the metering channel depth increased to match the greater conveying characteristics of the feed section.

#### 14. Barrel and Die Temperatures

For blown film applications, barrel and die temperatures are usually in the range 180°C to 225°C (350°F to 430°F). The target melt temperatures are in the range 190°C to 240°C (340°F to 465°F). Lower temperatures lead to high pres-

196 sures, high motor torque, and sharkskin. Higher temperatures lead to bubble instability. For cast film extrusion, melt temperatures are usually from 260°C to 280°C (500°F to 540°F). The table below shows typical zone temperature settings used to achieve a desired melt temperatures for two different MFR materials.

Typical MFR Value	1.0	0.5
	(°F/°C)	(°F/°C)
Melt Temperature	410 (210)	440 (225)
Zone 1 Temperature	365 (185)	375 (190)
Zone 2 Temperature	325 (180)	390 (200)
Zone 3 Temperature	365 (185)	395 (200)
Die Temperature	410 (210)	470 (240)

## 15. Die Design and Construction

For optimum production of blown film, spiral mandrel die designs are used. Such spiral dies should be designed to generate a relatively low back-pressure (< 3000 psi/200 bar). The melt rheology of LLDPE allows good gauge distribution, even at these low pressures. Such low pressures will cause fewer problems to the extruder. To reduce melt fracture, the die gap is set wider than with LDPE. For instance, the final inch of the die land might consist of 0.6in (15.2 mm) length at a 0.08in (2.03 mm) gap increasing to a gap of 0.1in (2.54 mm) over the final 0.25in (6.35 mm).

The effectiveness of the gap width in eliminating melt fracture, depends on the material, the output rate and the melt temperature. The following table, prepared with data from Exxon, illustrates the point. Output is per unit distance around the die circumference.

#### Minimum Die Land Gap for MFI/MFR of 1.0 LLDPE Output Rate Melt Temperature

	inert reinperature			
	(200°C/392°F)	(220°C/428°F)	(250°C/482°F)	
1.0 kg/h/cm	2.64 mm	2.18 mm	1.74 mm	
5.6 lb/h/inch	0.104 in	0.086 in	0.060 in	
2.0 kg/h/cm	3.86 mm	3.13 mm	2.50 mm	
11.2 lb/h/inch	0.152 in	0.122 in	0.099 in	
4.0 kg/h/cm	5.40 mm	4.32 mm	3.40 mm	
22.4 lb/h/inch	0.213 in	0.170 in	0.134 in	

## 16. Downstream Equipment

As LLDPE is a semi-crystalline material a large amount of heat must be removed after the extrudate has been formed in the die. For example, if it is assumed that the extrudate must be cooled to  $30^{\circ}$ C/86°F from a melt temperature of 210°C/410°F, then the maximum amount of heat that needs to be removed for each gram of extrudate is 577 Joules (J). Over the above temperature range, the specific heat is approximately 3206 J/kgk.

Because of its low extensional viscosity in the molten state, LLDPE blown film is less stable as a bubble and, if conventional cooling rings are used, outputs will be lower than LDPE. This has led to the introduction of dual lip cooling rings. With these designs, outputs similar to those for LDPE can be obtained.

The properties of solid LLDPE lead to rapid wear of cutting and slitting equipment, so specially hardened, tungsten carbide coated, blades are used.

## 17. Extruder Plasticizing Capacity

Typical figures for film extrusion, both with and without polymer processing aid (PPA) are shown in the following table.

Extrude	er Size	Without PPA	With PPA
(in)	(mm)	lb/h (kg/h)	lb/h (kg/h)
1.50	38	138 (63)	150 (68)
2.00	50	280 (127)	330 (150)
2.50	64	480 (218)	550 (250)
3.50	90	770 (350)	950 (432)

Cooling ring design affects the output capacity of the die. Some typical figures for differing cooling systems are as follows.

#### Cooling System

Output per Lb/Inch Die Circumference 5 to 7 9 to 13 14 to 18

Single Lip Venturi Dual Lip Venturi Dual Lip plus Internal Cooling

## 18. Starting Up

There are no special problems with this material. If LDPE has been used to purge the extruder, the temperatures can be set to approximately 205°C/400°F while the extruder warms up during the start up process. Then start purging, at slow screw speed, with LLDPE. At the same time, increase the temperatures to operating conditions. When operating conditions are reached, slowly increase the screw speed to the desired value.

## 19. Shutting Down

There is some evidence that LLDPE is slightly more susceptible to oxidative degradation than LDPE. On shut down, the material should be purged with LDPE and then the extruder and die temperatures lowered, while the extruder is running at a slow speed. Once temperatures are below 180°C/356°F, the extruder motor can be switched off and the power to the heater bands cut.

## 20. Reprocessing

When LLDPE is reclaimed, up to 25% may be blended into the virgin material. It should be remembered that regranulated polyethylene can be light and fluffy by nature and, because of this, has a tendency to bridge in the hopper. This will cause feeding problems, such as giving an inconsistent rate of material feed into the barrel assembly. Often, extra virgin material is added to regrind to add weight (bulk) to the regrind/virgin mix to overcome the bridging problems. When regrind material is added to virgin, it is not as critical, as with LDPE, that the regrind should be of the same density and/or MFR as the virgin polymer.

## 21. Finishing

The surface of this relatively inert material may be made more receptive to inks or to adhesives by pre-treatment using a corona discharge or ozone. This material may not be joined to itself using solvents, as there is no solvent at room temperature. Because of its inert, "non-stick" surface it also cannot be very successfully bonded with adhesives. There has been limited success, however, with contact or hot melt adhesives. If the surface is made polar using a flame or an electrical discharge, then this material may be bonded to metals, using epoxides or nitrile phenolic adhesives. Such treatments also improve printability.

It is commonly welded using techniques such as hot plate or hot shoe. When welding LLDPE, it is usual to cover the plates, or shoe, with PTFE to prevent the melted material from sticking to the hot surfaces. Machining of this plastic can be carried out using conventional machining methods and cutting tools. High cutting speeds and low feed rates are necessary to produce accurately machined surfaces. Plenty of cutting fluid should be used to cool the cutting surface and prevent heat build-up when machining. Surface grinding of LLDPE is extremely difficult and can only be done using specially developed processes. 197

# 198 22. Other Comments

LLDPE may be cross-linked using high-energy radiation or by the incorporation of peroxides. Carbon black reinforces such materials and will improve their light resistance at a comparatively low cost. Internal antistatic agents used for polyolefins (polyethylene and polypropylene) include glycol alkyl esters and betaine-type materials.

## 23. Typical Products

Film is the major market for this material. Because of its rigidity, higher tensile strength, higher elongation at break and high puncture resistance compared to LDPE, thinner films can be made (i.e., a product may be down-gauged). The higher crystallization temperature, compared to LDPE, means a faster setup time in the heat sealing operations involved in bag and pouch manufacture. This is especially important in form fill and seal operations. For a given MFR and density, LLDPE has a higher Vicat softening point (10°C to 15°C or 50°F to 59°F) than LDPE and also has good low temperature properties. The addition of very low density PE (VLDPE) will give the material increased elasticity and improve the low temperature impact strength.

Blends of LLDPE with other polyethylenes are widely used in film products. Blending LLDPE in a high molecular weight (low melt flow rate) LDPE will make greater draw down possible in the extrusion process. Without the addition of LLDPE there is difficulty in producing thin films of low MFR LDPE because the film melt ruptures in the bubble. These low MFR resins are required to give high impact, tough film products. Blends incorporating a small amount of LDPE in LLDPE have especially high hot tack strengths and thus. enable increased output in hot fill packaging operations. The compatibility of LLDPE and HDPE is also very good and such blends are, often used for blow molded containers.

Metallocene polymers (for example, a polyolefin plastomer (POP) terpolymer) may be used for LLDPE modification. It is claimed that their use results in superior dart impact strength (up 40%), higher tensile strength, 25°C lower heat-seal initiation temperatures, and up to 50% lower haze (better clarity). Ease of compounding and handling also result, as such a material is are available in pellet form.

Because of its rheological characteristics, LLDPE is not used to produce shrink film.

## 24. Yield

For the following values, the density of the LLDPE is assumed to be 0.92  $\ensuremath{\text{g/cm^3}}$ 

- Sheet of 0.1 in thickness has a surface area of 2.09 sq ft/lb. weight
- Sheet of 2 mm thickness has a surface area of 0.54 sq m/kg. weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 105 sq ft/lb weight
- · Film of 50 microns thickness has a surface area of 21.7 sq m/kg
- Tube of 3/8 in 1D and 1/16 in wall thickness has a length of 29.2 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 20.0 m/kg

# ≻ HDPE

- 1. Common Name
- 2. Abbreviation(s)

High density polyethylene (HDPE) HDPE: PE-HD: UHMW-HDPE

3. Alternative Names

Polyethylene - high density (PEHD): High density polythene: high density poly- (methylene): low pressure polyethylene: linear high density polyethylene:

ultra high molecular weight, high density polyethylene (UHMW-HDPE): Type 3 PE.

#### 4. Some Suppliers Aspell Borealis Chemicals **BP** Amoco Chemopetrol Chevron Chemical Co. Dow Carbide DSM (Dutch State Mines) DSM (Dutch State Mines) DSM (Dutch State Mines) Elenac Elf Atochem EniChem Equistar Chemicals Exxon Mobil Fina Oil & Chemical Co Huls Monmouth Plastic Inc. Monmouth Plastic Inc. Nova Phillips Polimeri Europa Solvay Chemical Ticona

## 5. Trade Names or Trade Marks Lacotene HDPE Statoil HDPE Rigidex Liten HD Dow MDPE Stamylan HD Stamylan HDR Stamylan MD (MDPE) Lupolen HD Lacgtene HD Eraclene Petrothene, Alathon Paxon. Mobil HDPE Finathene PF-HD Vestolen A Empee PE Empee PE (MDPE) Novapol, Sclair PE-m (HDPE) Unipol Fortiflex Hostalen

#### 6. Material Properties

Under conditions of comparatively low temperature and pressure, ethylene can be polymerized to give a thermoplastic material, a polyolefin called high density polyethylene (HDPE), which is substantially free from branching. This is achieved by using the stereospecific Ziegler-Natta (ZN) type catalysts. These catalysts direct the incoming monomer to make the polymer chain grow in a very ordered way. Because of its regularity, and lack of chain branching, this material has a higher level of crystallinity than LDPE or LLDPE. This increase of crystallinity produces a material that has a higher density, rigidity, tensile strength, hardness, heat distortion temperature, chemical resistance, viscosity and resistance to permeability. Although the impact strength As with all polyolefins, the weathering (UV) resistance is poor, but can be improved by the addition of carbon black and other additives.

Compared to PP homopolymer, HDPE has better resistance to low temperature impact and oxidation. HDPE has a lower stiffness, is more susceptible to environmental stress cracking (ESC) and has a high shrinkage. PP has a higher Vicat softening point, better resistance to flexing, a higher hardness, a higher tensile strength, higher elongation and will also cycle faster. The gloss on moldings can be similar. HDPE has good dynamic fatigue resistance (when injection molded there is some living hinge effect), but is not as good as PP.

The water vapor permeability of this type of PE, as with all types of PE, is low. LLDPE and HDPE are less permeable to gases and vapors than LDPE. Some grades of HDPE are accepted as suitable for containers for oil and petrol and they have been used for fuel tanks. In some cases, the formed containers are chemically modified by fluorination or sulfonation, to make them almost impermeable to fuels.

HDPE will resist deformation by boiling water. The properties of HDPE are significantly controlled by its MFR, density and molecular weight distribution. Injection molding grades of HDPE are manufactured having a narrow molecular weight distribution, compared to grades used for extrusion and blow molding.

HDPE, with a density of 0.940 to 0.965 g/cm<sup>3</sup>, is sometimes known as a Type 3 PE. The grades that have a density below 0.96 are produced using a second olefin monomer, such as butene-1 or hexene-1, at low levels (<1%). The use of the second monomer reduces the density by introducing short, side chain branching. Such materials may be known as MDPE. HDPE/MDPE is widely used in the blow molding industry. Approximately 55% of all plastics used in blow molding are HDPE/MDPE materials. This type of material is less notch sensitive than HDPE, has better ESC resistance, better shock resistance and drop resistance, and is not so hard or rigid as HDPE.

HDPE is a semi-crystalline thermoplastic, so shrinkage after processing is high (i.e., 0.015 to 0.04 mm/mm (in/in) or mm/mm or 1.5% to 4%). Shrinkage may increase to 5% for thick section moldings. Because of the wide shrinkage range for HDPE, it is difficult to accurately predict the correct shrinkage value to attain the desired dimensions for a particular part. The shrinkage value is dependent upon the degree of orientation and crystallinity in the finished component. Because of the wide possible variation, samples must be manufactured using typical processing conditions to determine the shrinkage values used for sizing. However, the degree of after-shrinkage that occurs with HDPE must also be taken into consideration. This means that the samples should be conditioned for several days before being measured.

#### 7. Flow Characteristics

This material is an easy flowing material with melt flow rate, or index, (MFR or MFI) values ranging from 0.1 to 28. However, because the impact resistance of HDPE can be low for low molecular weight grades, high molecular weight materials are preferred. For example, the molecular weight of a high molecular weight (HMW) blow molding grade, as measured by solution viscosity, may be 150,000. That of an injection molding grade, or a grade for small blow moldings, may be 75,000.

Using the MFR method of assessment (with a 2.16 kg weight at 190°C), high molecular weight (HMW) HDPE extrusion blow molding grade will have a low value (<0.3). Even medium molecular weight grades (MMW) are often below 0.6. Such low values pose difficulties of grade separation. To more readily distinguished between grades, the MFR test, for HMW resins, is carried out with a larger weight, often with 21.6 kg weight at a temperature of 190°C/374°F. The MFR (1900C/21.6 kg) may range from 2 to 10, depending on the grade.

The impact strength falls off with increasing MFR (flow is becoming easier) and the HDPE become more notch sensitive.

Viscosity values for a typical pipe grade are as follows:

MFR (5) = 0.43.	Power Law Index = 0 Viscosity (Pa-s)	.462	
(180°C/356°F)	(200°C/392°F)	(220°C/428°F)	
4187	3643	3169	
2884	2509	2183	
1761	1532	1334	
1213	1055	918	
836	727	633	
	MFR (5) = 0.43. (180°C/356°F) 4187 2884 1761 1213 836	MFR (5) = 0.43.     Power Law Index = 0 Viscosity (Pa-s)       (180°C/356°F)     (200°C/392°F)       4187     3643       2884     2509       1761     1532       1213     1055       836     727	

#### 8. Resistant to:

HDPE has no known solvent at room temperature, but is soluble in hydrocar-

bons and aromatic hydrocarbons at temperatures above 60°C/140°F. Hydrocarbons, white spirits and carbon tetrachloride will cause swelling at room temperature as this plastic is permeable to them. In this respect HDPE is superior to PP. It resists aromatic and chlorinated hydrocarbons better than LDPE. Theoretically, it is more resistant to oxidation than LDPE, but in practice they are similar. This could be due to catalyst residues in the HDPE.

#### 9. Not Resistant to:

PE is associated with environmental stress cracking (ESC), which occurs when a material is subject to strain, or an internal stress, in the presence of polar liquids or the vapors of such liquids. Environmental stress cracking (ESC) is also associated with detergents or silicone fluids, although there are many other environmental stress-cracking agents, such as chloroform, xylene, and paraffin. It is possible to minimize ESC problems by reducing residual stresses in components, careful component design, and by using the lowest MFR grade, at any particular density level. The resistance to a surface-active agent may be used to assess residual stresses in PE components. This can be done by immersion in a 2% aqueous solution of sodium lauryl sulphate at 40°C/104°F for 40 hrs. A component with a low residual stress level will with-stand this treatment.

If light, or UV resistance is unsatisfactory, then the cheapest way of improving this is by the incorporation of carbon black. Carbon black filled HDPE compounds are now being used for applications where very long term weatherability is required. Although HDPE is not as susceptible to oxidation at high temperatures (experienced during melt processing) as polypropylene (PP), HDPE has relatively limited resistance to oxygen at elevated temperatures. Antioxidants are used for protection. Unless HDPE is protected by antioxidants, the electrical properties will suffer. The antioxidants used are similar to those used for PP, but the level of use is lower (~0.05% to 0.15%) because of compatibility problems.

A metal deactivator, or copper inhibitor, may be added to this polyolefin to reduce the harmful effects of copper. Copper is not the only metal that will catalyze degradation, but it is very common metal to come into contact with a PO (for example, high density polyethylene is used to insulate copper wire). Examples of such metal deactivators include N,N'-dibenzaloxalyldihydrazide or N,N'-bis-(3-(3',5'-di-t-butyl-4'-hydroxy-phenyl)-propionyl)hydrazine at up to 0.5%. If the HDPE is in a cellular form, or in contact with petrolatum, then heat stability may be reduced.

#### 10. Material Detection or Identification

With a density of 0.94 to 0.965 g/cm<sup>3</sup> HDPE, solid, non-filled material will float in both water and in a saturated magnesium chloride solution. There is no room temperature solvent, but at higher temperatures (approximately 55°C/ 131°F) HDPE is soluble in hydrocarbons and chlorinated hydrocarbons (e.g., xylene and trichloroethylene). Below approximately 60°C/140°F, HDPE is insoluble in all organic solvents, but it does swell in aliphatic, aromatic and chlorinated hydrocarbons. The lower the density the more it swells.

The natural color of the material is a milky white, so a wide color range is possible. Transparent components are not achievable in thick sections. When this material is heated in a flame, it ignites easily, burns with a yellow-tipped blue flame, and gives off only a little smoke. It forms burning drops and when the flame is extinguished, gives off a smell like candle wax. HDPE is often rated as HB by the UL 94 test and has an oxygen index value of approximately 17. The melting point is 130°C to 135°C/266°F to 275°F (PP is approximately 165°C to 175°C/329°F to 347°F). Cross-linked HDPE will not melt, but will become rubbery at approximately 115°C/239°F.

When heated in the absence of a flame the material will soften and

202 melt, at approximately 135°C/275°F, to give a clear liquid, as the crystal structures are destroyed, HDPE is stable in the absence of air up to approximately 300°C/572°F when it decomposes to give low molecular weight hydrocarbons. It can be cut relatively easily with a knife and can be scratched with a knife or fingernail.

## 11. Coloring

As the natural color of the material is off-white, a wide range of colors, not including transparent colors, is possible. It is sold in both compounded colors and as natural material for coloring on processing equipment, by techniques such as masterbatching, dry coloring and liquid coloring. When dry coloring one should not use adhesion promoters such as paraffin. The mixing time should be controlled precisely, as too short a mixing time gives poor dispersion and inadequate adhesion. Too long a mixing time may cause pigment compaction. One should not exceed approximately 0.4% pigment concentration. Higher color concentrations are achieved with colored compounds.

Pigment selection is important, as some types of pigments increase the sensitivity to stress cracking and/or warpage. In general, inorganic pigments cause less warpage than organic pigments. For example, ultramarines, manganese violet and carbazole violet are better in this respect than quinacridone violet, indanthrone blue and phthalocyanines. It is best to consult coloring specialists for advice on the type of pigments that can be used for HDPE. When using carbon black as a dry colorant, care should be taken to prevent it from absorbing moisture during storage, otherwise, the colored polymer will need to be dried before processing.

## 12. Materials Handling

HDPE will absorb less than 0.01% water in 24 hours at room temperature. This means that drying is not normally necessary. If it is, the polymer should be dried in a hot air oven for 3 hrs at  $65^{\circ}C/149^{\circ}F$  or in a desiccant dryer for 1 to 2 hrs at  $80^{\circ}C/176^{\circ}F$ .

HDPE is available in both granular (pellet) and powder form. The powder form is cheaper, but can give problems with materials handling and feeding. The granular form is much easier to handle as it will flow under its own weight. There can, however, be problems if the plastics material is blended with masterbatch, as masterbatch separation can occur in the machine hopper.

## 13. Screw and Barrel Design

Because of its high viscosity at extrusion processing temperatures, there can be a problem, at high screw speeds, with shear heating raising melt temperatures to unacceptably high values. Screws should be designed and/or operated with this problem in mind. A relatively deep metering zone channel should be used and moderate screw speeds employed. With conventional screw designs, there should be a relatively short compression zone with a compression ratio of 3:1. For pipe extrusion, screw lengths are designed to be in the range 25 to 30 D, while for film the range is from 18 to 24 D.

Improved output is obtained with a decompression screw, where the metering zone channel is deepened in the final 8 D. Zone depths are given in the following table.

Screw	ı Diameter	Feed Zo	one (12D)	1 <sup>st</sup> Mete	ring (3D)	2 <sup>nd</sup> Mete	ring (8D)
(in)	(mm)	(in)	(mm)	(in)	(mm)	(in)	(mm)
2.5	63	0.367	9.3	0.143	3.6	0.236	6.0
3.5	89	0.450	11.4	0.186	4.7	0.294	7.5
4.5	114	0.517	13.1	0.220	5.6	0.337	8.6
6.0	152	0.616	15.6	0.268	6.8	0.417	10.6

Barrier type screw designs are commonly used with HDPE. Typically a 4.5 in (114 mm) barrier screw will have a 24:1 L/D ratio and be operated at up to 100 rpm. If grooved feed inserts are used in the barrel, they should be cooled and the grooves should be fine: for a 2.5 in (62 mm) extruder, 8 grooves should be used, each with a depth 0.10 in (0.25 mm) and width 0.3 in (0.4 mm). The grooved insert should extend 3.5 D beyond the feed throat opening and a longer screw, incorporating shear and mixing sections, should be used.

#### 14. Barrel and Die Temperatures

Since melt temperature depends on factors such as screw design, screw speed, barrel temperatures, and die settings, the following table should only be considered as a guide for a 5 MFR material.

Zone on Barre	l Sheet	Fil	m	Pipe
	(0.6 in/15.2 mm)	(0.17 in/4.3 mm)	(0.53 ib/13.5 mm)	) (0.43 in/10.9 mm)
Zone 1 (°C/°F)	185/370	190/375	190/375	190/375
Zone 2 (°C/°F)	195/380	230/445	200/390	205/400
Zone 3 (°C/°F)	210/400	250/480	220/430	215/420
Die (°C/°F)	210/400	250/480	220/430	215/420

Although HDPE has a wide processing temperature range and good thermal stability, accurate temperature control is necessary, particularly when using melt temperatures greater than 240°C/464°F, to produce product of a consistent color. Poor temperature control can often lead to degradation of the pigment, which results in color changes during the production run.

#### 15. Die Design and Construction

**Pipe Dies.** For smaller pipes, spider type dies are generally used. For large pipes, a spiral mandrel design is used, or the conventional mandrel is held in place by a breaker plate-type support. The land length should be 14 to 25 times the pipe diameter. The greater the output velocity the greater the necessary land length.

**Blown Film Dies.** These dies are of the spider type with a smear restriction downstream of the spider supports or they may be the spiral mandrel type. Typically, the die gap is between 0.03 in and 0.055 in (0.75 mm and 1.4 mm) and the die diameter between 3 in and 8 in (75 mm and 400 mm).

**Sheet Dies.** The types of dies used for high impact polystyrene (HIPS) are generally satisfactory. Sheet thickness of up to 0.5 in (12.5 mm) are possible. The die should have an adjustable land after the manifold and choker bar sections. Land lengths of 2.5 in (62 mm) have proved satisfactory.

#### 16. Downstream Equipment

If the HDPE has a melt temperature of 240°C/464°F, and the melt is cooled to 20°C/68°F, then the maximum amount of heat that needs to be removed for each gram of output is 800 Joules (J). Over the temperature range quoted, the specific heat of HDPE is approximately 3640 J/kgK. What must be borne in mind, when processing HDPE, is the large amount of heat that needs to be removed per gram of output. The above figure of 800 J/g, means that approximately twice the amount of cooling capacity is needed to remove the heat from HDPE, as compared to that required for PS.

Both pressure and vacuum sizing are used for pipe production. For pressure sizing, there is a slight expansion after the die (i.e., the die gap OD equals the pipe ID). For vacuum sizing the expansion is greater, with a 25% increase in diameter common.

In order to achieve reasonable impact values with blown film, a relatively large blow up ratio (between 3 and 6) is employed. The bubble has a long neck (between 6 and 9 times the die diameter) and a characteristic wineglass shape. To achieve greater stability with this shape of bubble, an internal stabilizer can be used, together with an outer "iris diaphragm". In these cases, the bubble often contracts to a diameter less than that of the die opening, before eventually expanding to the final film diameter. Sheet is extruded onto a polishing roll stack consisting of 3 to 5 polished, chrome plated, rolls of at least 8 in/200 mm diameter. The gap between polishing rolls is often about half of the die gap, or slightly more for the higher melt index resins. Roll temperatures are typically as follows:

The temperatures refer to the circulating fluid set points. Surface temperatures will be less until the extrudate is being cooled.

- Top Roll = 210°F/99°C
- Middle Roll = 230°F/110°C
- Bottom Roll = 225°F/105°C
- Fourth Roll = 190°F/88°C
- Fifth Roll = 150°F/66°C

## 17. Extruder Plasticizing Capacity

Line capacity depends on the extruder, the die and the cooling section. The table below shows some typical figures in lb/hr and kg/hr.

Extru	der Size	Blown Film	Thin Sheet	Thick Sheet
(in)	(mm)		(<0.04 in/1.00 mm)	(0.25 in/6.2 mm)
1.5	38	110/50		
2.5	62	370/168	330/150	215/98
3.5	88	680/309	640/291	415/189
4.5	114		1150/523	750/341
6.0	150		2250/1023	1500/682

## 18. Starting Up

There are no special problems with HDPE. In general, follow the procedures laid down for LDPE. Take care not to melt the polymer in the feed section before extrusion starts, by keeping temperatures low until just before the screw is started.

## 19. Shutting Down

There are no special problems in shutting down with HDPE, although for die and extruder cleaning it is helpful to purge with LDPE. To prevent oxidation, leave polymer in the extrusion equipment and reduce temperatures as fast as possible, when it is intended to start again without cleaning the equipment in the intervening period.

## 20. Reprocessing

When this material is reclaimed, up to 20% may be blended into the virgin material. One of the main problems when using regrind material is the inability to maintain a consistent rate of material feed into the barrel assembly. Regranulated polyethylene is light and fluffy by nature, and because of this, it has a tendency to bridge in the hopper, creating feeding problems. The use of a regrind/virgin blend or a mechanical feeder helps to overcome the bridging problem.

## 21. Finishing

This material may not be joined to itself using solvents, as it has no solvent at room temperature. Because of its inert "non-stick" surface, it also cannot be very successfully bonded using adhesives. Limited success has been achieved with contact or hot melt adhesives. If the surface is made polar by using a flame or an electrical discharge, this material may be bonded to metals using epoxides or nitrile-phenolic adhesives. Such treatments also improve print-ability. For some components, flaming is popular; to ensure satisfactory adhesion. The surface tension, after treatment, should be >44 mN/m. This is achieved by using an oxidizing flame at temperatures of >1000°C with short contact times (a few hundredths of a second). Corona discharge treatment may be used before printing on HDPE.

Because of the excellent resistance of HDPE to solvents, the use of solvent based adhesives is limited. It is commonly welded using techniques

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such as hot plate or hot shoe. When welding HDPE, it is usual to coat or cover the hot plates with PTFE to prevent the material from sticking to the surfaces of the hot plate. Printing, surface coating, hot foiling or vacuum metallizing can be used to decorate components.

Machining of this plastic material can be difficult because of its soft resilient nature. Do not apply too much pressure when machining, as the material will distort. HDPE can, however, be readily machined with cutting tools used for metals. Use high cutting speeds and low feed rates. The edges of the cutting tools must be sharp in order to achieve a good surface finish. Use plenty of cutting fluid to cool the cutting surface and prevent heat build up when machining. Because of it's low surface hardness, it is extremely difficult to achieve a high gloss finish (comparable to a molded finish) on the machined surface.

#### 22. Other Comments

The effect of increasing molecular weight (MFR), density, and molecular weight distribution (MWD) on the material properties are shown below.

Property	MFR	Density	MWD
Pseudoplasticity			Increases
Tensile strength at yield		Increases	
Tensile strength at break	Decreases	Increases	
Elongation at break	Decreases	Decreases	
Impact strength	Decreases	Slight decrease	Decreases
Modulus		Increases	
Transparency		Decreases	
Long term load-bearing		Increases	
ESC resistance	Decreases	Decreases	
Softening temperature		Increases	
Melt strength	Decreases		Increases
Melt elasticity	Decreases		Increases
Melt fracture tendency	Decreases		Decreases
Gloss			Decreases

HDPE may be cross-linked using high-energy radiation or by the incorporation of peroxides. Carbon black reinforces such materials and also improves light resistance.

Internal antistatic agents used for polyolefins (polyethylene and polypropylene) include glycol alkyl esters and betaine-type materials.

## 23. Typical Products

This material is strong and stiff, even at low temperatures. Although it has a higher density than PP, the cost difference is often low enough to allow HDPE to compete with PP for various applications. A major use for HDPE is in blow molding applications. Large containers or drums (e.g., 180 liters/45 gallons capacity) toys, and car components (fuel tanks and spoilers) are typical applications. HDPE is used in these applications because of its high impact strength and of its ability to retain its properties at low temperatures (e.g., -180°C). To obtain the required properties in the finished product (strength and creep resistance), it is often necessary to use high molecular weight resin (HMW HDPE or HDPE HMW).

For blow molding, most commercial HDPE resins have molecular weights in the range of 50,000 to 250,000. HMW material will be higher than this, say 250,000 to 1 million. Ultra high molecular weight materials (known as UHMW HDPE or HDPE-UHMW) will have molecular weights greater than 1 million. This material has tremendous properties, but processing is very difficult.

Because of the stiffness of this material, it is widely used as carrier (tote) bags or counter bags. In such applications, it is possible to make the product in very thin gauges because of the strength of this plastic. It is still very strong even though it is thin. The water resistance of this material is a big advantage in such applications. It is used in packaging applications in the form of thin, paper-like film, for example, for the wrapping cheese. Pipe, especially as gas and cold water supply pipe, is another major application for this plastic. This is because of the rigidity of this material and its comparatively low cost. Sheet for thermoforming is also made, but in comparatively small quantities. A disadvantage of HDPE in thermoforming is that its high specific heat leads to long heating and cooling cycles.

Plastic materials can be made biodegradable by the use of additives or by making special polymers. By copolymerization of ethylene with carbon monoxide, it is possible to produce a material that is biodegradable. As the carbon monoxide level increase so does the brittleness. This material can be made more biodegradable than paper board.

## 24. Yield

- Sheet of 0.1 in thickness has a surface area of 2.00 2.03 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.52 0.53 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 100 101 sq ft/lb weight
- Film of 50 microns thickness has a surface area of 20.8 21.1 sq m/kg
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 27.9 28.3 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 19.2 19.4  $\ensuremath{\text{m/kg}}$
- Pipe of thickness 0.1 in and 4 in ID has a length of 1.64 1.66 ft/lb
- Pipe of thickness 2 mm and 100 mm ID has a length of 1.44 1.46 m/kg
- Paper-like film: 1 lb of resin will produce 400 405 sq ft of film at a thickness of 0.0005 in (50 gauge). 1 kg of resin will yield 104 to 105 m<sup>2</sup> of 10 micron film

# ≻ PP

## 1. Common Name

#### Polypropylene

## 2. Abbreviation(s)

PP or sometimes PPR. A homopolymer may be identified as PP-H and/or as, PP-HO. A copolymer may be identified as PP-CO (such abbreviations are also seen without the hyphens). If PP is used without qualification, then the initials may stand for either a homopolymer (PP-H) or for a copolymer (PPCO). Copolymer is usually an ethylene-propy-lene block copolymer. PPRACO indicates a random (RA) copolymer (CO). The use of m, as in mPP or PP-m, indicates a material prepared using a metal locene catalyst. miPP is sometimes used for isotactic PP prepared with a metallocene catalyst and msPP is sometimes used for the syndiotactic polymer. PPAL indicates a polypropylene (PP) alloy (AL) while PPCOM may be used for a PP compound (COM). PPGF means polypropylene filled with glass fiber (GF) while PPRC means recycled (reclaimed) PP.

## 3. Alternative Names

Polypro: polypropene: poly(propylene): polymethylethylene. The homopolymer (PPH) may also be simply referred to as homopolymer or as homo-PP. The block copolymers, based on propylene and ethylene (PPCO), may be referred to as impact copolymer PP, propylene copolymer (PPCO), polyallomer, or as heterophasic copolymer PP (PP HECO). The random copolymers, based on propylene and ethylene (PPCO), may be referred to as random copolymer PP (PP RACO or PPr).

Propylene Homopolymers (PPH) an	d/or Copolymers (PPCO)
4. Some Suppliers	5. Trade Names or Trade Marks
Amoco Chemicals	Amoco PP
Bamberger Polymers	Bapolene PP
Borealis Chemicals	Statoil PP
Carmel Olefins	Capilene
Chemopetrol	Mosten
ComAlloy	Comalloy PP (PPCOM)
Discas	Reprean
Dow Carbide	Calibre, UCPP
DSM (Dutch State Mines)	Stamycom PPRC
DSM (Dutch State Mines)	Stamytec (PPCOM)
DSM	Xantar
DSM	Stamylan P
Eastman Chemical Co.	Tenite
El Paso	Rexene
Elf Atochem	Appryl
EniChem	Sinvet
Epsilon	Epsilon E
Exxon Mobil	Achieve (mPP)
Federal Plastics	FPC PP
Ferro	Gapex (PPGF)
Ferro	Ferrolene (PPCOM)
Fina	Fina
Huls	Vestolen P
Huntsman	Rexene
Lati	Latilon
Lati	Latene
MA Hanna	Maxxam
LNP	Statcon, Therm
Mitsui Plastics	Polypro
Monmouth Plastics	Empee PP
Montell-Targor	Pro-fax, Metocene, Adflex
Montell-Targor	Valtec
Montell-Targor	Hivalloy (a PPAL)
PCD	Daplen
Phillips Sumika	Marlex
Polifil	Polifil HM
Polikemi AB	Polyfill (PPCOM)
RTP	RTP
Shell Chemicals	Shell PP
Slovnaft	latren
Solvay Chemicals	LITEX P
Solvay Polymers	Fortilene
Sumitomo Plastics America	NODIEN PP
	I nermotil PP (PPCUNI)
Ticona	
	HUSLAIEN PP (MPP)
Ube Industries	
wasnington Penn Plastics	washpen PP

6. Material Properties

High molecular weight PP may be produced from the monomer propylene by the use of Ziegler-Natta (ZN) catalysts. PP is a semi-crystalline, thermoplastic

208 material with relatively high shrinkage (0.018 in/in (or mm/mm) or 1.8%). It can reach 2.5% in thick sections, but PP has a more uniform shrinkage than HDPE. Commercial PP is mainly isotactic (approximately 90% to 95% isotactic) and has a number average molecular weight of approximately 40,000 to 60,000. Being isotactic, the long chain homopolymer formed (PP-H), has sterically ordered side groups (all the methyl groups lie on one side of the chain). PP-H like PE, is a linear, hydrocarbon plastic, but is stiffer, harder and has a higher melting point. 100% isotactic PP has a melting point greater than 170°C/338°F and a specific gravity (SG) of approximately 0.91. Until recently, there were three major types of PP; PP homopolymers (PPH), impact copolymers, and random copolymers. Now the range of PP resins has been extended by the introduction of grades produced using a metallocene (m) catalyst, which may be identified as PPm or mPP.

PP-H is produced from propylene and is rigid, highly crystalline, plastic with high temperature resistance and good contact clarity (mPP grades can have a contact clarity comparable to that of polystyrene). In general, PP has high strength and stiffness, but a comparatively low notched-impact strength. It becomes very brittle at slightly below room temperatures and, for this reason, copolymers with ethylene (PPCO) are often preferred. Typical applications for PPH are for fibers, thermoformed dairy pots, oriented and non-oriented films, and thin-walled injection moldings. It is also used in applications that utilize its high heat distortion temperature (HDT). Examples include electrical housings, hair dryers, and deep fat fryers.

An ethylene-propylene block copolymer (i.e., based on propylene and ethylene) may be referred to as an impact copolymer, a heterophasic copolymer, a polyallomer, a propylene copolymer, or as an allomer. It may also be abbreviated as PP, PP-CO, or PP-B (block copolymer). Block copolymers, which can contain a higher ethylene content than a random copolymer, are often used in place of PPH in injection molding, since the molding of thick sections (without voids) is easier. These copolymers have a lower heat distortion temperature (HDT), less clarity, less gloss, and less rigidity, but greater impact strength (even at low temperatures), and fatigue resistance than PP-H. Their flow is stiffer and they demonstrate higher mold shrinkage than PPH. As the proportion of ethylene increases the material becomes softer and tougher. Commercial materials often have 5% to 15% of ethylene blocks and thus avoiding the problem of a loss of impact strength below room temperatures (the ductile to brittle transition can be as low as -40EC/-40EF). Typical applications are for sheet, profiles, crates, housewares, automotive battery cases and bumpers.

Flexible materials, with excellent clarity, may be produced by random copolymerization of propylene with ethylene. The ductile to brittle transition temperature is approximately 10°C/50°F. Typical applications for such soft grades of PP are for packaging films and for containers, where clarity is very important. Blow molding, injection molding, and thermoforming are used to produce these containers. Some random copolymers (may be known as PP RACO) are being promoted for blow molding applications, as they are clear materials with a high gloss and a Vicat softening temperature (VST) of approximately, 128°C/263°F (about 200C lower than PP-H). Such materials compete with other soft thermoplastic materials, such as PPVC and EVA. Grades of mPP are now available with a modulus of elasticity 60% greater than a PP-RACO, but with the same transparency.

PP (that is, PP-H and PP-CO) can have a Rockwell hardness (R scale) of approximately 90, a density of 0.9 g/cm3, a Vicat softening point of about 150°C/302°F and a heat distortion temperature of approximately 100°C/212°F (random copolymers have lower values than this). Parts made from PP can withstand boiling water and steam sterilization and do not suffer from

environmental stress cracking (ESC) problems. The maximum service temperature is above 100°C (110°C/230°F). The electrical insulation properties of PP are good and a high gloss, scratch-resistant, surface is possible.

PP is extensively modified by the addition of glass fibers (GF), mineral fillers, thermoplastic rubbers or a combination of these. For example, talc filling improves the rigidity, hardness, creep resistance, dimensional stability and the HDT of PP. However, it worsens surface appearance and elongation at break. Calcium carbonate filling improves the surface hardness, elongation at break, and the notched impact strength, at lower cost, compared to talc. However, a lower modulus, tensile strength and HDT, compared to talc, result. Glass fiber (GF) filling improves the rigidity, stiffness, hardness, creep resistance, dimensional stability and the HDT of PP but worsens the surface appearance and elongation at break. There is a greater improvement in properties, if the fillers/fibers are coupled to the PP. Rubber modification, to produce an elastomer modified grade, improves the low temperature impact strength and the elongation at break (of both PP-H and PP-CO). There is some reduction in stiffness and an increase in the cost. The use of rubber helps to offset the effect of filler addition. New polymerization technology now allows the production of, directly polymerized, soft grades that compete with elastomer modified PP. The flexural modulus of a directly polymerized, super-tough material can be as much as 80 MPa.

PP producers continue to develop and to create new markets. This is because of the incredible versatility of the material and the sophisticated nature of the polymerization catalyst systems now employed. For instance, two incompatible polymers may joined, forming what is called a graft copolymer. To do this, one monomer is polymerized in the presence of a formed polymer and some of the new polymer formed, chemically grafts, or joins, onto the existing polymer to covalently bond them together. Thus, one can produce a thermoplastic material, based on a polyolefin (PO), onto which is bonded, or grafted, an amorphous material. Such a material may be referred to as high value alloys (like Hivalloy from Montell). These alloys will compete with materials such as nylon, acetal and polyphenylene oxide (modified). By using different co-monomers it is possible to produce grades that have good weatherability, or good dimensional stability and impact strength or another that has good dimensional stability at high temperatures. A major advantage of these copolymers is their low densities, which range from 0.93 to 0.96 g/cm<sup>3</sup>, regardless of the grafted comonomer.

Most of the chemical companies that produce polyolefins, are investing in new technology, especially metallocene-based catalysts, to obtain a wider range of products (high product flexibility), at comparatively low product costs, from their production facilities. Because of such developments and investments, homopolymers and copolymers based on olefins will increasingly dominate the plastics industry. Metallocene catalyzed polypropylene (mPP) is predominantly based on propylene and produced using a metallocene constrained geometry catalyst. The PP produced can be a homopolymer or a copolymer and the process can produce both isotactic polypropylene (iPP or miPP) and syndiotactic polypropylene (sPP or msPP). In general, the miPP is claimed to have properties that are superior to other types of iPP, such as improved toughness, better clarity, and lower heat seal temperatures. It can be used in place of post-reactor peroxide-cracked polypropylene and medical grade PP (mPP has lower extractables). Extra stiff grades with good clarity and impact strength are now also available. At present, mPP costs more than conventional PP, but it can be processed on similar equipment.

#### 7. Flow Properties

PP extrusion resins can have melt flow rates (MFR) (230°C/21.6 N or 2.16 kg) between 0.4 and 8. The low MFR values refer to materials with high impact

210 strength, while the high values refer to those with easy flow characteristics. Low MFR material is used for pipe and sheet while the high MFR materials are for film. Increasing the molecular weight of PP (decreasing the MFR) reduces the tensile strength and stiffness, but increases the impact strength, as the high molecular weight material is more difficult to crystallize. The melt is more temperature and shear sensitive than PE, and the higher viscosity grades have fewer cavitation problems in thick sections. PP-CO is tougher than PP-H of the same nominal MFR. Such materials offer advantages for low temperature applications.

In the table below viscosity values are given for a grade with an MFR in the middle of the above range.

Shoar Data (s-1)	(204°C/400°E)	Viscosity (Pa-s)	(260°C /500°E)
Sileal hate (S)	(204 0/400 1)	(232 0/430 1)	(200 0/300 1)
20	2560	2110	1740
40	1570	1290	1060
100	820	680	560
200	500	410	340
400	310	250	210

## 8. Resistant to:

The chemical resistance of PP is very similar to HDPE. PP is resistant to a wide range of common solvents and organic liquids. It has outstanding resistance to hot water and chemicals. It can only be dissolved by aromatic hydrocarbons, such as xylene, at elevated temperatures. It is relatively unaffected by aqueous solutions, including strong acids and alkalis. Because of swelling problems at room temperature, it is not recommended for use with aromatic hydrocarbons (e.g., benzene) and chlorinated hydrocarbons (e.g., carbon tetrachloride, chlorobenzene, chloroform, and trichloroethylene). PP is also swollen by esters (e.g., DBP and DOP), ethers (e.g., diethyl ether), asphalt, camphor oil and various aqueous oxidizing agents (e.g., dilute nitric acid and potassium permanganate).

PP may eventually yellow and lose some elongation at break as a result of radiation, although containers and PP bags may be sterilized by gamma radiation. Specifically designed formulations, involving additive packages, are available for PP and such a stabilized material can be exposed to 5 Mrad of gamma radiation and still maintain useful physical properties for up to four years. The additive package may contain a hindered amine light stabilizer (HALS) and antioxidants. PP items may be steam sterilized as the material can withstand temperatures of the order of 125°C/257°F. Because of the inert nature of this material, ethylene oxide (EtO) sterilization is also possible.

## 9. Not Resistant to:

In general, PP is not resistant to outdoor exposure unless protected by carbon black or UV stabilizer. It is less resistant to UV than HDPE. The most commonly used non-black, light stabilizers are those based on ultraviolet absorbers (e.g., 2-hydroxy-4-alkoxybenzophenones and 2-(2'hydroxyphenyl) benzotriazoles, nickel (11) chelates, 3,5-di-tert-butyl-4-hydroxy-benzoates, and hindered amine light stabilizers (HALS). Mixed stabilizer systems are often much more efficient than a single stabilizer. The introduction of polysiloxane-based hindered amine light stabilizers has dramatically improved UV stability.

Because of the presence of tertiary carbon atoms, PP is susceptible to oxidation at the high temperatures experienced during melt processing. Combinations of antioxidants are widely used, as it is found that such combinations are much more efficient than a single material. A widely used combination, used as a processing stabilizer package (because both materials are
acceptable for food packaging) is butylated hydroxytoluene (BHT) and dilauryl thiodipropionate (DLTDP). A higher molecular weight, sterically hindered phenol may also be used as a long term heat stabilizer. The use level ranges between 0.05% to 0.25%.

PP is dissolved by aromatic and chlorinated hydrocarbons at elevated temperatures (85°C/185°F). Strong oxidizing agents, such as oleum and fuming nitric acid (especially when warm) degrade it. It is less resistant to thermal oxidation than HDPE. High temperatures and contact with copper, or cuprous alloys, will cause rapid decomposition. However, grades are available that contain thermal stabilizers to effectively reduce this degradation problem.

PP materials will burn easily, but various flame-retardants are available. Ammonium polyphosphate is a popular non-halogenated flame retardant for PP that is used in masterbatch form and is stable at temperatures up to 260°C/500°F. Because of the relatively high processing temperatures required for PP, the use of aluminum trihydrate (ATH) as a flame-retardant is limited, as it decomposes at ~200°C/392°F. Compounds based on magnesium hydroxide, which decomposes at higher temperatures, may be used.

### 10. Material Detection or Identification

With a density of 0.9 g/cm<sup>3</sup> the material (solid, non-filled material) will float in both water and in saturated magnesium chloride solution. The homopolymer, which is 90% to 95% isotactic, consists of an approximately 50:50 mixture of amorphous material (SG 0.85) and crystalline material (SG 0.94). This gives an average SG of 0.9 for the unfilled material. The addition of 30% glass fiber can raise the density to approximately 1.1 g/cm<sup>3</sup>, but decrease the shrinkage to about 0.7%. The unfilled material will float in isophorene, but sink in an 80:20 mixture, by volume, of alcohol and water.

The natural color of the material is translucent to clear. When the percentage of haze is greater than 30%, a material is considered to be translucent rather than transparent (haze is the amount of light scattered in a forward direction by surface irregularities and/or, inhomogeneities). When haze is measured on a 1 mm injection molded sheet, the value for a nucleated PP RACO may be 15%, for PP HECO 75%, and for a nucleated PPH 18%. Sodium 2,2'- methylene-bis-4-t-butyl phenyl phosphate is an organo-phosphorous salt that may be used as a nucleating agent for PP.

PP components usually feel hard and have a pleasant, dry, handle or feel. It may feel slightly waxy. Molded surfaces cannot be readily scratched or scuffed and are virtually unbreakable unless cut. New types of PPH are very stiff (modulus of 2400 MPa) and yet still have haze values of only 22%.

When PP (PPH or block PPC0) is heated in a flame then, it softens to give a melt at ~165°C/329°F and burns readily with a faintly luminous yellow tipped blue flame. There is very little residue, and flaring and burning drips are produced together with a candle-like smell. When heated in the absence of a flame the material will melt at ~170°C/338°F and decomposition will commence at about 300°C/572°F with the evolution of low molecular weight hydrocarbons. These can be ignited by a flame or radiant heat source and once started the material will continue to burn even if the ignition source is removed. Burning is accompanied by the formation of flaming molten droplets. In general, PP can be distinguished from other polyolefins by its hardness, density and high melting point. LDPE has a melting point range of 110°C to 125°C, LLDPE 115°C to 128°C, HDPE is 130°C to 135°C and PP is 160°C to 165°C.

The melting point of mPPH (147°C to 158°C/297°F to 316°F) is lower than conventional PPH (160°C to 165°C/320°F to 320°F). This is thought to be due to the occasional reversal of the propylene molecules when they add to the growing polymer chain. A reversal of the head-to-tail addition reduces **212** crystallinity, melting point and heat of fusion. The density of mPP-H is similar to conventional grades.

### 11. Coloring

PP is a semi-crystalline thermoplastic, but as the amount of the amorphous phase is not too dissimilar to the crystalline phase, PP is less opaque than HDPE. Often the natural color of the material is translucent to clear and this means that a wide color range is possible. By the use of nucleating agents (such as those based on dimethylbenzylidene sorbitol isomers) grades with improved clarity, and which cool faster, are possible. Such a material is available as pellets, in masterbatch form and works best at a melt temperature of between 220°C and 240°C.

Best coloring results are achieved by the use of fully compounded material, as dry colors and master batches can give rise to streaking, due to dispersion problems. When using various coloring systems, it is often recommended to try to match the particle size or granular shape of the polymer to that of the coloring system. Use powder color concentrates or dry colors with powder PP, and master batches with similar particle size to the plastic granules, when using granular feedstocks. This is not always the case with dry colorants, as the use of powdered PP with a dry colorant requires a very long mixing time (that is, up to 1 hr) to adequately disperse the dry colorant within the mix. Certain types of blue and green pigments, like a phthalocyanine type, can increase the degree of crystallinity in PP and increase the stiffness. However, it also reduces the impact strength and can cause distortion.

When PP is used for outdoor applications, it requires some form of UV stabilization. A well dispersed carbon black, at 2.5% concentration, is particularly good for both functions; coloring and weather protection. In general, opaque colors can also offer some protection to PP from the effects of weathering; however, they are not as resistant as black. Nickel-based UV stabilizers should not be used with pigments that contain traces of sulfur, as discoloration can occur. The most common group of pigments that react in this way are cadmium-based yellow, orange and red types. Use of the hindered amine-type light stabilizers (HALS) prevents such discoloration problems from occurring.

Various types of master batches (MB) are used with PP. These include PE-based, PP-based and the universal type. Levels greater than 3% of the PE-based type can lead to poor weld line strengths and delamination, due to poor dispersion, or inhomogeneity, of the PE in the PP. Typical concentration levels of 1% to 2% are recommended. Universal MB levels of 1% to 2% are also recommended, otherwise problems similar to the PE-based type occur. When using a high MFR grade of PP, a specially formulated PP MB may be required to ensure that the correct density of color is obtained. Such a MB is usually produced from a base PP polymer having the same MFR as the material being processed.

### 12. Materials Handling

PP resins are available in both powder and granular forms, which are usually stabilized to protect them against decomposition during processing. They will absorb ~0.02% water in 24 hrs at room temperature. This means that drying is not normally necessary. However, if the material has absorbed a small amount of moisture as a result of long storage, it is advisable to dry it in a hot air oven for 2 to 3 hrs at 80°C/176°F or in a desiccant dryer for 1 to 1.5 hrs at 90°C/194°F.

The use of gravity feed hoppers is suitable for granules. For powder feed, hopper agitation or crammer feeders are helpful to prevent blockages.

# SECTION 8: GUIDES FOR THE FOLLOWING MATERIALS

### 13. Screw and Barrel Design

The screw and barrel assembly should be between 20 and 33 diameters in length. The extruder should have an L/D ratio of 27:1 and a constant pitch of 1 D. The feed zone should be 8 D, the compression zone 10 D, and the metering zone 6 D. A mixing head of 3 D should be used. A compression ratio of about 3:1 should be sufficient and is obtained as shown below.

	Typical Screw Dimensions			
Screw diameter	2.5"/64 mm	3.5"/90 mm	4.5"/115 mm	
Feed Zone Depth (8D)	0.41"/10.4 mm	0.52"/13.2	0.58"/14.7 mm	
Compression Zone (10D)				
Metering Zone Depth (6D)	0.14"/3.56 mm	0.17"/4.32 mm	0.19"/4.83 mm	
Mixing Head (3D)				

For higher output, extruders with a grooved and cooled barrel insert in the feed section are used. These extruders can give 80% more throughput than a traditional machine. The barrel has an L/D ratio of about 30:1. Grooves, 0.16 in/4 mm deep, are spaced, at about every 1.25 in/31.8 mm, around the inside of the feed section of the barrel. It is important that the grooved section is kept cool. Decompression screws similar to those used in HDPE production have also been successfully employed.

### 14. Barrel and Die Temperatures

For pipe and blown film extrusion, melt temperatures in the range 200°C to 230°C/390°F to 445°F are required, while for cast film the temperature is usually in the range of 240°C to 260°C/465°F to 500°F. The table below gives a guide to set temperatures (°F/°C) required to provide the above process temperatures.

Zone	General Extrusion	Cast Film
Zone 1 Temperature	390/200	410/210
Zone 2 Temperature	430/220	430/220
Zone 3 Temperature	430/220	465/240
Adapter and Die	490/255	490/255

PP is more susceptible to oxidation than PE, as PP contains many tertiary carbon atoms. This means that at a temperature of 260°C/500°F the maximum residence time in the barrel should not exceed 5 to 6 minutes. At a temperature of 270°C/527°F the maximum residence time in the barrel should not exceed 2 to 3 minutes. One should not exceed a melt temperature of 275°C/527°F, if possible, since oxidation will cause the viscosity of PP to change rapidly. In general, as the melt temperature increases, the stiffness and impact strength of both PPH and PPCO will decrease. High melt temperatures and long residence times produce increases in flow rates and reduced toughness, caused by material breakdown.

The melting point of mPPH (147°C to 158°C/297°F to 316°F) is lower than conventional PPH (160°C to 165°C/320°F to 329°F) and this means lower melt temperatures are possible. This should be good for blow molding and thermoforming where the higher melt strength, resulting from low melt temperatures, is an advantage. The energy required for heating and cooling in processes such as extrusion and injection molding should also be reduced. Whether the speed of processing is significantly reduced is debatable, due to the fact that the rate of crystallization for mPP is comparatively slow. For extrusion, one should use a reversed temperature profile ((230°C/446°F) at the feed and 215°C/420°F in the metering zone) and a barrier screw. Because of the narrow MWD of mPP-H, extruder torque will be comparatively high, but as PP has a comparatively low viscosity, the torque is usually well within the extruder capabilities

# 214 15. Die Design and Construction

In pipe dies, spiral mandrel designs are increasingly being used, especially for large diameter pipes. Otherwise, dies based on the spider arm principle are employed. The land length should be 15 to 25 times the pipe wall thickness. Long lands are needed for high-speed production. If pressure sizing is utilized, the die diameter is about equal to the pipe diameter. When vacuum sizing is used the pipe is drawn down and the die diameter is up to 25% larger than the pipe diameter, with an even greater reduction in wall thickness.

Cast film and sheet dies are of the coathanger type, preferably with pushpull adjustment of the die lips. Entry angles to the land should be about 10° or less and the land length about 1in/25 mm. For film up to 0.01 in/0.25 mm, die gap widths of 0.02 in/0.5 mm are suitable. Tubular film dies can also be used to produce PP film.

### 16. Downstream Operations

If a melt temperature of  $240^{\circ}$ C/464°F is used, and if it is assumed that the extrudate must be cooled to  $20^{\circ}$ C/88°F, then the maximum amount of heat that needs to be removed for each gram of output is 645 Joules (J). Over the temperature range quoted, the specific heat is approximately 2930 J/kgK.

In the production of cast film, the PP melt is cooled on polished steel or chromium plated rolls through which a coolant is circulated. Low roll temperatures, produce film of greater clarity and toughness, due to the lower level of crystallinity in the product. Higher temperatures, however, will reduce blocking and increase film stiffness. Chill roll temperatures are usually in the range 10 to  $30^{\circ}$ C/50 to  $90^{\circ}$ F. The air knife, used to press the film against the chill roll, must be carefully set to prevent air bubbles occurring between the film and the chill roll. Too strong an air-flow will cause corrugation of the film. The air should be filtered to prevent dust from reaching the molten film. The film is wound on reels, at low tensions, as some post-extrusion crystallization and consequent shrinkage may occur, over a period of two weeks at room temperature. Film is not usually used during this time, which allows slip additives time to migrate to the film surface.

For high clarity blown film, PP is extruded downward and water at  $10^{\circ}$ C/50°F is used to cool the film by means of a weir. If air is used, a very hazy (crystalline) film results. In the production of weaving tapes, the film is slit with razor blades and then oriented by passing between two sets of rolls called Godet rolls. The second set of rolls has a surface speed between 5.5 and 8.5 times that of the first set, which produces the orientation. An air oven or hot plate, at 120°C to 180°C/250°F to 355°F, is placed between the two sets of rolls to enable orientation without breakage. PP film and sheet may also be biaxially oriented using a tenter frame to give orientation perpendicular to the machine direction.

### 17. Extrusion Capacity

As with most materials, the maximum extrusion capacity is not used in production. Some output is held in reserve. In the following table the maximum extruder output, using grooved barrels, is shown together with typical maximums for several processes.

Typic	al Max	cimum	Outputs i	in lb/hr (kg/hr)		
Extru	ıder Ca	pability	1	Sheet	Sheet	Pipe
(in)	(mm)	(lb/hr)	(kg/hr)	(0.3 in/7.6 mm)	(<0.04 in/1 mm)	)
1.5	38	160	72*			80 (36)
2.5	64	400	182*	190 (86)	300 (136) *	200 (91)
3.5	90	700	318*	370 (168)	570 (259) *	700 (318) *
4.5	115	960	436*	660 (300)	960 (436) *	960 (436) *
6.0	150	1700	773*	1400 (636) *	1700 (773) *	1250 (568)
8.0					2000 (909) *	
*					, ,	

\*using a grooved barrel

Compared to conventional PP, mPP has a narrower molecular weight distribution (Mw/Mn = ~2). To get such a narrow MWD, a conventional PP must be modified (post-reactor modified), because its Mw/Mn, as polymerized, is in the range 3 to 6. A narrow MWD results in reduced shear sensitivity of the polymer melt and a higher achievable extrusion rate, since the extrusion process is less susceptible to surging. The molten polymer also has reduced melt elasticity and elongational viscosity, which is seen in reduced die swell and improved melt draw down. Thus, in film and fiber applications, the melt can be drawn down relatively easily into thinner sections. As mPP is free of low molecular weight, volatile, polymer components, spinning smoke is virtually eliminated in such processes. It should be noted that by catalyst modification, the MWD and the composition may be tailored (broadened) to meet product needs, if required.

### 18. Starting Up

PP presents no special problems, though it is less thermally stable than the polyethylenes and is extruded at higher temperatures. Extruders and dies should not, therefore, be left heated at operating temperatures for long periods before starting to run, if there is PP in the system.

### 19. Shutting Down

At the processing temperatures used in extrusion, PP is relatively stable and it is not necessary to purge with other materials. For die cleaning, the PP can be burned off, provided exhaust fans remove the gases produced. Do not use copper tools (such as copper cleaning pads) when PP is to be processed, as traces of copper accelerate the degradation of PP.

### 20. Reprocessing

When this material is reclaimed, up to 15% can be blended into the virgin material. If the material has been recycled too many times then it will degrade. This is shown by a decrease in MFR, an increase in brittleness, excessive odor from the products, and signs of decomposition, such as burning or streaking. mPP is more chemically stable than conventional PP. This results in less chemical breakdown during processing, so that MFR (molecular weight) remains more constant. For this reason mPP can be recycled more often, or used at higher feedstock levels, than conventional PP.

### 21. Finishing

PP may not be joined to itself by solvents, as there is no known solvent at room temperature. Although it can be heat welded, its high melting point makes this a difficult operation. Sometimes PP is coextruded with LDPE, which acts as a heat sealable layer. For printing, ink adhesion is improved by flame or corona discharge treatments. Recently, primers have been developed for preparing surfaces for printing. These are also useful if the PP is to be vacuum metallized.

PP can be readily machined with cutting tools used for metals. Use sharp tools, high cutting speeds and low feed rates. The edges of the cutting tools must be sharp in order to achieve a good surface finish. Use plenty of cutting fluid to cool the cutting surface, to prevent heat build up when machining. Typical operations that can be readily performed are milling, drilling, tapping, turning, sawing and planing. PP cannot be readily surface ground and polished. Specialized techniques are necessary to produce machined surfaces to a very high accuracy and quality.

### 22. Other Comments

Internal antistatic agents used for polyolefins (polyethylene and polypropylene) include glycol alkyl esters and betaine-type materials.

### 23. Typical Products

With its combination of light weight, toughness, high temperature resistance, rigidity, and excellent resistance to chemical attack, PP is suitable for a wide

216 range of components. However, despite this materials high stiffness, it is subject to creep and should not be used in applications where it will be continuously loaded under high stress. By taking advantage of the integral hinge effect, very long hinges can be continuously produced. PP-HO produces hinges that are slightly superior to PPCO. Easy flow, but not filled, grades are used for this application.

Because it is chemically inert, it possesses high rigidity, and its resistance to reasonably high temperatures, PP is used in pipe manufacture for applications such as under-floor heating, high-pressure pipe, chemical transport, and ball-point pen refills. PP fire retardant grades are used for domestic water pipes and fittings.

Because of its good water barrier properties, PP film is used for packaging bread and other goods which must be kept dry, such as potato chips and cigarettes. When biaxially oriented, these films have good toughness and are clear. There is a large market for PP tapes that can be used for carpet backing or converted into twine.

Glass reinforced PP is now being extruded into profiles and sheet. It has higher rigidity and better thermal stability than un-reinforced grades. Extruder wear is much higher with these grades. Foamed or expanded PP is increasingly being used for many applications due to its lightness, low cost and good structural properties.

The density of mPP is similar to conventional PP (0.9 g/cm<sup>3</sup>). It is used in place of post-reactor, peroxide-cracked polypropylene (CR PP) and for medical grades of PP, as the mPP has lower extractables. The mPP costs more than conventional PP, but can be processed on similar equipment.

Other metallocene polyolefins (mPO) may be used for PP modification in place of EPDM and/or EPR rubbers. It is claimed, that use of a polyolefin elastomer (POE) or a polyolefin plastomer (POP) as a modifier, results in a superior stiffness/toughness balance, high clarity, ease of compounding, and ease of handling (because they are available in pellet form). When the POE and POP are based on an ethylene-butene copolymer, the mPO modified PP has higher flow, and, reduced shrinkage and warpage. The resultant shrinkage may be similar to, or lower than, that of ABS, and the coefficient of thermal expansion (CLTE) may be as low as  $2.4 \times 10^5$  in/in. A mPP modifier may also be used in conjunction with nucleated, random PP, to produce a clear material with good low temperature toughness.

### 24. Yield

The figures below assume a density of 0.91 g/cm $^{\rm s}$ . The actual densities vary somewhat according to the level of crystallinity

- Sheet of 0.1 in thickness has a surface area of 2.12 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.55 sq m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 106 sq ft/lb weight
- · Film of 50 microns thickness has a surface area of 22.0 sq m/kg
- Tube of 3/8 in ID and 1/16 in wail thickness has a length of 29.6 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 20.3 m/kg
- Pipe of 0.1 in wall thickness and 4 in IinD has a length of 1.733 ft/lb weight
- Pipe of 2 mm wall thickness and 100 mm ID has a length of 1.52 m/kg

# ≻ PMMA

1. Common Name

2. Abbreviation(s)

Polymethyl methacrylate PMMA

3. Alternative Names

Acrylic: poly[1-(methoxycarbonyl)-1-methylethylene]: polymethylmethacrylate.

4. Some Suppliers	5. Trade Names or Trade Marks
Agomer	Degalan G
Atohaas	Altuglas
BASF	Lucryl G
Chi Mei Corp	Acryrex
Cyro Industries	Acrylite Cyrolite
Elf Atochem	Plexiglas
Glasflex	Glasflex
Goodfellow	PMMA
ICI Acrylics	Perspex, Lucite
Swedlow	Acrive

### 6. Material Properties

Acrylic plastics are based on polymers made with acrylic acid, or a structural derivative of acrylic acid. That is, the polymers are based on acrylic monomers that may be represented as  $CH_2$ =CXCOOY:

where: X = Y = H: It is acrylic acid X = H and  $Y = CH_3$ : It is methyl acrylate  $X = CH_3$  and  $Y = CH_3$ : It is methyl methacrylate X = H and Y = CN: It is acrylonitrile X = H and  $Y = C_2H_5$ : It is ethyl acrylate

Such monomers readily polymerize so that a wide range of homopolymers and copolymers is possible. Many of these materials are noted for their optical properties. The best known member of this group is polymethyl methacrylate (PMMA).

When methyl methacrylate (MMA) is polymerized an optically clear plastics material, PMMA is obtained. It is a water white (or crystal clear), amorphous plastic with a glass transition temperature (Tg) of ~100°C/212°F. At room temperature it is a hard and rigid material that maintains its clarity, even in thick sections, even after long exposures to outside atmospheres (i.e., it has excellent UV resistance). Only surface scratching, caused by airborne dust or by cleaning, limits the clarity during outdoor exposure. The clarity can be approximately 92% and so this material is widely used as a glass replacement. It is significantly lighter than glass, easier to shape and easier to machine. PMMA is considered to be one of the hardest thermoplastics. The hardness of PMMA is about the same as that of aluminum. To retain the clarity of this material, when processed, it is important to maintain the surface finish to a very high standard. A high standard of storage and/or drying, or a vented barrel is therefore required.

Acrylic components are rigid, as the material is hard and stiff. Components are dimensionally stable, odorless, resistant to many common chemicals, and easy to decorate. The material has low moisture absorption, high gloss, excellent UV resistance, low smoke emission when burned, and good arc and track resistance. However, the material has poor solvent resistance, burns easily and fiercely, is brittle, is notch sensitive, has poor fatigue resistance and a low continuous use temperature (approximately 50°C/120°F).

It is possible to obtain grades that are softer and tougher than the standard grades. These are referred to as high impact PMMA (PMMAHI) or as toughened PMMA (PMMAT). Such an impact resistant PMMA polymer is made by the incorporation of a rubbery material into a PMMA polymer. Therefore, it is a two-phase material. The rubber phase is based on a styrene-modified acrylic ester (e.g., polybutyl acrylate), whose refractive index is the same as PMMA. This impact resistant molding compound can be blended with standard PMMA if required. It should be noted that the notched impact strength is only about twice that of a standard grade (at 0.04 kJ/m). It has better environmental stress cracking (ESC) resistance than the standard grades, but lower

218 tensile strength, stiffness, and hardness. Such materials are almost as transparent and UV resistant as the basic grades, but demonstrate more haze. The better craze resistance is useful when products come into contact with aqueous detergents or soap solutions. The heat distortion temperatures (HDT) of such materials are slightly lower than basic grades. For example, the HDT, at 0.45 Mpa, for high impact PMMA is 90°C/194°F and at 1.8 MPa it is 75°C/ E16°F, while for a standard material the HDT, at 0.45 Mpa, is 103°C/218°F and at 1.8 MPa 95°C/203°F. The Vicat softening point (VSP) of the impact modified material is slightly lower than that of the basic grades, by approximately 5°C/41°F.

### 7. Flow Properties

PMMA is a comparatively stiff flow material. Its flow is stiffer than PS or ABS. PMMAHI is easier flowing than standard PMMA.

At low melt temperatures and shear rates, PMMA viscosities are higher than those of most extrudable plastics. The viscosity is relatively sensitive to shear rate and melt temperature. Higher viscosity grades are used for sheet extrusion and lower viscosity grades for pipe and profile. The viscosity of one of the latter grades is shown below.

Shear Rate (s <sup>-1</sup> )			
	(204°C/400°F)	(221°C/430°F)	(238°C/460°F)
20	15100	12100	9720
40	8330	6680	5350
100	3790	3040	2440
200	2090	1670	1340
400	1150	920	740

### 8. Resistant to:

PMMA is resistant to dilute acids and alkalis; concentrated alkalis and concentrated hydrochloric acid; aqueous solutions of salts and oxidizing agents, amines, fats, oils and aliphatic hydrocarbons (like white spirit and paraffin), dilute alcohols and detergents. It is also resistant to high molecular weight esters, i.e. to those with more than 10 carbon atoms in the molecule. It shows good resistance to gases such as carbon dioxide, carbon monoxide, dry chlorine and to dry sulfur dioxide.

PMMA is transparent to UV light and thus, is more resistant to UV attack than most other thermoplastic materials. A UV absorber (for instance 2-(2'-hydroxy-5'-methyl-phenyl)benzotriazole) may, however, be added to PMMA in order to filter out such light and protect another material located behind the PMMA. Products that demand very good long term UV stability are stabilized with UV absorbers such as 2,4-dihydroxy benzophenone, phenyl salicylate, methyl salicylate or, resorcinol monobenzoate or stilbene

### 9. Not resistant to:

PMMA is not resistant to concentrated, oxidizing acids (such as nitric and sulfuric) and alcoholic alkalis, which cause decomposition. The resistance to most organic solvents is poor. For example, PMMA is soluble in most aromatic hydrocarbons, chlorinated hydrocarbons (such as toluene and chloroform), low molecular weight esters (like ethyl acetate), phenols and ketones. Although it is not attacked by alcohol alone, alcohol in association with carbon tetrachloride and ether will cause swelling. PMMA is plasticized by ester-type materials such as tritolyl phosphate and dibutyl phthalate and is swollen by phenols. Ethyl acetate can be used to detect strain. It has poor resistance to gases such as hydrogen sulphide, wet chlorine and to wet sulfur dioxide.

### 10. Material Detection or Identification

With a density of 1.18 g/cm<sup>3</sup>, PMMA (solid, non-filled material) will sink in water and float in a saturated magnesium chloride solution. The material is an amorphous, thermoplastic material whose natural color is a very clear, waterwhite, so a very wide range of both clear and transparent colors is possible. It has a refractive index of approximately 1.49, the Abbe number is 58, and it can transmit about 92% of visible light. The shrinkage of PMMA materials is approximately 0.2% to 1.0%.

When this material is heated in a flame, it will ignite easily and continue to burn very readily and fiercely, with an almost smoke-free flame. There is little residue after burning, and the material does not smolder on flame extinction. The flame is blue, tipped with yellow, and a sweet fruity odor is emitted. PMMA will continue to burn, with crackling noises, after the source of ignition is removed. Frothing may occur when the flame is extinguished. When heated in the absence of a flame, it will soften at approximately 100°C/212°F and decompose at temperatures above 300°C/572°F to give vapors, which are mainly the monomer methyl methacrylate (MMA). Vacuum distillation occurs at temperatures above 500°C/932°F and has been used to reclaim this material.

PMMA cannot be cut easily with a knife, but is relatively easy to scratch. This material will dissolve in solvents such as toluene and chloroform, but will not dissolve in aliphatic hydrocarbons (paraffin and white spirits). It will swell in alcohols and phenols and is plasticized by some ester-type plasticizers, such as tritolyl phosphate and dibutyl phthalate.

### 11. Coloring

Since the natural color of the material is clear, a wide range colors is possible. This includes both transparent and opaque colors. It is sold in both compounded colors and as natural material for coloring on the machine, by techniques such as dry coloring, masterbatching, and liquid coloring.

PMMA may be available as granules or spherical beads. The latter type is considered more suitable for dry coloring due to the similarity in particle size of polymer and dry colorant. However, the granules provide a more "grinding out" (mixing) action, both in the tumble mixer and in the feed zone of the extruder. Tumble blending is used to carry out the addition of dry colorant. The procedure for addition is important to ensuring the optimum dispersion. To obtain a uniform dispersion of colorant in the mix, the drum should be filled with half of the polymer charge, then the dry colorant should be added, followed by the rest of the polymer. The total charge should only occupy half, to two thirds, of the drum's capacity. The speed of rotation of the drum is important to the even distribution of the colorant throughout the polymer. Long mixing times can reduce the particle size of some colorants, giving adverse effects with respect to its performance in the final product (e.g., translucency). A typical mixing time is 10 minutes. When dry coloring granular material, the use of a binder or wetting agent is recommended and the coloring procedure is similar to GPPS. Typical colorant concentrations range from 0.2%-1.0%.

Master batches are widely used for opaque or dense colors and where color matching is required. Universal type master batches are suitable for many coloring requirements, however, they are not used for tinted shades. The mixing time for masterbatch incorporation, and level of usage, is similar to that required for ABS. Liquid coloring is suitable for both opaque colors and tints, although the liquid carriers have to be tailored accordingly.

### 12. Materials Handling

To maintain the clarity of this material, it is important to maintain the surface finish of the products to a very high standard. For best results, the resin should be dry before extrusion. High water absorption can occur on prolonged contact with a damp atmosphere, so strict control must be exercised over storage conditions. A very strict Fi-Fo policy is required to assure that materi220 al is not in store for more than 6 months. If left exposed to the atmosphere, PMMA will take up between 0.15% and 0.7% moisture. The material should remain in a pre-drier, at a temperature of 85°C/15°F for a period of 4 hours. Higher temperatures can be used when drying in an air circulation oven, but agitation of the granules is required to prevent 'caking', or fusing, of the granules.

If the drier is in line, its capacity can be calculated from the bulk density of the material and the output rate. Volume =  $4 \times \text{output}$  rate (lb/h) /bulk density (lb/cu ft).

Feed can consist of pelletized material, or a mixture of pellets and polymer grains (not pre-extruded) with up to 50% of grains. Gravity feed hoppers are usually adequate, but they should be covered.

### 13. Screw and Barrel Design

Barrels should be vented and have an L/D ratio of between 27 and 33. The barrel should be lined with a wear resistant material such as Xaloy. To ensure trouble free operation, the highest possible vacuum should be maintained in the vent zone of the screw using a vacuum pump with a relatively high capacity. Suggested screw configurations are shown below.

Extruder Diameter	3.5 in/90 mm	4.5 in/115 mm
Feed zone depth (4 D)	0.56 in/14.2 mm	0.62 in/15.8 mm
Compression (8 D)		
1st metering depth (5 D)	0.18 in/4.57 mm	0.20 in/5.08 mm
Decompression (2 D)		
Vent zone depth (4 D)	0.68 in/17.27 mm	0.75 in/19.05 mm
Compression (4 D)		
2nd metering depth (3 D)	0.31 in/7.87 mm	0.34 in/8.64 mm

### 14. Barrel and Die Temperatures

The extruder temperature settings depend on the product. The table below shows values for three different types of extrudate (Note: If rod is being manufactured, it is often extruded very slowly, at very low melt temperatures, to avoid voids in the rod).

### Typical Extrusion Temperatures for PMMA Products

### (Temperatures are in °F with centigrade equivalents in brackets.)

Zone	Sheet	Tube	Slit Tube
Zone 1	375 (190)	375 (190)	375 (190)
Zone 2	430 (220)	400 (205)	400 (205)
Zone 3	410 (210)	410 (210)	390 (200)
Zone 4	430 (220)	410 (210)	390 (200)
Adapter	445 (230)	390 (200)	390 (200)
Die	445 (230)	390 (200)	390 (200)

### 15. Die Design and Construction

Sheet dies are of the coathanger type. Ideally, the detailed coathanger shape should be designed from considerations of the melt rheology of the grade of material to be used and for a particular output rate and melt temperature. This will give the best quality of output in terms of even flow, and freedom from striations, as well as temperature uniformity, which should be to better than 10°F. Dies are fitted with a choker bar and adjustable lips. The die gap/sheet thickness ratio should be kept between 1.2 and 2.0 with die land lengths as follows:

Sheet Thickness	Land Length
<:0.1 in/2.54 mm	2.5 in/64 mm
0.1 to 0.2 in/2.54 to 5.08 mm	3.5 in/82 mm
0.2 to 0.4 in/5.08 to 10.16 mm	4.5 in/115 mm

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Dies can be up to 12 ft/3.66 m wide. They should be made from tool steel and hard chrome plated. The finish of the die lips is critical, as any damage will leave marks on the sheet. The die should be split into separately controlled heating zones, spaced at no more than 1 ft/0.3 m intervals across the width of the die and heated by means of cartridge heaters rather than by surface plates. Additionally, there should be separate controls for each side of the die.

Acrylic tubing is formed using dies based on the spider arm design. To reduce the risk of web lines, formed when the melt fails to knit together completely after the spiders, there must be adequate compression after the spider and land lengths should be at least 20 times the wall thickness. Lowering output will reduce this defect. Tube dies are also used to produce profile for acrylic fluorescent light fittings by slitting the tube with a knife. Profile dies are also used with PMMA.

Acrylic melt shows a tendency to stick to the die, especially at high temperatures and throughput rates. This sticking is reduced by chrome plating the flow surfaces or by using steels with a high chrome content.

### 16. Downstream Operations

Sheet is normally cooled on a three-roll stack, with highly polished chrome surfaced rolls kept at a uniform temperature across their width. For a bottom fed stack, roll temperatures are typically:

- Bottom Roll = 175°F/80°C
- Middle Roll = 200°F/93°C
- Top Roll = 220°F/104°C

If a melt temperature of  $230^{\circ}$ C/446°F is used, and it is assumed that this melt must be cooled to that of the bottom roll ( $80^{\circ}$ C/175°F), then the maximum amount of heat that needs to be removed for each gram of output is 313 Joules (J). Over the temperature range stated, the specific heat is approximately 1900 J/kgK.

The sheet is pulled from the stack by rubber coated nip rolls. To prevent too much orientation in the sheet, the tension between the polishing rolls and nip rolls should be low. The melt pool, before the nip in the polishing roll, must not be too large and the roll speed must not fall too low. Sheet, cooled without use of polishing rolls, will have an inferior surface finish and will draw in sideways as it is pulled from the clips. When cutting sheet to size, its temperature must be controlled to allow for any subsequent shrinkage. Sheet is usually protected by covering it with LDPE film. The residual heat in the sheet causes the film to stick slightly to the surface of the sheet. The polyethylene film can remain on the sheet while it is thermoformed and stay as a protective layer until final end use.

Lighting fittings are made by post die forming of a tubular extrudate, which is slit at its topmost point by a blade fitted the die face. The soft extrudate is then shaped by passing it through a series of internal and external metal forming plates. These plates are 0.25 in/6.35 mm thick and made of steel or brass. Air is blown on the surfaces of the extrudate from a series of holes in a pipe bent to the shape of the product.

In tube production both pressure and vacuum sizing methods are employed.

### 17. Extrusion Capacity

The highest outputs are achieved when producing sheet material. Typical figures are shown in the table below.

222	Typical	Outputs for Pl	MMA Sheet Ext	rusion		
	Extrude	r Diameter	Maximum S	Sheet Width	Maxim	um Output
	(in)	(mm)	(in)	(mm)	(lb/hr)	(kg/hr)
	3.5	90	60	1500	550	250
	4.5	115	78	2000	900	409
	6.0	150	100	2500	1200	545
	8.0	200	140	3555	1900	864

### 18. Starting Up

It is important to prevent premature melting of any residual PMMA on the feed section of the screw. This can be prevented during warm up if the screw is fitted with water cooling in the feed section. Ensure there is water cooling on the feed throat and keep the Zone 1 temperature at 200°F/93°C until extrusion starts. The temperature should then be raised to the operating setting. It is important to start at low screw speed as the PMMA melt is very viscous. Check the pressure readings as the screw speed is gradually raised. In sheet production, once the sheet is feeding through the polishing rolls, the thickness distribution can be evened out. If there are thin areas they will appear as dull lakes, where they fail to contact the polishing roll. Thick areas often give a chevron pattern. Dies are adjusted using the push-pull bolts and by changing zone temperatures. Results from the latter adjustment can take up to 30 minutes to be effective and temperature settings should be changed slowly.

### 19. Shutting Down

It is best to purge PMMA out with PP or HDPE when shutting down. If the extruder is to be stopped for a short while, drop the temperature of the feed zone during the pause in production. Because of the excellent clarity of this material, the smallest trace of contamination is immediately visible.

### 20. Reprocessing

When this material is reclaimed, up to 25% may be blended into the virgin material (all regrind must be dried). However, for the best quality clear sheet, regrind should not be used. As the proportion of regrind increases, feeding from a gravity hopper will become more uncertain. When regranulating acrylic, strict housekeeping is essential to prevent any contact with other polymers that may cause contamination. Also when using regrind, the mixing of different batches or grades should not take place, as the optical properties of the resultant products may become greatly reduced.

### 21. Finishing

PMMA may be joined to itself using solvents such as chloroform or by using solutions of PMMA in methylene chloride. One can also use solutions of PMMA in MMA. These cements are set by the addition of a catalyst or hardener. If cements are applied from metal tubes or containers, then it must be remembered that the material of construction will affect setting. Copper and brass inhibit setting, whereas lead speeds it up. When cementing PMMA, be careful that a build up of vapors does not occur. These are inflammable, toxic and can cause stress cracking of components. PMMA is commonly welded using techniques such as ultrasonic welding, hot plate and friction.

Machining of this plastic can give satisfactory results, but one should take great care not to cause scratching. A high polish may be put on cut edges by buffing and polishing. Small surface blemishes or scratches can be removed by manually polishing with 'Bluebell' metal polish and a soft cloth. Silk screen printing, vacuum metallization, and hot stamping can be used to decorate components.

### 22. Other Comments

When thermoforming PMMA sheet, zoned heating on both sides of the sheet is preferred, with higher temperatures on the outer zones. Aluminum is the optimum mold material: it must have a very smooth, and blemish free sur-

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face. It is relatively easy to scratch this material, but such scratches can be easily polished out. Because of the ease of scratching, one should be very careful when handling the material and keep it covered as long as possible during any handling or machining operation. Only clean the products with liquid cleaners. Even fine dust will cause scratching if rubbed in.

PMMA can become electrostatically charged during processing and readily attracts dust as a result. To remove the dust, one may wipe the components with a soft cloth. However, this may only aggravate the problem and result in more dust accumulating on the surfaces. To obtain dust-free components an antistatic treatment of the surfaces is required.

### 23. Typical Products

PMMA possesses excellent light fastness and resistance to weathering. It is also hard, rigid, transparent, and can have a very good gloss. It is also not subject to microbiological attack. These properties dictate the applications of this class of material. The ease of decoration of this type of material should not be forgotten as such decoration can lead to an enhanced appearance of PMMA components.

It is widely used in the building industry as a roofing material in place of glass. Present day, optical production methods can give clear or tinted sheet that is of high enough quality to replace cast PMMA or glass. It is not as scratch resistant as glass, but offers the advantage of being able to be cut or sawn. It also is used to make bath and shower trays by sheet forming techniques. In both these applications the light weight of this plastics material is a tremendous advantage as it makes installation easier. The high coefficient of thermal expansion of PMMA and its ease of burning should, however, be noted. PMMA is now available in double-walled, extruded profiles whose thermal conductivity is very low. When used as a glazing material this reduces heat losses and cuts down on condensation. Its weatherability makes it useful for outdoor light fittings.

### 24. Yield

- Sheet of 0.1 in thickness has a surface area of 1.63 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.42 sq m/kg weight
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 22.8 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 15.6 m/kg
- Pipe of 0.1 in thickness and 4 in ID has a length of 1.34 ft/lb weight
- Pipe of 2 mm thickness and 100 mm ID has a length of 1.17 m/kg

### PPVC

- 1. Common Name
- 2. Abbreviation(s)

### 3. Alternative Names

Plasticized PVC or Plasticised PVC PPVC; PVC-P

Plasticized polyvinyl chloride: plasticised polyvinyl chloride: soft PVC: flexible PVC. PVC may also be referred to as poly-(vinyl chloride) or as poly(monochloroethylene). PPVC is based on a vinyl plastic material. The term vinyl plastics is often taken to mean vinyl chloride plastics. These are plastic materials that are based on homopolymers of vinyl chloride orcopolymers of vinyl chloride with other monomers, the vinyl chloride being in the greatest abundance by mass.

### 4. Some Suppliers

Alpha Gary A Schulman BASF Colorite Plastics

## 5. Trade Names or Trade Marks Alpha

Polyvin Vinoflex Unichem

# 4. Some Suppliers

Condea Vista Flf Atochem Elf Atochem El Paso PVC EVC Evode Plastics Ltd. Formosa Plastics Hydro Polymers Hydro Polymers Huls Huls Hvdro Polvmers Keysor-Century LVM LVM Novatec **OxvVinvIs** Piltec Chemicals Rimtec Solvay **Syneraistics** Teknor Apex Wacker Chemicals

### 5. Trade Names or Trade Marks

Condea Vista Nakan Lacovyl El Paso Sicron Garv PVC Formolon Peviikon (PVCE) Norvinyl (PVCS) Trosiplast 3 Vestolit Hi-Vin Kevsor Marvylflo PVC Marvylex TPE-PVC Novablend Geon Temprene TPE (PVC/NBR) Rintec/Kohinor Benvic **Syneraistics** Teknor Apex/Ultra Vinnolit

PPVC plastics may be purchased as compound (which means that it has been melt compounded), it may be blended in a high speed mixer and then fed to the extruder, or it may be blended, compounded and extruded inhouse. For this reason the material may be known by the resin or plastic supplier's name or it may be known by the name of a compound. In general, processing of PVC compounds gives the best properties, but the processing of dry blends can offer cost advantages.

### 6. Material Properties

Vinyl chloride, also known as vinyl chloride monomer (VCM), may be represented as  $CH_2$ =CHCI. When VCM is polymerized, without another monomer present, a homopolymer is produced, which is commonly known as polyvinyl chloride (PVC). As VCM is a carcinogen, the level of monomer left in the plastic material must be kept very low (e.g., <1 part per million (<1 ppm) of monomer in the polymer). Vinyl chloride homopolymers are the most widely used type of PVC plastics used at the present time. They are made by emulsion (PVC-E), suspension (PVC-S) and mass or bulk (PVC-M) polymerization processes. Plasticized PVC (PPVC) is usually based on PVC-S or PVC-M. PVC-E is widely used to make PVC pastes (also known as plastisols), and for unplasticized PVC (UPVC). It is used as UPVC since the PVC particles contain a large amount of well-dispersed surfactants that aid in processing.

The discovery that polyvinyl chloride (PVC) polymer can be converted to an elastomeric type material, by the addition of low molecular weight, liquid plasticizers was made in the 1920s, when attempts were made to dissolve the polymer. Polymeric plasticizers (that is, plasticizers which are themselves polymers) were used approximately 10 years later. The use of elastomers or rubbers, as property modifiers, was introduced shortly afterwards. At that time the preferred elastomers were copolymers of acrylonitrile and 1,3 butadiene, known as nitrile elastomers (NBR). Such materials are still sometimes used at levels of up to approximately 33 parts per hundred (phr).

Plasticizers are added to PVC compounds to produce flexibility and softness although, originally they were added to ease processing. As there are

many different plasticizers which may be used in different amounts or combinations, a wide range of plasticized compounds is possible. Such compounds will differ not only in flexibility and softness, but also in other properties such as tensile strength, resilience and ease of flow. The plasticizers most commonly employed are high boiling point esters of  $C_{\text{s-10}}$  alcohols, such as phthalates, phosphates and sebacates. Examples of common plasticizers include dioctyl phthalate (DOP or 2-ethylhexyl phthalate), di-iso-octyl phthalates (DIOP) and dialphanyl phthalate (DAP). The term 'plasticized' means that there is plasticizer present in the material, but there may be other additives mixed in with the PVC resin as well.

Additives are essential to PVC technology, since without them PVC cannot be processed. In addition, some additives enhance the properties of the base PVC and permit its use in a wide range of applications. Examples of additives commonly used include heat stabilizers, lubricants, processing aids, fillers and anti-static agents. The use of such additives enables PPVC to be processed by methods similar to other thermoplastic materials. When the ingredients of the PVC formulation are blended together (for example, by high speed mixing), the porosity of the PVC particles must be high enough to permit rapid absorption of the plasticizer during blending to produce a dry, freeflowing blend. This blend is then melt compounded. In order to realize the optimum properties of this material, it is necessary to put in just enough work and heat, in this processing step, for the blended material to be fully gelled or fused, but not so much that it is degraded. Decomposition can be easily caused by over-heating or by excessive shear. Once decomposition starts, it can spread very rapidly, because one of the products of decomposition, hydrochloric acid (HCl), will catalyze further degradation. Since HCl is an acid, it also readily attacks metals, such as steel, and causes pitting and corrosion. Its effects on human beings are very harmful. Control over PVC mixing and compounding stages is very important, since the level of mixing affects material flow and end-use properties.

Despite its heat stability problem, PPVC is widely used because an extremely broad range of cheap, tough, flexible and easy to process compounds may be produced. The material is incredibly versatile. Elongation at break and impact strength increase with increasing plasticizer addition. However, the inherent flame retardancy of PVC is often reduced and properties may change over time if plasticizer is lost from the compound (the plasticizer lost from PPVC may cause environmental stress cracking (ESC) of other amorphous polymers). The chemical resistance is good, and, when correctly formulated, transparent components are readily obtained. This requires that appropriate stabilizers be used, including tin stabilizers and calcium/zinc complexes.

PVC may be made softer by 'internal plasticization', which means that a second monomer is introduced during manufacture. Such copolymers are softer, easier flowing and more readily fabricated than homopolymers, but they are usually more expensive.

### 7. Flow Properties

The ease of flow of a PVC polymer depends on the molecular weight of the resin, which is characterized for PVC by the K value. The higher the K number, the higher the molecular weight and the more stiff the flow. For a particular K value the ease of flow may be dramatically altered by the use of additives such as lubricants or plasticizer. Since PPVC contains plasticizer, which increases flow, they have relatively high K values (for example, DIN K values between, approximately, 65 and 70). While plasticizer reduces the compound viscosity, the addition of fillers will cause it to increase.

A K value of about 70 corresponds to a viscosity number (DIN 53726) of 124 (Note: The inherent viscosity (ASTM), often given for PVC, is approximately 1/100 of the viscosity number). For cable coating, hose, and flexible profiles (where the product is to be subjected to a wide range of temperatures or particularly good mechanical properties are required) resins with K values up to 91 (viscosity number 234) are used. The table below shows the viscosities for a fairly stiff compound designed for cable coating.

### Viscosities of a PPVC Cable Compound

Shear Rate (s <sup>-1</sup> )		Viscosity (Pa-s)		
	(350°F/177°C)	(375°F/190°C)	(400°F/204°C)	
20	2340	1690	1190	
40	1450	1050	740	
100	770	560	390	
200	480	340	240	
400	300	210	150	

### 8. Resistant To:

PPVC compounds have good UV resistance and reasonable chemical resistance, but not as good as UPVC. They are resistant to water and salt solutions. Dilute acids and alkalis have little effect at room temperature, but at elevated temperatures some hydrolysis and extraction of the plasticizer may occur. Concentrated acids and alkalis slowly hydrolyze plasticizers when cold and more rapidly when heated. Most organic liquids will extract the plasticizers and cause the compound to harden. Some organic liquids (e.g., aromatic and chlorinated hydrocarbons, aliphatic and aromatic nitro compounds, ketones, aliphatic and cyclic ethers) will also cause softening. The effect of stabilizers. lubricants and pigments on the chemical resistance of PVC compounds is less marked than the effect of plasticizers and copolymers. The type and amount of plasticizer or copolymer will affect the chemical resistance. To improve chemical resistance one should replace a monomeric plasticizer, such as dioctyl phthalate (DOP), with a polymeric type such as polypropylene adipate (PPA) or polypropylene sebacate (PPS). PPA is useful for oil and fuel contact and PPS resist soaps and detergents. PPVC is less permeable than PE; however, the permeability increases with plasticizer content.

Chemical resistance and weatherability may be good, but they are made worse by the addition of impact modifiers. Weathering resistance is improved by the use of UV stabilizers and/or carbon black. Homopolymers of PVC are more resistant to chemical attack than copolymers.

### 9. Not Resistant To:

PPVC is not resistant to acids, alkalis and oxidizing agents. These include concentrated oxidizing acids such as sulfuric, nitric and chromic acids, which cause decomposition. The rate of decomposition may be accelerated in the presence metals like zinc and iron. Bromine and fluorine attack it even at room temperature.

PPVC is more readily attacked by solvents, such as tetrahydrofuran, cyclohexanone, methyl ethyl ketone (MEK), dimethylformamide, than is UPVC. It is unsuitable for use in contact with aromatic hydrocarbons, chlorinated hydrocarbons, ketones, esters, cyclic ethers, nitro-compounds, acetic anhydride, aniline and acetone. These solvents penetrate the PVC and cause marked swelling. Bacteria may attack some plasticizers. To minimize this attack bactericides and/or plasticizers based on linear esters (those based on straight chain alcohols) are used. Plasticizers will be lost by exposure to high temperatures, water, and solvents, but a correct choice of the plasticizer used will help to minimize this loss.

Although pure PVC does not absorb light above a wavelength of approximately 220 nm, commercial materials are degraded by UV light. They

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contain impurity groups or structural features that contribute towards degradation. Heat stabilizers (such as phosphites) do help with light stabilization, but for the best results one should use a UV absorber. A simple, low molecular weight HALS stabilizer may have little or no effect on light stability.

PPVC has poor heat resistance and is more flammable than UPVC. **10. Material Detection or Identification** 

PVC is an amorphous thermoplastic material that does not exhibit a sharp melting point. It has a glass transition temperature of 82°C; however, addition of plasticizer will lower this value. With a density of approximately 1.4 g/cm<sup>3</sup>, PVC (solid, non-filled, non-plasticized material) will sink in both water and in a saturated solution of magnesium chloride. The addition of plasticizer will lower the density (they have a density of about 1 g/cm<sup>3</sup>), while the addition of inorganic fillers will raise the density (fillers generally have a density greater than 2.5 g/cm<sup>3</sup>). The density of commercial compounds may range from about 1.15 to 1.35 g/cm<sup>3</sup>. These compounds will float in a saturated magnesium chloride solution and sink in water. The presence of inorganic fillers can be shown by burning off the organic portion of the compound, which is mainly the PVC and the plasticizer. Stabilizers, particularly the metal compounds, are also partially responsible for an increase in the density. Shrinkage is of the order of 1.0% to 2.0%. Shrinkage variations are due primarily to differences in the hardness value (plasticizer level) of the compound.

The natural color of the unfilled material is clear when it is stabilized with an appropriate stabilizer, such as a barium cadmium or a tin stabilizer. If the material is stabilized with lead stabilizers and/or filled, the natural color is usually off-white. In either case a wide color range is possible.

The behavior of PVC, when it is heated in a flame, depends on the compound composition, especially the amount and type of plasticizer. Most plasticizers burn easily and their presence often increases the flammability of a compound. However, some plasticizers are naturally flame resistant (the phosphates, trixylyl phosphate or TXP) and compounds based on them will not burn easily. Because of their high price, phosphates are only used where flame resistance is essential, since they are toxic and give high cold flex temperatures. In general, therefore, plasticized PVC compounds will burn, producing a yellow sooty flame, often with a green tip, which leaves a black residue. If a piece of a PVC compound is heated on a copper wire, the flame will often be colored green.

When heated in the absence of a flame, low molecular weight plasticizers will be distilled off and can be analyzed if required. A better way to analyze for the plasticizer is to solvent extract a known quantity of the PPVC material. The solvent from the extraction is evaporated and then liquid plasticizer is collected and analyzed. A useful test, for characterization of the plasticizer type, is the boiling point.

PPVC compounds can be cut very easily and can be scratched easily with a knife or fingernail. This material will dissolve in solvents such as tetrahydrofuran, cyclohexanone, methyl ethyl ketone (MEK), dimethylformamide. Aromatic and chlorinated hydrocarbons, nitrocarbons, acetic anhydride, aniline and acetone also readily swell PPVC. This material will not dissolve in solvents such as aliphatic hydrocarbons, but such solvents can leach out plasticizers.

### 11. Coloring

As the natural color of the PVC is clear, a wide color range is possible, both transparent and opaque. It is sold in both compounded colors and as natural material for in process coloring by techniques such as dry coloring, masterbatching, and liquid coloring. With PVC-P, only pigments are used because of color bleeding or leaching problems with dyestuffs. Certain types of pigments, such as metal powders or oxides, fall into this category. It is much more difficult to color match opaque PPVC compounds than clear ones due to color variations in the base materials. Depending upon the nature of the PPVC compound, granular or powder form, a binder may be required to adhere the colorant to the polymer during the mixing stage. A typical binder is DOP (dioctyl phthalate), which is added to the mix at a concentration level of 0.1%. Many PVC compounds may use a plasticizer system other than DOP, but the inclusion of such a small amount of DOP will not significantly alter the end properties of a component. Typical dry colorant loadings of 0.05% to 2.5% are used. The actual percentage depends upon whether a transparent or opaque color is required.

PVC-based master batches are used to produce good color compatibility, but their usage is relatively limited. Universal master batches are sometimes used, but due to their partial compatibility with the PVC compounds, not all of them are suitable. The manufacturer's advice should be sought before use. Masterbatch additions, for both PVC-based and universal types, are between 1% to 5%, which produces typical pigment loadings of 0.5% to 2% in the final product (depending upon the denseness of color required). Liquid colors can also be used, but their addition must be controlled to prevent changes in the properties of the compound (as a result of the liquid carrier).

### 12. Materials Handling

PVC will absorb less than 0.02% water in 24 hrs at room temperature. This means that drying is not normally necessary. If it is, the material should be dried in a hot air oven for 2 hrs at 65°C/149°F or in a desiccant dryer for 1 to 1.5 hrs at 70°C/158°F. When drying PVC-P compounds, strict temperature and time control must be observed to avoid too great a loss of plasticizer (especially when heating highly plasticized compounds or those containing a highly volatile type of plasticizer). Such losses affect the processing characteristics and the properties of the PVC compound. Prolonged heating can affect the thermal stability of the compound during processing. Do not pre-dry other materials with PVC-P, as plasticizer evaporating from the compound may contaminate them. When a desiccant dryer is used for PVC-P compounds, it is essential that a plasticizer trap be fitted within the dryer to collect vaporized plasticizer and prevent it from contaminating the desiccant bed.

To produce a PPVC dry blend, the PVC polymer and other ingredients (with the exception of the plasticizer and lubricant) are fed into a high speed mixer and blended at high speed until a temperature of between 90°C to 95°C/194°F to 203°F is reached. The plasticizer is then added to the mixture and when 110°C to 125°C/230°F to 257°F is reached, lubricant may be added. The resultant mix is then dumped into a cooler/blender. The large cooler/ blender (usually a water jacketed ribbon type blender) cools the mix, improves the batch-to-batch consistency, and also reduces the electrostatic charges on the dry blend. The dry blend may then be used as is or pelletized using an extruder. Pelletizing improves dispersion, but adds to the cost and reduces the thermal stability of the material.

To improve the batch-to-batch consistency of a dry blend, the additives (e.g., stabilizers and lubricants) may be incorporated in pre-weighed sachets. These are known as 'one shot' or 'one pack' sachets. Their use enables the processor to easily obtain the correct percentage of additive and eliminates the hazards associated with the handling/weighing of the individual ingredients.

Since the inhalation of vinyl chloride monomer (VCM), over prolonged periods, is linked with carcinogenic effects, the level of VCM in the atmosphere is controlled. Although PVC compounds can release traces of VCM into the atmosphere, these levels in normal plant conditions are much below those considered dangerous to health. However, care must be taken in the use of vessels that hold bulk quantities of PVC, especially powder blends, as these have a higher surface area to volume ratio than pellets. The contents of tankers and containers should be sampled without personnel entering the container using a long handled scoop. If it is necessary to enter a container, it should be first purged with air. VCM levels should be monitored and breathing apparatus used if these levels are too high. PVC powder blends also can produce a fine dust, and dust masks should be used where there is likelihood of dust being present in the air. As much as possible, powder blends should be contained and not allowed to blow into the plant atmosphere. Some of the additives used in PVC compounds (lead stabilizers, flame retardants, colorants, etc.) may also be toxic. Smoking, drinking and eating should not occur where blends containing toxic ingredients are present. It is safe to assume that all unmixed PVC compounds are toxic.

If pelletized cubes are used, then gravity feed hoppers are satisfactory. A correctly designed hopper is essential when processing dry blends. The angle of the hopper walls should be between 45° to 60° as angles of <45° can cause bridging problems. If bridging does occur, the use of an agitator can overcome such a problem or one may need to use a crammer design of hopper. The base of the hopper should be cooled to prevent blockage.

### 13. Screw and Barrel Design

The components of the barrel assembly that are in direct contact with hot PVC must be able to resist the corrosive hydrochloric acid (HCl). In addition, the barrel assembly should be designed to avoid sharp corners and abrupt changes in cross-section and direction, to prevent the material from becoming trapped in low flow areas ("dead spots") where it can degrade. Barrels should have a length to diameter ratio of at least 24:1 and should be lined with a material such as Xaloy to reduce wear and resist corrosion. Grooved feed zones are not recommended. Screws need to be flame hardened on their flights or coated with a wear resistant material. Chrome plating also helps to extend the life of the screw. Screws possessing a deep nitrided surface offer good resistance to HCl attack.

Clearance between screw and barrel of 0.005 in to 0.008 in (0.12 mm to 0.2 mm) is recommended. Older designs have a continuous compression from feed to delivery with a compression ratio of about 3. These screws were generally water-cooled, as the lower temperature of the screw improved the mixing action. However, screw cooling also dramatically reduces output. Therefore, more recent designs use a three-zone screw with a long compression section. Mixing rings, consisting of a ring of studs set into the root of the screw, are used to break up the flow pattern and improve mixing. Usually, two or three of these rings are incorporated into the length of the screw.

Screw dimensions depend on the type of feedstock. Screws for powdered feeds need a greater compression ratio than those used for cube (pelletized) feeds. Typical screw dimensions are shown in the following table.

Screw Diameter	2.5"/63.5 mm	3.5"/88.9 mm	4.5"/114.3 mm
Feedstock	Cubes/Powder	Cubes/Powder	Cubes/Powder
Feed Zone Depth (4 D)	0.35/0.36	0.43/0.44	0.50/0.52
Compression (17 D)			
Metering Depth (4 D)	0.14/0.09	0.17/0.11	0.2/ 0.13

### 14. Barrel and Die Temperatures

Since PPVC compounds cover a wide range of compositions, there is no single set of temperature settings. The actual temperatures used are selected for a given compound and process. However, no matter what the composition, temperatures greater than 205°C/400°F will significantly increase the rate of material degradation. High temperatures may also cause premature plasticiza229

230 tion in the feed if dry blends are used. The following temperatures are only given as a starting point.

Zone Temperatures	°F	°C
Zone 1 (feed)	265	130
Zone 2	340	170
Zone 3	355	180
Adapter	365	185
Die	365	185

At the correct melt temperature, PPVC purgings are smooth and glossy. If they have a bumpy or lumpy appearance, then the material is non-homogeneous (that is, not sufficiently thermally plasticized), indicating that the set temperatures are too low. If the purgings have a bubbly/frothy appearance, and give off an excessive amount of smoke, the material is too hot.

### 15. Die Design and Construction

Breaker plates and screens are used between extruder and die. For cube feed, one 20 and two 40 mesh screens are suitable (US standard sieve sizes). For powder feedstock, the meshes are 20/40/100/100/40/20, put together to form a screen pack.

For producing blown film, bottom fed dies are used. They need to be designed for easy assembly/disassembly, so that the required frequent cleaning can be carried out as speedily as possible. In cable coating and sheathing operations, for small wires where dimensional variation is small, a pressure die is used. Here melt and conductor meet in the die. The die exit gap OD is the same size as the diameter of the insulated product. The length of the land is usually also about the same value. Dies can be supplied with fixed or adjustable centering. It is useful to be able to adjust the distance between the tip where melt and conductor meet and the die exit. Tubular dies are used for larger cables. Here melt and conductor meet outside the die, with the melt being drawn down onto the die by the application of a vacuum where the conductor feeds into the die. Again, tip adjustment relative to the outer ring is beneficial. The melt is drawn down onto the cable. For even drawing, the ratio of the cross sectional area of the die exit gap, to the cross sectional area of the PVC sheath, must be the same as the die exit gap diameter to the final cable diameter. Typical ratios are 1.13.

Hose and profile dies are designed on similar principles to those for rigid PVC. Chrome plating of the dies will reduce wear from corrosion by the acid decomposition products of PVC.

### 16. Downstream Operations

If a melt temperature of  $200^{\circ}$ C is used, and the output must be cooled to  $20^{\circ}$ C, and the specific heat in this range is 1564 J/kgK, the maximum amount of heat that needs to be removed for each gram of output is 282 Joules (J).

When producing PPVC film, problems can occur at low gauge because of the softness of the product. In particular, the film is prone to center creasing at the nips and to blocking. To prevent this, collapsing frames should be designed to give minimum friction on the film and the film should be as cold as possible before collapsing. It is possible to produce oriented shrink film in line, by use of a temperature controlled roll stretching unit.

In cable coating, it is necessary to coordinate the whole line including unwind, extruder, cooling, haul-off and winder. A non-rotating payoff (unwind) is used whenever possible, as this enables the ends of cables to be brought out of the pay-off for joining. The cable is drawn over the end of its spool and this process imparts a twist to it. Solid wire unwinds can run up to 5000 ft/min (1525 m/min). For higher speeds, a wire drawing and annealing machine is placed in line with the coating operation. Larger cables, and those to be sheathed, are unwound from rotating drums, using an accumulator if necessary, to allow time for joining.

A water filled trough is used for cooling. PVC can be cooled more rapidly than polyethylene, because it does not crystallize and hence has less tendency to shrink away from the conductor. For sheathing, the troughs are between 50 ft and 150 ft (15.15 m and 45.45 m) long, depending on output rate. An approximate formula for trough length is: Trough Length (ft) = 0.75 X output (lb/h) X PVC radial thickness/sheath diameter.

For fine wire telephone cables, with line speeds up to 7000 ft/min (2135 m/min), troughs are about 120 ft (36.6 m) long. They are up to 700 ft (213.5 m) long when producing cables for building applications. Smaller cables are pulled through the plant using capstans, while caterpillars are employed for larger diameters (reel winding is not used to pull the cable through the plant). The internal diameters of reels are at least 20 times the cable diameter.

In hose production, coilers are used to wind up the hose. These are equipped with a traversing mechanism that ensured the hose coils are positioned correctly on the reel. Automatic strapping of the hose when reeled can also be done on the coilers. There is the possibility of manufacturing braided hose in-line with a primary hose production extruder. The under hose may be preheated before a spiral winding unit applies the reinforcement by rotating the spools of fiber around the hose. Following braiding, a second coat of PPVC is applied by in-line coating.

### **17. Extrusion Capacity**

Extrusion capacity depends on the compound composition and application. Though it is possible to extrude 600 lb/hr (271.2 kg/hr) from a 2.5 in (63.5 mm) extruder, in braided cable production, output figures of 180 lb/h (81.4 kg/h) are common from the same sized extruder. In the table below, figures are given as a guide for a cable coating operation with the maximum output using the most suitable compound and operating conditions.

Extruder Size		Cable Coa	ting Output	Maximum Extruder Output		
(in)	(mm)	(lb/hr)	(kg/hr)	(lb/hr)	(kg/hr)	
1.25	32	40	18			
2.5	64	280	127	600	273	
3.5	90	780	354	1300	591	
4.5	115	1200	545	2000	909	
6.0	150	2030	923	3500	1591	
8.0	200	3700	1682			

### 18. Starting Up

When warming the extruder, the initial temperatures should be set to about 162°C/350°F for the greater part of the warm up period. Ensure that cooling water is present on the hopper throat. Then raise the temperatures to operating conditions and start the extruder slowly. Increase the screw speed to operating conditions as the temperature rises, ensuring that pressures and/or motor current do not exceed predefined limits. One should ensure that all purge material has been removed from the extruder and die before producing extrudate for sale.

### 19. Shutting Down

Since the thermal stability of PPVC is poor, the shut-down procedure is very important. Safe barrel residence time (to avoid degradation) depends upon temperature At a temperature of 200°C/392°F the maximum residence time in the barrel should not exceed 5 minutes. When the temperature is 205°C/401°F then the maximum residence time in the barrel decreases to approximately 3 minutes.

One should ensure that all traces of the PPVC have been removed from the barrel by purging thoroughly with another thermoplastic material, which is more thermally stable and does not react with PPVC (like PMMA, PP, LDPE, HDPE or GPPS). In this way, the PPVC will be replaced with an inert resin such as HDPE before stopping the extruder for a shut-down. When cleaning dies or screws which may be coated with PVC, acrid acidic fumes may be given off. These will cause rusting of unprotected steels and should not be inhaled.

If there is a temporary hold up (less than a few minutes), then purging is not usually necessary. However, a check must be made for signs of degradation when restarting. When shutting down for relatively short periods, switch off the barrel heaters and purge the barrel clean.

Never mix polyacetal (POM) and PPVC together in the same barrel! The resultant vigorous chemical reaction can cause considerable damage to the machine or serious injury to personnel. If PPVC is to follow POM (or visa versa), first, thoroughly purge the barrel clean with one of the inert resins mentioned above.

### 20. Reprocessing

When PPVC is reclaimed, up to 15% may be blended into the virgin material. However, if there are any signs of degradation in the reclaimed material, do not use it. The end use of the product dictates the level of regrind that can be used. What must be borne in mind, is that each time PPVC is reprocessed its resistance to thermal degradation decreases and its flow may be considerably reduced. Problems may, therefore, occur not only because of an excess percentage of regrind being incorporated; they may also occur as a result of using regrind which has been degraded as a consequence of being reprocessed too many times.

### 21. Finishing

Techniques such as high frequency or radio frequency welding, or heated tool and solvent welding can be used to weld PPVC. The effectiveness of the joining method is dependent upon the amount of plasticizer and the formulation of the compound. Depending upon its hardness, PPVC can often be machined using conventional machining techniques. One should, however, keep the surfaces being cut well cooled. PPVC is a material that readily accepts specially designed printing inks. Embossing can be used and is often done in-line.

### 22. Other Comments

When correctly formulated, transparent products are possible. This requires the use of appropriate stabilizers and avoiding the use of fillers or opaque colorants. If impact modifiers are used, the refractive index of the impact modifier and the PVC must be matched. This match may only be possible over a limited temperature range. Where transparency and low toxicity is required organo-tin stabilizers, that are salts of alkyl tin oxides and carboxylic acids called organo-tin carboxylates (e.g., dioctyl tin dilaurate) are used. Organo-tin compounds that have at least one sulfur-to-tin chemical bond are also called organo-tin mercaptides (e.g., dioctyl tin octylthioglycolate). Such organo-tin mercaptides are very efficient heat stabilizers for polyvinyl chloride (PVC) which can yield brilliantly clear compounds.

When PPVC components are produced, it is probable that they will be covered with a thin (invisible) film of lubricant that will contain relatively high levels of stabilizer. If the stabilizer used is heavy metal based (e.g. lead), care should be taken when the product is handled. When handling PVC parts, always wear gloves to avoid transferring the heavy metal onto your skin or into your mouth.

### 23. Typical Products

One of the major uses of PPVC is for wire and cable insulation and sheathing.

It is used in preference to polyethylene (PE), because of its resistance to environmental stress cracking, its self extinguishing characteristics and its versatility. By changes in formulation, such as the type and amount of plasticizer, it is possible to control or alter properties such as flexibility and temperature resistance. However, its inferior voltage breakdown resistance and dielectric behavior at high frequencies preclude it from use at high voltages or frequencies. For sheathing, compounds are made softer than those used for insulation. For example, the Shore hardness of a sheathing grade may be 76, whereas the value for insulation is 86.

It is possible to produce high clarity compounds used in applications for film, hose and flexible profiles. Flexible compounds are available that are formulated to provide high clarity for energy efficient PVC strip curtains used in retail display cabinets or in factory entrances. Use of such curtains provides substantial energy savings, improved temperature control and better hygiene. In film form, the softness and self-adhesive properties of PPVC make it ideal for cling film and stretch wrap.

PPVC is the predominant plastic used in the medical industry because of its cost to performance ratio, transparency, range of hardness, weldability, and ease of sterilization. PVC and its associated plasticizers (such as DEHP) have approximately 40 years of safe and effective use in hospitals and clinics.

PVC is widely used in disposable packaging. A common way of disposing of waste is to burn it in incinerators, however, PVC has for many years been under suspicion as being potentially harmful to health, and to the environment, as being a precursor in the formation of dioxins and furans. Incineration studies do not support this view; therefore, some believe that incineration is a safe and viable disposal option. Operating conditions during burning, rather than the material being burned, appear to have a more significant effect on toxic gas emissions. If incinerators are operated at temperatures below 750°C/1400°F high dioxin levels may result. However, most waste plastics, including PVC, can be recycled and the trend towards recycling such materials is growing.

### 24. Yield

Because PPVC is compounded from a wide range of materials, there is a wide range of relative densities (e.g., from about 1.2 to 1.6). Plasticizer reduces density, but fillers increase the density. Since transparent compounds have no fillers they have the lowest densities and hence greatest yields.

For sheet, figures are given for a range of relative densities from 1.25 to 1.55.

- Sheet of 0.1 in thickness has a surface area of 1.24 to 1.54 sq ft/lb
- Sheet of 2 mm thickness has a surface area of 0.32 to 0.40 sq m/kg
- For film, the relative density is assumed to be 1.29
- Film of 0.002 in (200 gauge) thickness has a surface area of 74.7 sq ft/lb
- Cling film of 0.0005 in (50 gauge) thickness has a surface area of 300 sq ft/lb
- · Film of 50 microns thickness has a surface area of 15.5 sq m/kg
- Cling film of 12.5 microns thickness has a surface area of 62 sq m/kg For hose compounds a density of 1.22 g/cm is assumed
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 22 ft/lb
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 15.1 m/kg

- 1. Common Name
- 2. Abbreviation(s)

### 3. Alternative Names

Unplasticized PVC or Unplasticised PVC UPVC: RPVC: PVC-U: uPVC

Unplasticized polyvinyl chloride or unplasticized PVC: rigid PVC (RPVC): hard PVC. PVC may also be referred to as poly(vinyl chloride) or as poly(monochloroethylene). Type I rigid PVC is a term sometimes used to describe UPVC. Type II rigid PVC is a term sometimes used to describe a grade of PVC that has improved impact strength, but reduced corrosion resistance relative to Type I rigid PVC. Type III rigid PVC is a term sometimes used to describe a grade of PVC that has much higher impact strength than Type I rigid PVC.

UPVC is based on a vinyl plastic material. The term vinyl plastics is often taken to mean vinyl chloride (VC) plastics and refers to materials that are based on homopolymers of VC or, copolymers of VC with other monomers, the VC being in the greatest amount by mass.

5. Trade Names or Trade Marks
Alpha/Dural
Vinidur
Vinoflex
Unichem UPVC
Vista
Lacovyl
Ronfaloy V (ABS/PVC)
Ravinil
Tuf-Stif
UPVC
Pliovic
Hyvin
Trosiplast 1
Vestolit
Keysor UPVC
Novablend
Oxyblend/Oxyclear, Geon
Benvic EV (PVC-GF)
Synergistics
Vinnolit
Westlake

UPVC plastics may be purchased as compound (which means that it has been melt compounded), it may be blended in a high speed mixer and then fed to the extruder, or it may be blended, compounded and extruded inhouse. For this reason, the material may be known by the resin or plastic supplier's name or it may be known by the name of a compound. In general, processing of UPVC compounds gives the best properties, but the processing of dry blends can offer cost advantages.

### 6. Material Properties

PVC homopolymer is an amorphous thermoplastic material with a syndiotactic structure, that is, the chlorine atoms alternate on either side of the main polymer chain. Because of the strong inter-chain attractions which these chlorine atoms generate, the material is harder and stiffer than polyethylene (PE), although it is not significantly crystalline. Because of weak bonds along the chain, PVC will start to decompose or degrade at approximately 74°C/165°F. This problem of poor heat stability is much worse for UPVC than for plasticized PVC (see PPVC).

As the thermal stability of PVC is poor, it is required that the polymer be protected against thermal degradation by using a heat stabilizer. Such heat stabilizers are often based on metal compounds of lead, calcium/zinc mixtures, or tin complexes. Epoxidized soy bean oil (ESBO or ESO), at low concentrations, is used as a component of some stabilizer systems. It is important to ensure that the compound contains sufficient stabilizer so that reprocessing is practical. A heat stabilizer is an example of one of the many additives that are essential in PVC technology. Without additives the plastic cannot be successfully processed. Some additives enhance the properties of the base PVC and permit its use in a very wide range of applications. Apart from colorants, other examples of additives used include:

**Plasticizers:** Plasticizers are added to PVC compounds to confer flexibility and softness. However, they are not used with UPVC as the addition of small amounts of plasticizer can reduce the impact strength of the product and larger amounts reduce its rigidity.

Lubricants: Both internal and external lubricants are used. Calcium stearate is an example of an internal lubricant while synthetic waxes and fatty acid esters are external lubricants. Lubricant packages are formulated specifically to suit a particular application/machine combination. The level of use is often kept low (<1 phr or one part per hundred of resin) in order to reduce plate out problems and a loss of impact strength.

**Processing Aids:** Also known as PA, they are used to reduce the gelation time, the extruder power requirements, and to improve the surface finish of extrudates. Processing aids are often based on an acrylic polymer. Polyhydroxybutyrate (PHB) can be used as a PA for UPVC, because, at a 1% addition level, it reduces the gelation time, power requirements, and improves the surface finish of UPVC compounds.

Fillers: To extend the scope of PVC and/or to make it cheaper, particulate fillers (such as china clay, calcium carbonate and whiting) are often used in PVC compounds.

Anti-Static (AS) Agents: Sulfur-containing AS agent is used as an internal AS agents for PVC as such a material does not interfere with heat stabilizers (e.g., use alkylpolyglycol ether sulphate). Some nitrogen-containing AS agents not recommended for PVC are N,N-bis-(2-hydroxy-ethyl)-alkyl amine or N-(3-do-decyl-2-hydroxy-propyl)-ethanolamine. Phosphoric acid derivatives are used for all materials and especially for PVC as they can function as plasticizers. N,N-bis-(2-hydroxy-ethyl)-N-(3'-dodecyloxy-2'-hydroxy-propyl)-methyl ammonium sulphate is an external AS agent sometimes used with UPVC.

**Impact Modifier:** An impact modifier is an additive that improves the impact strength, particularly at low temperatures. These include methacrylate-butadiene-styrene polymer (MBS), acrylonitrile-butadiene-styrene polymer (ABS), elastomers, and rubbers. When correctly formulated, transparent products are still possible. This means using appropriate stabilizers, and matching the refractive index of the impact modifier and the PVC. Contamination must be avoided during blending or mixing.

Frequently, the PVC resin and additives are blended in a high-speed mixer before being melt compounded. The problems associated with additive use (dispersion, contamination, toxicity, etc.), are difficult in some UPVC applications, as the products are often used as containers for food or drinks (UPVC has good vapor barrier properties).

To obtain the best properties with this material, it is necessary to put in sufficient work and heat to fully gel, or fuse, the blend, but not so much that it is degraded during melt processing. This means that it is important to process the material at the correct melt temperature, avoid excessive shear, and minimize the residence time in the barrel. One or all of these will cause resin decomposition or degradation, signaled by a change in color. Once decomposition starts, it can spread very rapidly, as one of the products of 236 decomposition, hydrochloric acid (HCl), catalyzes further degradation. This acid readily attacks metals (such as steel), causing pitting and corrosion, promotes rust as it strips protective layers away from mild steel and also attacks human tissue.

Despite these problems, UPVC is widely used, as it is a relatively cheap, stiff material. It has good chemical resistance (oil, acid & alkali resistance), reasonable UV resistance, is flame retardant (oxygen index of 45), and is stiff and strong. PVC compounds exhibit low shrinkage (0.2% to 0.6%) and have a low coefficient of thermal expansion.

PVC is now being blended, or alloyed, with other plastics or rubbers to develop new materials with different properties at comparatively low cost. One such blend is of PVC and ABS, which gives a flame retardant, ABStype material. When PVC is blended with an appropriate grade of nitrile rubber (NBR), it is possible to produce a type of thermoplastic elastomer (TPE). Ionic crosslinking of TPE materials is also possible. Polymeric additives are available that will improve the heat distortion temperature (HDT) of UPVC. HDT may be improved by 30°C, by a 20% addition level of one additive. In this way, this relatively inexpensive material can compete with the more expensive engineering thermoplastics. Because of the improvements that blending brings, it is probable that PVC alloys/blends will increase in importance in the plastics industry.

### 7. Flow Properties

UPVC is a relatively stiff flow material with a limited temperature processing range. The ease of flow depends on the molecular weight, which is characterized for PVC by the "K" value. The higher the "K" number, the greater is the molecular weight and the more difficult the flow. For a particular "K" value, the ease of flow may be dramatically altered by the use of additives such as lubricants. In general, relatively low K value resins are used for injection molding as lower molecular weight materials can be processed much more easily. Slightly higher values are used for extrusion. A DIN K value of 55 to 62 corresponds to ISO viscosity numbers of approximately 74 and 95. The inherent viscosity (ASTM) is approximately 1/100 of the viscosity number. When the K value is 50 to 60, then the number average molecular weight is approximately 36,000 to 55,000.

Typical K values for pipe and profile extrusion, are between 66 and 68 (viscosity numbers 110 to 116 according to ISO - R174). For clear sheet and profiles, a less viscous grade, with a K number of around 62 is used.

The table below shows how the viscosity of a UPVC resin varies with temperature and shear rate.

Viscosities of a UPVC Compound (Solvay UPVC PIR945)					
Shear Rate (s <sup>-1</sup> )		Viscosity (Pa-s)			
	(177°C/350°F)	(190°C/375°F)	(204°C/400°F)		
20	12800	9800	7360		
40	7600	5830	4390		
100	3830	2940	2210		
200	2280	1750	1320		
400	1360	1040	780		

Because of its high melt viscosity and elasticity, UPVC has a tendency to generate a lot of shear heat during extrusion. In single screw extrusion, this has traditionally meant extrusion at low speed to prevent overheating of this thermally sensitive material. Low speed extrusion is still used for some profile production where the stability of the low temperature melt is beneficial. Changes in screw design and operating conditions, however, have lead to high speed extrusion of UPVC, even on single screws. In this operation, although the melt temperature is higher, the residence time in the extruder and die is less. The reduced residence time, to some extent, counteracts the adverse effect (on degradation) of the higher temperatures. The material is, nevertheless, softer and more prone to deformation when leaving the die than in low speed extrusion. Because of its difficult flow properties and because of the use of powder feeds, most UPVC extrusion now is done using twin screw extruders, as such machines can have high outputs without excessive shear heating.

### 8. Resistant to:

UPVC is resistant to water, salt solutions, oxidizing agents (for example, hydrogen peroxide), reducing agents, hypochlorite solutions, aliphatic hydrocarbons, detergents, fatty acids (at room temperatures), non-oxidizing acids and concentrated alkalis. It also resists most oils (animal, vegetable and mineral), fats, alcohols and petrol. The plastic is not attacked by strong acids (e.g., any concentration of hydrochloric acid at temperatures up to 60°C/140°F, up to 90%, sulfuric acid at temperatures up to 60°C/140°F, and cold, 50% nitric acid). Chemical and weathering resistance is good to excellent, but is generally made worse by the addition of impact modifiers. Homo-polymers of PVC are more resistant to chemical attack than copolymers. Although the chemical resistance is good, it is possible to join UPVC by solvent welding.

UPVC compounds are not generally recommended for continuous use above  $60^{\circ}$ C/140°F. However, some modifiers can now raise the heat distortion temperature (HDT) by approximately 30°C, so that some compounds have a quoted value of  $90^{\circ}$ C/194°F.

### 9. Not Resistant To:

UPVC is not resistant to hot, concentrated, oxidizing acids (sulfuric, nitric and chromic acids) that cause decomposition. The rate of decomposition may be accelerated in the presence of metals such as zinc and iron. Long exposure to hot, fatty acids can cause softening. Bromine and fluorine attack it even at room temperature. It is unsuitable for use in contact with organic acids (such as acetic acid and formic acid at elevated temperatures), glacial acetic acid, aromatic hydrocarbons, chlorinated hydrocarbons, ketones, nitro-compounds, esters and cyclic ethers which can penetrate the PVC and cause marked swelling. Methylene chloride can be used to detect an under-gelled compound. The material may be subject to environmental stress cracking (ESC) if a component is subject to stress when in contact with acids, alkalis and aliphatic alcohols. Common examples of ESC agents include 2-propanol and sodium hydroxide.

UPVC may have high impact strength, but can be very notch sensitive. UPVC is sensitive to comparatively small changes in temperature. It can become brittle at approximately 5°C/40°F, unless it is impact modified and has a low continuous use temperature (~50°C/120°F). Although pure PVC does not absorb light at wavelengths above ~220 nm, commercial materials contain impurity groups or structural features which contribute to degradation by light. Heat stabilizers (for instance phosphites) do help with light stabilization, but for the best results, use a UV absorber (a simple, low molecular weight HALS stabilizer may have little or no effect on light stability). When PVC is degraded by light, a chalk-like dust may be produced, which may be harmful if ingested. This is particularly true if the PVC contains a heavy metal stabilizer such as one based on lead.

Where transparency and low toxicity is required organo-tin stabilizers, that are salts of alkyl tin oxides and carboxylic acids called organo-tin carboxylates (e.g., dioctyl tin dilaurate) are used. Organo-tin compounds that have at least one sulfur-to-tin chemical bond are also called organo-tin mercaptides (e.g., dioctyl tin octylthioglycolate). Such organo-tin mercaptides are 238 very efficient heat stabilizers for polyvinyl chloride (PVC) which can yield brilliantly clear compounds

### 10. Material Detection or Identification

PVC is an amorphous thermoplastic material, which is produced in the form of fine powder. It does not exhibit a sharp melting point, but a glass transition (Tg) at about 82°C and degrades easily when heated. It does not soften below its Tg. With a density of approximately 1.4 g/cm<sup>3</sup>, the material (solid, non-filled material) will sink in both water and in a saturated solution of magnesium chloride. The density of commercial compounds may range from 1.34 to 1.45 g/cm<sup>3</sup>, as the compounded material may contain up to 5% additives. If filled, then this level will be exceeded. The natural color of the material is clear when it is unfilled and stabilized with lead stabilizers and /or filled the natural color is usually off-white. In either case this means that a wide color range is possible. UPVC is a hard stiff material. Many compounds have a hardness, on the Shore D scale, of about 80 and a Vicat softening point (50 N load) of 65°C to 90°C/149°F to 194°F.

When this material is heated in a flame, it softens slightly and chars. It is self-extinguishing, but if heated strongly, a dirty yellow flame, with green tinged edges, is seen accompanied by heavy smoke and an acrid smell. When heated in the absence of a flame, this material will first release HCI and then various hydrocarbons. If a piece of this material is heated on a copper wire, the flame will be colored green. It can be cut easily with a knife and can be scratched relatively easily with a knife, but is harder than PPVC. UPVC has a low softening point (82°C), well below that of boiling water.

UPVC material will dissolve in solvents such as tetrahydrofuran (THF), cyclohexanone, methyl ethyl ketone (MEK) and dimethylformamide. Solvent mixtures based on acetone or benzene also dissolve it. UPVC will not dissolve in aliphatic hydrocarbons, but will swell in solvents such as aromatic and chlorinated hydrocarbons, nitro compounds, acetic anhydride, aniline, and acetone.

### 11. Coloring

As the natural color of the material can be clear, a wide color range is possible. This includes both transparent and opaque colors. UPVC is sold in both compounded colors and as natural material for coloring on the machine, by techniques such as dry coloring, masterbatching and liquid coloring. When coloring UPVC, both dyestuffs and pigments can be used. However, certain types of pigments can catalyze decomposition. Metal powders or oxides fall into this category. The color matching of opaque UPVC compounds can sometimes be difficult, due to color variation in the base material, which may occur as a result of changes in the additives used. Depending upon the form of the UPVC compound (granular or powder form), a binder may be required to make the colorant adhere to the polymer during the mixing stage. A typical binder would be DOP (dioctyl phthalate), which is added to the mix at a concentration level of 0.1%. Typical dry colorant loadings of 0.05% to 2.5% are used. The actual percentage is dependent upon whether a transparent or opaque color is required.

The most commonly used type of masterbatch is PVC based. Only a few universal master batches are suitable and, therefore, advice should be sought from the manufacturers before use. Masterbatch additions of 2% to 4% are used for typical pigment loadings of 1% to 2% in the final product, depending upon the denseness of color required. Liquid colors can also be used, however, the addition level must be controlled so as not to create changes in the properties of the product compound as a result of the addition of liquid carrier.

### 12. Materials Handling

UPVC is used in the form of granules or as a dry blend. Granules (pellets) present no special handling problems. To produce a UPVC dry blend, the PVC polymer is added together with other ingredients into a high-speed mixer. This mixture is blended at high speed until a temperature of between 110°C to 125°C is reached, when the lubricant and/or impact modifier is added. The resultant mix is dumped into a cooler/blender that is used to cool the mix, to improve the batch-to-batch consistency of the blends, and to reduce the electrostatic charges on the dry blend. The dry blend may then be used directly or pelletized by an extruder. Pelletizing improves dispersion, but adds to the cost and reduces the thermal stability of the material.

Many users prepare their own dry blends as this reduces cost and the compound design can be kept in house. For large tonnage producers, the weighing and mixing is done under automatic control. Sometimes the main mixing is done as described above and mixing of dry color additives is done in ribbon blenders as a separate operation. To improve the batch-to-batch consistency of a dry blend, the additives used, (stabilizers, lubricants, processing aids, impact modifiers and fillers) can be incorporated in pre-weighed sachets (one shot or one pack sachets). This eliminates the hazards associated with the handling and weighing out of the individual ingredients such as heavy metal stabilizers.

Because of the dust problem associated with UPVC dry blends, additives are now being supplied in a granular, flake or spaghetti form. Although the dispersion characteristics of these forms are not as good as when the additive is a powder form, they are preferred by processors because of safety considerations. PVC powders are prone to dispersal in the atmosphere, so they should be kept in closed containers at all times. They are best transported about the factory using automated material transfer systems. It is recommended that the level of exposure not exceed 10 mg/m<sup>3</sup> for total PVC dust in air and 5 mg/m<sup>3</sup> for respirable PVC dust in air. When using powder feed, there is a tendency to blocking in the hopper that will cause a feed failure. This can be overcome by using a forced feed screw in the hopper or by vibrating the hopper to prevent the formation of "rat holes".

If extrusion is performed at low speed, moisture and volatiles will out-gas backwards during extrusion, so there is usually no need to pre-dry the material (UPVC absorbs less than 0.02% water in 24 hrs at room temperatures). However, at the high extrusion rates used commercially, it is recommended that the material be dried in a hot air oven at a temperature of 105°C/220°F for 1.5 hrs or at 65°C/150°F for 3 hrs (application of a vacuum during drying will help). If using a desiccant dryer, then the material should be dried at 80°C/176°F for 1 to 1.5 hrs. Alternatively, use a vented extruder.

### 13. Screw and Barrel Design

The largest tonnage of UPVC is extruded on counter-rotating twin screw extruders, although co-rotating designs are also used. Both cylindrical and conical twin screws have been used successfully. The advantage of twin screw extrusion is the ability to get high outputs without excessive shear in the melt and thus lower melt temperatures than are possible with high speed single screw extrusion. Details of the twin screw design is outside the scope of this handbook.

The tables below show typical screw designs used for single screw extrusion, at low speeds and low melt temperatures and at higher speeds and temperatures. The high-speed screws are designed as two stage screws. The first stage compresses and melts the compound, which is then decompressed before the second stage. A vent may be positioned at the decompressed stage to remove volatiles. Care must be taken with the two stage designs, not to allow material to fill the decompression section (as the result of too great a die resistance). The figures in brackets indicate the length of each zone.

Low Speed Extrusion - Single Screw					
Diameter	2.5"/63.5 mm	3.5"/88.9 mm	4.5"/114.3 mm		
Feed Zone Depth (4 D)	0.43"/10.9 mm	0.65"/16.5 mm	0.68"/17.3 mm		
Compression Zone	(16 D)	(16 D)	(16 D)		
Metering Zone (4 D)	0.009"/0.23 mm	0.27"/6.85 mm	0.28"/7.11 mm		
High Speed Extrusion -	Single Screw				
Diameter		2.5"/63.5 mm	3.5"/88.9 mm		
1st Feed Zone Depth (3	D)	0.38"/9.6 mm	0.53"/13.5 mm		
Compression Zone		(7 D)	(6 D)		
1st Metering Zone Dept	h (4 D)	0.13"/3.3 mm	0.17"/4.3 mm		
Decompression (2 D) to	Depth of	0.39"/9.9 mm	0.68"/17.3 mm		
Compression 2nd Stage		(5 D)	(4 D)		
Final Metering Section (	3 D)	0.185"/4.7 mm	0.31"/7.9 mm		

Single stage screws are also used for UPVC at high speeds. They should have a compression ratio of about 2.5:1 and are often fitted with 2 - 4 sets of pin mixing studs, which are a ring of studs that stand up from the screw root to a height of about half the channel depth. The screw flight can be broken to accommodate the ring of pins. Barrier screws have been used successfully for UPVC compounds. Other designs include screws incorporating an additional flight inside the normal channel. Such designs may put the extra flight in the first stage for powder feeds, but in the second stage for pellet feed.

All high-speed screws should be temperature controlled by having a hollow section through which a fluid can be circulated. Heated oil, at a temperature at 90°C to 160°C/190°F to 320°F, is the best cooling medium, but water or air-cooling is possible.

### 14. Barrel and Die Temperatures

The table below shows a typical temperature profile for the extrusion of UPVC, in degrees Fahrenheit. Figures in brackets are the centigrade equivalents.

Screw Type Temperatures [°F(°C)]	Low Speed Single (Vented)	High Speed Single	Twin
Zone 1	300 (150)	350 (180)	330 (165)
Zone 2	320 (160)	350 (180)	340 (170)
Zone 3	340 (170)	370 (190)	350 (177)
Zone 4		370 (190)	363 (184)
Zone 5		350 (180)	360 (182)
Adapter	365 (185)	390 (200)	375 (190)
Die	365 (185)	390 (200)	380 (193)
Melt	355 to 370°F	380 to 400°F	383 (195)
	(180 to 190°C)	(190 to 205°C)	

The correct melt temperature of UPVC can be readily identified by the appearance of barrel purgings. If the purgings have an lumpy appearance, the material is non-homogeneous (not sufficiently plasticized), indicating that the set temperatures are too low. If the purgings have a frothy appearance and give off an excess amount of smoke, then the material is too hot. Typically, at a temperature of 200°C/392°F, the maximum residence time in the barrel should not exceed 5 minutes. When the temperature is 210°C/419°F, then the maximum residence time in the barrel should not exceed 3 minutes.

### 15. Die Design and Construction

When designing dies for UPVC compounds, the high viscosity of the melt and the poor thermal stability of PVC must always be considered. In some ways, these two issues lead to opposite solutions. The high viscosity means that flow channels should be generous in size to prevent high pressures. To avoid degradation, caused by long residence times, the die volume should be small. If the flow channels are made too small, then the large amount of pressure generated is transferred into heat. This heat will appear as a local temperature rise in the regions where the material is being sheared the most. These local temperature increases can be very high and may lead to severe material degradation. One must ensure that there is a continuous flow of material at all points in the flow path, with no stagnation at sharp corners or at chips and scratches in any metal surface. Regions of compression and decompression should be avoided and the flow path kept very streamlined. UPVC dies are usually chrome plated.

In sheet dies, a choker bar is not used, as this provides a space where material can hold up and degrade.

In pipe dies, there has to be compression after the spider to encourage the melt to weld together. Typically, the torpedo diameter ratio at the spider, compared to the land, is about 2:1. The overall volume compression ratio is in the range 7:1 to 10:1, so the channel height also decreases. The length of the die land must be about 20 times the die exit gap width. For thin walled pipes, the ratio can be somewhat less, but should be more for thick walled pipes. UPVC pipe dies come with a range of interchangeable rings and pins. This means that one die body can produce a range of pipes, though not as wide a range as with a die for polyolefins. The table below shows a typical range of UPVC pipe dies.

Die	Pipe Diameter	Wall Thickness	Maximum Output Rate
	("")	("")	(16/111)
A	0.2 - 0.6	0.04 - 0.07	100
В	0.5 - 2.0	0.04 - 0.15	200
С	1.3 - 3.0	0.07 - 0.28	500
D	2.0 - 6.0	0.07 - 0.40	900
E	3.0 - 9.0	0.07 - 0.55	1300
F	4.5 - 15.0	0.08 - 0.90	1700
G	6.5 - 18.0	0.10 - 1.1	2200
Н	15.0 - 27.0	0.15 - 1.2	2200

Profile dies are designed to achieve streamlined flow. In designing components for manufacture, if possible, wall thickness' should all be about the same and sharp internal or external corners avoided. Hollow cored sections with internal struts replace large section, if they are necessary. Designs where a strut meets a flat surface can cause problems. Dies are made from hardened chrome nickel steels and need to be highly polished.

### 16. Downstream Operations

When a melt temperature of 200°C is used, and the output must be cooled to 20°C, the specific heat over this temperature range is approximately 1820 J/kgK. This means that the maximum amount of heat that needs to be removed for each gram of output is 328 Joules (J).

Calibration (sizing) dies are needed to ensure satisfactory dimensional tolerances. Vacuum calibration is essential when producing window frame profile. To illustrate, with a production rate of 200 lb/hr (91 kg/h) and a line speed of 6 ft/minute (1.8 m/min), a set of four vacuum sizing units is used, each unit being about 2 ft (0.6 m) long. At double the production rate, extra sizing and cooling units must be added (for example, another 5 ft or 6 ft (1.5 m to 1.8 m) of unit length).

In both profile and pipe lines, lengths are sawn or cut automatically using computer controlled saws. The saw blades are tungsten carbide tipped and the tooth form is specially designed for PVC. The shaft speed is such as to give the saw blade a cutting speed of about 160 ft/s (48.6 m/s).

Automatic monitoring of pipe thickness is becoming more common. VDU displays are linked to the monitoring equipment and display machine settings, operating temperatures, pressures, and pipe thickness profile. The measurements can also be used for automatic line and die adjustment.

### 17. Extrusion Capacity

The figures below refer to typical maximum extruder output when using high viscosity UPVC resins.

Extruder Size		Low Speed Screw		Hig	High Screw Speed		
(in)	(mm)	(lb/hr)	(kg/hr)	(rpm)	(lb/hr)	(kg/hr)	(rpm)
2.5	64	70-90	30-40	15-25	250-350	110-160	40-70
3.5	90	80-150	40-70	10-20	420-550	190-250	40-60
4.5	115	120-180	50-80	5-15	600-750	270-340	20-30

Output from twin screw extruders is shown in the next table (Note: The extruder output figures refer to pipe production).

Diameter of Screws		Maximu	m Output	Pipe Diameter	
(in)	(mm)	(lb/hr)	(kg/hr)	(in)	(mm)
2.9	73	400	180	0.63-6.3	16-160
3.6	91	880	400	1.25-10.0	32-254
4.9	125	1540	700	4.30-25.0	109-635
5.2	132	2200	1000	4.30-25.0	109-635

### 18. Starting Up

Before starting up, one should ensure that the extruder is not contaminated with acetal (POM) or a plastic material which contains a halogen flame retardant. *If there is any doubt, purge the extruder.* When changing to a food grade, purge the extruder thoroughly of any non-food grade material.

### 19. Shutting Down

Since the thermal stability of UPVC is poor, the extruder shut down procedure is very important. One must ensure that all traces of the UPVC have been removed from the barrel, by purging thoroughly with another thermoplastic material that is more thermally stable and does not react with UPVC. Reduce the temperature of the extruder, and then purge the barrel clean with a compatible material such as PMMA or ABS. If these are not available, use PP or HDPE. After completing a production run, the metal surfaces of the die should be washed scrupulously with a mild alkaline solution such as sodium nitrate. After drying, the metal surfaces should be sprayed with oil or silicone based spray.

Never mix polyacetal (POM) and UPVC together in the same barrel, as the resultant vigorous chemical reaction can either cause considerable damage to the machine or serious injury to personnel. If UPVC is to follow POM (or visa versa), thoroughly purge the barrel clean with one of the materials stated above first.

When PVC components are produced, it is probable that they will be covered with a thin (invisible) film of lubricant. This lubricant film will probably contain relatively high levels of stabilizer. If the stabilizer used is heavy metal based, like lead, then care should be taken when the product is handled. Take care when handling PVC product and always wear gloves, to avoid transferring the heavy metal onto your skin or into your mouth.

### 20. Reprocessing

When this material is reclaimed, up to 15% may be blended into the virgin material. The end use of the molded component dictates the level of regrind that can be used. What must be borne in mind is that each time UPVC is

reprocessed its resistance to thermal degradation is considerably reduced. Instances have occurred where components fail in service due to the abuse of regrind. Problems such as these occur, not only because of the percentage of regrind being incorporated, but also as a result of using regrind which has been degraded as a consequence of being reprocessed too many times.

### 21. Finishing

UPVC can be machined without difficulty, but the surfaces being cut should be kept well cooled. When drilling or sawing UPVC, lubricants should not be used. It may be welded by various methods such as ultrasonic, heated tool, hot gas, friction, and solvent techniques. UPVC can be decorated using various methods such as painting, hot foiling, vacuum metallizing, EMI shielding, and silk screening. Because PVC is a polar material, it can be printed or decorated without the need for flaming or corona discharge. Inks are available for printing onto PVC, which, because of its polar nature, readily accepts the ink.

Hot plate welding is widely used in the window frame industry. Surfaces to be joined are brought into contact with a PTFE fabric-covered hot plate at a temperature of 210°C to 230°C/410°F to 450°F. The surfaces are held against the hot plate for 30 - 35 seconds, after which they are pulled away from the plate (which is removed) and immediately pushed together. After being held together, for up to a minute while the weld cools, the welded component is trimmed to remove weld flash. About 0.1 in (0.004 mm) is lost off the extrudate during hot plate welding. Solvent welding is another widely used method. The initial set up is quick, but joints need 24 hrs to develop their full strength. Joints require finishing after welding to give a polished surface. The process of grinding and polishing can be very time consuming.

### 22. Other Comments

Vinyl chloride (VC) polymers may be made by emulsion (giving PVC-E), suspension (PVC-S) and bulk, also known as mass, polymerization (PVC-M). UPVC is usually based on PVC-S or, PVC-M. As VC is toxic, the level of monomer remaining in the plastic must be kept very low (< 1 part per million (1 ppm)).

Post-chlorinated PVC (PVC-C or CPVC) is more difficult to process than UPVC, but is more chemical and temperature resistant. It may be referred to as PVCHT, because of its high temperature resistance (~100°C/ 212°F). It is used for hot water piping and for chemical equipment.

### 23. Typical Products

One should remember that to get consistent results from the UPVC extrusions, of the products mentioned below, it is essential to accurately control the heat/shear history of this material during processing.

The major application is in pipes for waste for drainage, etc. Usage of UPVC profiles for window frames, has increased considerably because of the materials good weatherability, good color range, stiffness, toughness, and relatively low cost. Other profile shapes include runners, venetian (and other) blinds, cladding, framing, fencing, room partitioning and electrical conduit. It is widely used in chemical plants because of its good chemical resistance, rigidity, and non-flammability.

As it is an amorphous material, it will soften at comparatively low temperatures (Do not use above 60°C/140°F). The temperature resistance may be raised by the use of additives. Some of these modified materials are claimed to be able to rival ABS for particular applications.

Rainwater down pipes and guttering are also extruded from UPVC and CPVC compounds because of their rigidity, UV resistance, and excellent chemical resistance.

In film and foil, UPVC is used in the thickness range from 80 to 160 gauge as a twist wrapping film, while thicker gauges are produced as

**244** materials to be thermoformed into packaging for cookies, cakes, etc. Such products, like many other items of packaging, are sometimes disposed of by burning in incinerators. PVC incineration has been under suspicion as being potentially harmful to health and the environment in the formation of dioxins and furans. Incineration studies do not support this view (see PPVC). UPVC is also under pressure because of concerns about by-products when the material burns. However, UPVC resists ignition, needs a long exposure to an ignition source to cause ignition, has a low rate of heat release, and produces less smoke than many other thermoplastic materials when burned.

### 24. Yield

- Sheet of 0.1 in thickness has a surface area of 1.38 sq ft/lb weight
- Sheet of 2 mm thickness has a surface area of 0.36 sq.m/kg weight
- Film of 0.002 in (200 gauge) thickness has a surface area of 68.8 sq ft/lb weight
- Film of 50 microns thickness has a surface area of 14.3 sq m/kg
- Tube of 3/8 in ID and 1/16 in wall thickness has a length of 19.2 ft/lb weight
- Tube of 10 mm ID and 1.5 mm wall thickness has a length of 13.2 m/kg
- Pipe of 4 in ID and 1/10 in wall thickness has a length of 1.13 ft/lb weight
- · Pipe of 100 mm ID and 2 mm wall thickness has a length of 989 mm/kg

# SECTION 9 USEFUL INFORMATION

- ≻ SI UNITS ADVICE ON USE
- > UNIT CONVERSION
- **> TEMPERATURE CONVERSION TABLE**
- > THERMOCOUPLE SELECTION
- > APPLICATIONS OF RHEOLOGICAL DATA FOR EXTRUSION
- MATERIAL PROPERTY GUIDELINES
- > GLOSSARY OF EXTRUSION TERMS
- ► BOOK LIST

# SI UNITS ADVICE ON USE Système International d=Unité

SI is an abbreviation used for Système International d=Unité. SI units were derived from the MKS system and are sometimes known as MKSA metric. The units issued by this organization are an international coherent system now widely used for scientific and technical work. The seven basic units are the meter (length), kilogram (mass), second (time), ampere (electric current), Kelvin (temperature), mole (amount of substance) and the candela (luminous intensity). There are supplementary units for plane angle (radian) and solid angle (steradian) and some SI derived units that have special symbols. These units include the Newton and the Pascal. Certain other non SI units are permitted such as cm and °C.

# Suggestions

Singular and plural forms of SI unit abbreviations or symbols are the same. That is, do not put the letter s after the unit abbreviation, or symbol, if specifying more than one of a particular unit. SI symbols are always written in roman type - not in italics.

A period (full stop) is not used with the unit abbreviations or symbols, except at the end of the sentence.

A space is left between the number and the unit abbreviation or symbol, except when the temperature is specified in degrees Celsius (centigrade).

When the temperature is specified in degrees Celsius (centigrade), the word Celsius begins with a capital C (upper case). The unit abbreviation or symbol is also written with a capital C prefixed with a small zero written level with the top of the C. Unit abbreviations or symbols, are written in lower case letters except when the unit abbreviation or symbol, is derived from a proper name. The full name of the unit abbreviation, or symbol, is written in lower case letters even when it is derived from a proper name. So, the units named after Pascal would be written as Pascal and abbreviated to Pa. One Pascal <sup>6</sup> would be abbreviated to 1 Pa and 14 Pascals would be abbreviated to 14 Pa and not to 14 Pas. Adding an "s" forms plurals of unit names: except for henries, hertz, lux and siemens.

Compound units formed by multiplication are written in a number of ways. For example, Newton meters may be written as Nm or as, N.m or as, N m.

Compound units formed by division are written in a number of ways. For example, Newtons per square meter may be written as  $N/m^2$  or as,  $Nm^2$ . The suggestion  $N/m^2$ , is preferred.

Compound prefixes are not used. That is, one million meters would be written as 1 Mm and not 1 kkm.

Common fractions are not used. That is, one half of a kilogram would be written as 0.5 kg and not 1/2 kg.

Prefixes are not used in the denominator of a compound unit except for kilograms - as the kilogram is the base unit of the SI system. That is, one million Newtons per square meter would be written as 1 MN/m<sup>2</sup> and not as 1 N/mm<sup>2</sup>. (One Newton per square millimeter is the same as one million Newtons per square meter.)

It is suggested for simplicity, when calculations are being performed, that all prefixes be changed so that powers of ten (decimal multiples) are used.

It is suggested, when decimal multiples are used, that the prefix used should be 10 raised to a power that is a multiple of 3.

It is suggested for ease of understanding, when density is discussed, that the units are  $Mg/m^3$  rather than kg/m<sup>3</sup>. This gives values that have the same numerical values as the well-established g/cm<sup>3</sup> values or SG values.

To avoid misunderstandings avoid the use of word billion as it can have more than one meaning: use a prefix such as G for giga. Note the prefixes E,P,T,G and M are all capitalized.

To avoid misunderstandings avoid the use of a comma to separate groups of digits as a comma may be used as a decimal marker in some countries.

# ► UNIT CONVERSION

To make recognition easier, the figures have been divided by spaces where appropriate. For example, spaces have been used as a thousand marker to the left of the decimal point. To the right of the decimal point, a space divides the digits into groups of three. In some cases a back slash, or /, has been used in place of the word 'per'. The use of an asterisk \* indicates an exact number.

Knowing	Multiply by	To Get
(Atmospheres - means st	andard atmospheres	unless otherwise stated.)
Atmospheres	1.013 250	bars
Atmospheres	75.999 989	centimeters of mercury
Atmospheres	29.921 256	Inches of mercury (at 0°C)
Atmospheres	1.033 228	kilograms square centimeters
Atmospheres	101.325 0	kilonewtons/sq meter
Atmospheres	101 325.0	pascals
Atmospheres	14.695 949	pounds per square inch
Avdp = avoirdupois.		
Bar	0.986 923	atmospheres
Bar	100 000.0 *	newtons per square meter

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Knowing	Multiply by	To Get
Bar	1 000 000.0	dynes per square centimeter
Bar	750.061 576	millimeters of mercury
Bar	14.503 774	pounds per square inch
Btu = British thermal unit	S.	
Btu	251.995 764	calories (gram calories)
Btu	1 055.055 863	joules (Newton meters)
Btu	0.251 996	kilocalorie
Btu	0.000 293	kilowatt hours
Btu/hour	0.069 998	gram-calories/second
Btu/hour	0.293 071	watts
Calories - large	1 000.0 *	calories
CaloriesIT	0.003 968 321	British thermal units
Calories	0.000 001 163	kilowatt-hours
Calories	4.186 80	joules (Newton meters)
Centimeters	0.1 *	decimeters
Centimeters	0.032 808	teet
Centimeters	0.393 /01	inches
Centimeters	0.010 ^	meters
Centimeters	10 000.0	microns
Centimeters	10.0	millimeters
Centimeters/second	1.968 504	reet/minute
Centineiers squared - se	e square centimeters.	dung gagand/ag gantimator
Continuises	1.0	arame/continueter cocond
Continuises	0.010	
Cantinoises	1.0 *	pulses
Centinoises	0.001.0	nascal-seconds
Cubic centimeters	0.001 0	cubic feet
Cubic centimeters	0.061 023 744	cubic inches
Cubic centimeters	0.000.001	cubic meter
Cubic decimeters	10	liters
Cubic feet	28316.84659	cubic centimeters
Cubic feet	1728.0	cubic inches
Cubic feet	0.028 316 847	cubic meters
Cubic feet/minute	1.0	cumins
Cubic feet/minute	0.471 947	liters/second
Cubic feet/second	1.0	cusecs
Cubic inches	16.387 064	cubic centimeters
Cubic inches	0.000 578 704	cubic feet
Cubic inches	0.000 016 387	cubic meters
Cubic inches	16 387.064 00	cubic millimeters
Cubic meters	1 000 000.0	cubic centimeters
Cubic meters	61 023.744 09	cubic inches
Cubic meters	35.314 666	cubic feet
Cubic meters	10.0	hectoliters
Cubic meters	1000.0 *	liters
Cubic yards	0.764 555	cubic meters
Cumins	1.0	cubic feet/minute
Cusecs	1.0	cubic feet/second
Cycles per second	1	hertz
Deciliters	0.000 1	cubic meters
Deciliters	0.1	liters
Decimeters	10	centimeters
Decimeters	3.937 008	incnes

247

248	Knowing	Multinly by	To Get
	Decimeters	$\cap$ 1	meters
	Dekaliters	10.0 *	liters
	Dekameters	10.0 *	meters
	Denier (international)	1.111 x 10-7	kilograms per meter
	Dynes	0 000 000 1	ioules/centimeter
	Dynes	0.000 001 02	kilograms-force
	Dynes	0 000 01 *	Newtons (ioules/meter)
	Eras	0.000 000 1 *	ioules (Newton meters)
	Feet	12.0 *	inches
	Feet	0.304 80 *	meters
	Feet per minute	0.508 0	centimeters per second
	Feet per minute	0.304 80 *	meters per minute
	Feet per second	30.480	centimeters per second
	Foot pounds	12.00 *	inch pounds
	Foot pounds	0.138255	kilogram (force) meters
	Foot pounds	1.355 818	Newton meters (joules)
	Gauge	0.254	microns
	Grams	0.001 0	kilograms
	Grams	0.035 274	ounces (avdp)
	Grams	0.002 205	pounds (avdp)
	Hectoliters	0.10	cubic meters
	Hectoliters	1 000.0	deciliters
	Hectometers	100.0	meters
	Horsepower (550 ft lbf/s)	745.700	watts
	Horsepower (electric)	746.0 *	watts
	Horsepower (metric)	/5.0 *	kilogram meters/second
	Inch pounds	0.083 333	foot pounds
	Inch pounds	0.112 985	Joules (Newton meters)
	Inch pounds	0.011 521	kilogram (force) meters
		0.025 40	meters
	Inches	20.40	mile
	Inches per minute	1 000.0 0 402 222	IIIIIS millimators par second
	loules (Newton motors)	0.423 333	
	Joules (Newton meters)	0.00094701	
	Joules (Newton meters)	0.230 040 1 0 v 107	
	Joules (Newton meters)	0 737 562	foot nounds
	Ioules (Newton meters)	8 850 746	inch nounds
	Joules (Newton meters)	10 197 162	kilogram (force) centimeters
	Joules (Newton meters)	0 101 907	kilogram (force) meters
	Joules (Newton meters)	10*	Newton meters
	Joules (Newton meters)	10	watt-second
	Kilobar	100.0	megapascals/meter squared
	Kilocalories	1 000.0	calories (gram calories)
	Kilocalories	4.186 80	kiloioules
	Kilogram calorie = kilocalor	ies	
	Kilogram (force) meters	7.233 014	foot pounds
	Kilogram (force) meters	86.796 217	inch pounds
	Kilogram (force) meters	9.806 650	joules (Newton meters)
	Kilograms	1000.0	grams
	Kilograms	2.204 62	pounds
	Kilograms	0.001 0	tonnes (metric)
	Kilograms	0.000 984 207	tons (long)
	Kilograms/cubic meter	0.062 428	pounds per cubic foot

Knowing Kilograms/liter	Multiply by	To Get
Kilojoulas	1.0	ioules (Newton meters)
Kilomotore		contimeters
Kilomotoro	2 200 020 005	foot
Kilomotoro	3 200.039 095	metero
Kilometere		milee
Kilometers per bour	0.021 371	fact per second
Kilometers per hour	0.911 344	miles per bour
Kilonende (kn.er.kne)	0.0213/1	lilles per liour
Kiloporius (kp or kps)	1.0	kilografiis (kgi)
Kilopounds (Kips)		pounds Pritich thermal units
Kilowall hours	3 412.141 000	British thermal units
KIIOWATT NOURS	859 845.227 9	calories
KIIOWatt nours	3 600 000.0	Joules
Liters	1.0	cubic decimeters
Liters	61.022 /44	cubic inches
Liters	0.035 315	cubic feet
Liters	2.113 3/6	pints (US liquid)
Megabars	100.0	giganewtons/square meter
Megapascals (MPa)	10.0 x 106	bar
Megapascals	1.0	
meganewtons/square meter	•	
Megapascals	145.038 736	pounds per square inch
Meters	100.0	centimeters
Meters	10.0	decimeters
Meters	3.280 840	feet
Meters	0.001	kilometers
Meters	1 000.0	millimeters
Meters per second	196.850 394	feet per minute
Meters per second	2.236 94	miles per hour
Meters per minute	3.280 840	feet per minute
Meters per minute	0.037 28	miles per hour
Metric tonnes	1 000.0	kilograms
Metric tonnes	2 204.622 60	pounds
Microinch	0.000 001	inches
Microns	3.937	gauge
Microns	10 000.0 *	Angstroms
Microns	1.0 x 106	meters
Miles	5 280.0 *	feet
Miles	1.609 344	kilometers
Miles	1 760.0 *	vards
Millibar	100.0	newtons per square meter
Millimeters	0.10	centimeters
Millimeters	0.003 281	feet
Millimeters	0.039 370	inches
Millimeters	1 000.0	microns
Millimeters	39.370 079	mils
Mils	0.002 54	centimeters
Mils	0.001	inches
Mils	0.025 4	millimeters
Newton centimeters	0.010	Newton meters
Newton meters - see Joules		
Newtons/square meter	0.000 009 869	atmospheres
Newtons/square meter	1.0 *	Pascals
	-	

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250	Knowing Newtons/square meter Newtons/square meter Ounces (avdp) Ounces (avdp) Ounces (avdp) Ounces per cubic inch Pascals Pascal-seconds Pascal-seconds Pints (UK) Pints (UK) Pints (US dry) Pints (US liquid) Poise Poise Poise Pounds (avdp) Pounds (avdp) Pounds (avdp) Pounds per square inch Pounds per square inch Square centimeters Square centimeters Square feet Square feet Square meters Square yards Tons (long) Tons (long) Tons (US) Tons (US) Tons (US) Tons (US)	Multiply by 10.0 * 10.00 145 038 28.349 523 0.028 350 0.062 50 1.733 1.0 1.0 1.0 0.568 261 1.200 950 0.550 610 0.473 2 1.0 * 1.0 * 1.0 * 0.10 453.592375 0.453 592 375 16.0 * 0.016 019 0.068 046 0.070 306 979 0.068 046 0.070 306 979 0.006 894 76 6 894.757 361 1.101 122 946.352 946 62.427 960 0.578 038 0.001 076 0.155000 929.030 400 0.092 903 6.451 60 10 000.0 10.763 910 0.836 127 1.016 047 2 240.0 * 1.120 0.907 185 2 00.0 * 0.892 857 3.412 142 859 845	To Get dynes/sq centimeter pounds per square inch grams kilograms pounds (avdp) grams per cubic centimeter Newtons per square meter Newtons seconds/sq meter poise liters pints (US liquid) liters liters dyne second/sq centimeter gram/centimeter second pascal-seconds grams kilograms ounces (avdp) gram per cubic centimeter atmospheres kilograms/sq centimeter megapascals newtons per square meter liters cubic centimeters pounds per cubic foot ounces per cubic inch square feet square inches square centimeters square centimeters square decimeters square decimeters square feet square meters square feet square meters metric tonnes pounds (avdp) tons (short) metric tonnes pounds (avdp) tons (long) British thermal units aram-calories
	Tons (US) Tons (US)	2 000.0 * 0.892 857	pounds (avdp) tons (long)
	Watt-hours	3.412 142	British thermal units
	Watts	809.840 3 412 142	gram-calones British thermal units/hour
	Watts	1.0 *	joules per second
	Yards	3.0 *	feet
	Yards	91.440 0	centimeters

## **> TEMPERATURE CONVERSION TABLE**

"Starting Value" is the temperature to be converted. Its value in degrees centigrade (°C) is found to the left and in degrees Fahrenheit (°F) to the right.

°C	Starting Value	°F	°C	Starting Value	°F	°C	Starting Value	°F
-18	0	32	149	300	572	316	600	1112
-12	10	50	154	310	590	321	610	1130
-7	20	68	160	320	608	327	620	1148
-1	30	86	166	330	626	332	630	1168
4	40	104	171	340	644	338	640	1184
10	50	122	177	350	662	343	650	1202
16	60	140	182	360	680	349	660	1220
21	70	158	188	370	698	354	670	1238
27	80	176	193	380	716	360	680	1256
32	90	194	199	390	734	366	690	1274
38	100	212	204	400	752	371	700	1292
43	110	230	210	410	770	377	710	1310
49	120	248	216	420	788	382	720	1328
54	130	266	221	430	806	388	730	1346
60	140	284	227	440	824	393	740	1364
66	150	302	232	450	842	399	750	1382
71	160	320	238	460	860	404	760	1400
77	170	338	243	470	878	410	770	1418
82	180	356	249	480	896	416	780	1436
88	190	374	254	490	914	421	790	1454
93	200	392	260	500	932			
99	210	410	266	510	950			
104	220	428	271	520	968			
110	230	446	277	530	986			
116	240	464	282	540	1004			
121	250	482	288	550	1022			
127	260	500	293	560	1040			
132	270	518	299	570	1058			
138	280	536	304	580	1076			
143	290	554	310	590	1094			

## THERMOCOUPLE SELECTION Cold Junction Compensation

Thermocouples are the most widely used temperature sensors in plastics extrusion. This is because of their reliability, small size, low cost, and ease of interfacing with process monitoring and control instrumentation. A thermocouple consists of two dissimilar metal wires joined together to form a measuring junction. As a result of the different thermo-electric properties of the two metals, a small emf, which depends on temperature, is produced. The magnitude of this emf can be used to measure temperature.

Various metals can be used, the choice of which will determine the size of the emf and the temperature range over which the thermocouple can be used. Standard types of thermocouple's have been specified and identified by a letter by organizations such as ANSI. An example is the the "J" type, which is formed by iron and constantan (copper/nickel alloy) wires.

The thermocouple wires are connected to an instrument that measures the emf produced. This instrument measures the difference in the emf produced by the measuring junction from that of a second junction kept at a constant reference temperature. This reference, or cold junction compensation, was in the past set at 0°C ( $32^{\circ}F$ ) using an ice water bath. It is now simulated electrically.

## Extension/Compensating Cable

Thermocouple wires are generally kept small in order to obtain a rapid response. This, together with their characteristically high resistivities, results in high electrical resistance. That can be a problem when the measuring system is located far from the sening thermocouple. To overcome this problem, special cable is used to convey the signal from the the thermocouple to the measuring device. The correct cable, for use with a specific thermocouple, is identified by a color code on the wire insulation.

## Types of Thermocouples and Color Codes of Their Extension Wires

ANSI Type	+ wire	- wire	Temp. Range	Extension Insulation Color (ANSI)	Comments
E	Nickel- chromium	Copper -nickel	32 to147°F	+ Magenta	High sensitivity
	(Chromel)	(Constantan)	0 to 80°C	- Orange	High resistance
J	Iron	Copper	70 to130°F	+ White	Used in reducing
		(Constantan)	20 to 70°C	- Orange	Atmospheres
К	Nickel	Nickel	32 to 200°F	+ Yellow	Wide range
	(Chromel)	(Alumel)	0 to 110°C	- Orange	Widespread use
Т	Copper	Copper- nickel	-300 to 57°F -185 to 30°C	+ Blue - Orange	Low temps. Moist areas SI. Oxidizing/ Reducing areas
S	Platinum-	Platinum	32 to 290°F	+ Black	High temps.
	Rhodium		0 to 160°C	- Orange	

## EMF (millivolts) Values for a Limited Temperature Range

Thermocouple Type						
Temp. °C	E	J	K	Т	S	
100	6.317	5.268	4.095	4.277	0.645	
120	7.683	6.359	4.919	5.227	0.795	
140	9.078	7.457	5.733	6.204	0.950	
160	10.501	8.560	6.539	7.207	1.109	
180	11.949	9.667	7.338	8.235	1.273	

252

	253
S	
1.440	S
1.611	Ē
1.785	- 3
1.962	ō
2.141	Z
2.323	9
	C
	<u>IS</u>
	Ę
	<b>S</b> 1.440 1.611 1.785 1.962 2.141 2.323

## Insulation Materials for Thermocoup

J

10.777

11.887

12.998

14.108

15.217

16.325

Thermocouple Type

Е

13.419

14.909

16.417

17.942

19.418

21.033

Temp. °C

200

220

240

260

280

300

Insulation Material	Temperature Range	Comments		
PVC	-15 to 220°F	General purpose, for non-aggressive		
	-10 to 105°C	environments. Waterproof. Very flexible.		
PTFE	-105 to 525°F	Mechanically strong and flexible.		
	-75 to 275°C	Good chemical resistance (oils, acids, etc.)		
Glass fiber	-75 to 710°F	Allows ingress of fluids. Reasonably		
(varnished)	-60 to 375°C	flexible, but not mechanically strong.		
Good temperature range.				
High temp.	-75 to 1300°F	Allows ingress of fluids.		
glass fiber	-60 to 700°C	Only moderately flexible and rather weak.		
Ceramic fiber	30 to 1800°F	Resists high temperatures, but will		
	0 to 1000°C	not protect against fluids or mechan- ical disturbance.		
Glass fiber (varnished)	-75 to 710°F	Allows ingress of fluids. Mechani- cally strong.		
Stainless steel over-braid	-60 to 375°C			
Mineral insulated	-100 to 2000°C	Water, oil, and gas tight. Moderately		
Metal sheathed	-70 to 1100°C	flexible. Strong. Resists oxidizing atmospheres. Metal sheath is stain less steel. Insulation is magnesium oxide.		

Κ

8.137

8.938

9.745

10.560

11.381

12.207

## **Conductor Size Equivalents for American Wire Gauge** (Brown and Sharpe)

Gauge	In	MM	Gauge	In	MM
0	0.3449	8.25	13	0.0720	1.83
1	0.2893	7.35	14	0.0641	1.63
2	0.2576	6.54	15	0.0571	1.45
3	0.2294	5.83	16	0.0508	1.29
4	0.2043	5.19	17	0.0453	1.15
5	0.1819	4.62	18	0.0403	1.02
6	0.1620	4.11	19	0.0359	0.912
7	0.1443	3.67	20	0.0320	0.813
8	0.1285	3.26	21	0.02485	0.724
9	0.1144	2.91	22	0.0253	0.643
10	0.1019	2.59	23	0.0226	0.574
11	0.0907	2.30	24	0.0201	0.511
12	0.0808	2.05	25	0.0179	0.455

INFORMATION

254	Gauge	In	MM	Gauge	In	MM
	26	0.0159	0.404	39	0.0035	0.089
	27	0.0142	0.361	40	0.0031	0.079
	28	0.0126	0.230	41	0.0028	0.071
	29	0.0113	0.287	42	0.0025	0.064
	30	0.0100	0.254	43	0.0022	0.056
	31	0.0089	0.226	44	0.0020	0.051
	32	0.0080	0.203	45	0.00176	0.046
	33	0.0071	0.180	46	0.00157	0.040
	34	0.0063	0.160	47	0.00140	0.036
	35	0.0056	0.142	48	0.00124	0.031
	36	0.0050	0.127			
	37	0.0045	0.114			
	38	0.0040	0.102			

## > APPLICATIONS OF RHEOLOGICAL DATA FOR EXTRUSION

## Pressure Drop in a Die

In order to force a polymer melt through an extrusion die, pressure must be applied. In extrusion dies this pressure is usually applied by a screw extruder that pumps material to the die. Pressure is built up by the restricting effect of the die.

## **Simple Die Characteristics**

In the case of a simple die where the cross-section of the channel, whatever its shape, is constant from its inlet to exit, it is found that:

- 1. The pressure is greatest at the die entry.
- 2. The pressure is virtually zero at the die exit (and for most purposes may be equated with zero).
- 3. The pressure drops linearly from the inlet to the exit.
- 4. If the inlet pressure is denoted by  $\Delta P$  and the length of the die is L, then  $\Delta P/L$  is constant along the whole length of the tube.

#### Calculation of Pressure Drop in a Capillary Die

The pressure drop in a die (for which the die cross-section is constant and which is fed from a reservoir of material at the die head) is related to the output rate, the die dimensions, and the melt viscosity. For a simple rod die, the pressure drop is calculated by the following procedure:

- 1. Determine the apparent wall shear rate from the formula  $\dot{\gamma}_{w,a} = 4Q/\pi R^3$ .
- 2. Read off the wall shear stress  $(\tau_w)$  corresponding to this shear rate value from a polymer flow curve appropriate to the extrusion conditions. (Figure 35.)
- 3. Calculate the pressure drop (i.e., the pressure developed at the back of the die parallel) from the shear stress by the formula  $\mathbf{P} = 2\mathbf{L}\tau_w/\mathbf{R}$  where L is the length and R the radius of the die parallel portion of the die.

#### Calculation of Pressure Drop in a Slit

For a slit die the shear rate is given by  $\dot{\gamma}_{w} = 60/wH^{2}$  where w is the width of the slit and H its thickness (or depth). The flow curve, even though it may have been prepared from capillary data, may then be used to read off the corresponding shear stress. In turn the pressure drop is obtained from the expression P =  $2L\tau_{w}/H$ .



Figure 35. Polymer Flow Curves Used for the Determination of  $\tau_{\!w}$ 

#### Calculation of Pressure Drop in a Tapered Capillary Die

In the case of a tapered die, the radius at the inlet is called  $R_1$  and at the outlet  $R_2$ . As with the capillary die, the shear rate is first determined applying the capillary formula using the inlet radius. The shear stress at that shear rate is determined from the flow curve. The pressure drop is then given by: **P** = [2  $\tau_1$ / 3tan0] [R<sub>1</sub>/R<sub>2</sub>)<sup>3</sup>-1].

## **Sizing of Die Components**

During rheology testing, die swell measurements can be made relatively easily. A common use of such measurements is to size die components.

#### **Need for Sizing**

The cross-section of an extrudate will not always be the same as the outlet cross-section of the die from which it has been extruded. There are three reasons for this:

- 1. Shrinkage of the extrudate on cooling.
- 2. Extrudate or die swell.
- 3. Draw down (stretching of the extrudate on emergence from the die).

The magnitude of the shrinkage is usually quite small and since die swell measurements are usually based on cold extrudates this factor is usually built into any die swell assessment. Whereas die swell increases the cross section, drawing down reduces it.

#### **Correction Factors**

The relationship between the cross-section dimension of an extrudate E and the cross-sectional dimension of a die D, is  $\mathbf{E} = \mathbf{sdD}$ .

In this equation, s is the die swell ratio at the wall shear rate occurring during extrusion. **d** is the draw down factor defined by  $\sqrt{(L_a - L_a)}$ .  $L_a$  is the length of material produced in a unit of time in the process in which draw down has taken place.  $L_a$  is the length of material naturally extruded in unit time without any drawing down having occurred. (The square root term arises since it is the cross-sectional area rather than a single dimension e.g. diameter that decreases inversely with the extent of draw-down.)

## 256 Sizing Pipes

A further complication arises in pipe manufacture, where it is common practice to inflate the tube to the inner diameter of a sizing die. In this case, if the outer diameter of the extrusion die annulus is do and that of the inner diameter of the sizing die annulus is ds, then the thickness of the pipe T will be related to the annular gap of the die, A, by the relationship:

 $T = s \sqrt{(L_d / L_u) (d_s / d_o) A}.$ 

## **Estimation of Pressure Drops in Extrusion Dies**

The relationships between pressure drop (P), output rate (the flow rate Q), and viscosity for various flow cross-sections may all be arranged to take the form **P**=  $\eta$  **x R x Q**. Where R is called the die resistance.

### **Die Resistance**

Die resistance is a measure of the resistance of a flow channel to the passage of the fluid. For regular channels of simple shape it is easily calculated from their dimensions. Regular extrudate sections include rod (cylinder), sheet (rectangle), and pipe (annulus). The formulae for calculating the die resistance in each case are as follows:

- 1.Rod or Cylindrical Channel Length = L; Radius = r; Diameter = d. Die resistance = R = 8 (L)/ $\pi$ r<sup>4</sup> = 128 (L)/ $\pi$ d<sup>4</sup>
- 2. Rectangular Channel

Length = L; Width = w; Thickness = t.

- a.) Where width is more than 10 times thickness
- Die resistance = R = 12(L)/wt<sup>3</sup>
- b.) Where width is less than 10 times thickness
- Die resistance = R = 12(L)(S)/wt<sup>3</sup>
- S = Shape factor and is given in Figure 36.
- 3. Pipe or Annular Channel.

Length = L; inner diameter =  $d_1$ , outer diameter =  $d_0$ ,

Gap thickness = t = 1/2(d\_{o}-d\_{i}), Average diameter = d = 1/2(d^{o} + d^{i}),

Die resistance =  $R = 12(L)/\pi d t^3$ 



Figure 36. Shape Factors for Rectangular Dies

#### Calculating Die Resistance (Example Pipe Die Land)

Consider a land of length 50mm, with an internal diameter of 100mm, and external diameter of 104mm. The average diameter is 102mm and the gap thickness is 2mm. Using the formula given above:

Die resistance = R =  $[12 (50)]/[\pi (102.2)^3] = 0.234 \text{ mm}^3$ 

#### Flow Rate

When calculating pressure losses, flow rates must be expressed as volume flow per unit time. If mass flow rate, for a melt of density D at the processing temperature, is known as M(kg/h) then the volume flow rate (Q) in mm<sup>3</sup>/s is **D** = **278.000 (M / D)**.

Values of the melt density (D), for a plastic melt can be determined using a melt flow rate tester. If a melt flow rate tester that can do a Method B test is available the melt density may be measured directly as described in the section on Melt Flow Rate Testing.

#### Method for Plastic Melts

Sicne most molten plastics are non-Newtonian there are two complications in calculating pressure losses. First, the viscosity of molten plastic is not constant and second, the flow pattern in a channel is different from that of a simple Newtonian fluid. (The viscosity of a simple, or Newtonian fluid is a constant, which depends only upon the temperature.) These problems are overcome by using apparent viscosity and representative values.

#### **Apparent Viscosity**

If the shear stress at a particular shear rate is known for a plastic melt then the ratio shear stress/shear rate is called the apparent viscosity. The apparent viscosity decreases as shear rate increases. To calculate pressure losses etc. it is necessary to have a graph of apparent viscosity against shear rate for the molten material at a particular temperature. These flow curves can be obtained from material suppliers, measured in the laboratory (See Figures 8a and 8b and the Section on Rheology Equipment and Testing), or it may be calculated approximately from the MFR of the plastics material. Figure 37 shows a flow curve that will be used as an example here.



LLDPE

Figure 37. Polymer Flow Curves Shown as Viscosity vs. Shear Rate

## 258 Representative Values

For a given overall output rate, the flow pattern of a molten polymer in a channel will differ from that of a simple Newtonian fluid. **Figure 38** shows flow patterns through a tube for a Newtonian and for a non-Newtonian fluid (polymer material). Both materials have zero flow velocity at the walls of the tube and maximum flow velocity at its center. For the same output rate (and hence the same average flow velocity) the plastic melt has a lower maximum velocity and more of a plug like flow. That is, the flow velocity is nearly constant across the middle of the tube and decreases rapidly near the walls. As a result, the shear rate, which controls the localized rate at which layers flow over one another, is greater near the walls for a plastic material than for a Newtonian fluid. Near the walls of the flow channel there can be a large difference in shear rates as shown in **Figure 39**.

There is a fairly complex way of relating Newtonian and non-Newtonian shear rates at the wall called the Rabinowitsch correction. However, it is simpler to consider the situation where the two lines in **Figure 38** intersect. Here the Newtonian and non-Newtonian shear rates are equal. The value of shear rate at this point is called the representative value. By using this value it is possible to calculate pressure drops using a method similar to that applicable to Newtonian fluids (see W. Michaeli in his book Extrusion Dies).





#### **Representative Shear Rate**

It has been found that the position of the representative shear rate in a flow channel is nearly constant for plastic melts. For a tube, it is at 83% of the distance from center to walls. **(Figure 39)**. The formulae for calculating the representative shear rate are as follows:

For a rod die:  $\dot{\gamma}^* = 3.3Q/(r^3)$ 

For a rectangular channel:  $\gamma^* = 4.6 \text{ Q/(w t}^2)$ 

#### For an annulus: $\dot{\gamma}^* = 1.46 \text{ Q/(d } t^2)$

#### Representative Viscosity

Once the representative shear rate is determined, the flow curve for the plastic melt is used to find the apparent viscosity of the melt at this shear rate. This apparent viscosity is called the representative viscosity ( $\eta_{\text{R}}$ ) and if substituted in the equation:  $P = \eta_{\text{R}} R Q$ , where  $\ =$  die resistance and Q = flow rate, it may be used to calculate pressure drops.

#### Pressure Drop Calculation Using Representative Viscosity

Suppose the pipe die whose die resistance was calculated above, extrudes plastic melt at 180 kg/h, the melt has a density of 800 kg/m<sup>3</sup>, and its flow properties are as shown in **Figure 38**.

Flow rate: Q = 278,000 x 180/800 = 62,550 mm<sup>3</sup>/s.

Representative shear rate:  $\dot{\gamma}^* = (1.46 \times 62550)/(102 \times 2^2) = 224 \text{ sec}^1$ From the flow curve, the representative viscosity (@ 230 °C in **Figure 38**) is 940 Pa-s (Ns/m<sup>2</sup>)

Pressure drop: P =  $\eta_{\text{R}}$  R Q = 940 x 0.234 x 62550 = 13760000 Pa (or N/m<sup>2</sup>) = 13.76 MN/m<sup>2</sup> (or 2005 psi)



Figure 39. Shear Rates in a Die for a Newtonian Fluid and Polymer Melt

#### Flow Resistance of Complete Dies

Most dies can be considered as a number of connecting sections. The die resistance depends mostly on the smallest dimension of the channel crosssection (for example, the thickness of a slot die) or on the gap thickness of a pipe die. Hence flow resistance is usually greatest across the die land. First calculate the die resistance and pressure drop of this section of the die. Only if this is satisfactory is it necessary to calculate pressure drops for the other die sections. Total pressure drop is the sum of the pressure drops for each section.

MATERIAL PROPERTY GUIDELINES							
Property	ASTM Method	ABS	Nylon 6	Nylon 66	РММА		
Specific Gravity (g/ml)	D792	1.02-1.06	1.13	1.13-1.15	1.17-1.20		
Tensile Str. (psi)	D638	3500-7800	7250	11,500	8000-11000		
% Elongation	D638	30-90	200	60-300	2-7		
lensile Modulus (10⁵psi)	D638	1.4-3.6	2.18	3.8-4.7	3.5-4.5		
Impact Str.(ft-lb/in) 1/8" notched Izod	D256	2.6-11.4	1.8	1.0-2.0	0.3-0.4		
Hardness (Rockwell M or R, Shore A or D)	D785	R75-105	M86	R120	M80-100		
T <sub>g</sub> (°C)	D3418	100-110	-	50	105		
T <sub>m</sub> (°C)	D2117	-	220	265	-		
Heat Deflection T (°C)	D648						
66 psi		77-115	>160	105	74-113		
264 psi		77-105	55-75	244	72-102		
Thermal	C177	4.5-8	5.8	5.8	4.0-6.0		
Conductivity							
(10 <sup>-₄</sup> cal/sec cm°C)							
Specific Heat @ 23°C (cal/(g°C)	-	0.3-0.4	0.10	0.11	.035		
Thermal Exnan	D696	9 5-13	45	8.0	5-9		
Coeff.	2000	0.0 10	1.0	0.0	00		
(10 <sup>-5</sup> in/in°C)							
Dielectric	D150						
Constant							
60 Hz		2.4-5	-		3.5-4.5		
1 kHz		2.4-4.5	-	5.2	3.0-3.5		
1 MHz		2.4-3.8	7.0	4.1	2.1-3.2		
Dissipation	D150						
Factor				0.04	0.05.0.00		
60 HZ		0.003-0.008		0.04	0.05-0.06		
		0.004-0.007	0.02	0.04	0.04-0.00		
I IVINZ Flectrical		0.007-0.015	0.03	0.05	0.02-0.03		
Resistivity	D257	1-5 x 1016	-	10 <sup>13</sup>	10 <sup>15</sup>		
Water Absorption	D570	0 20-0 45	13-19	11-15	0 02-0 04		
(%/24hr-1/8" thick bar)	2010	0.20 01.0			0.02 0.01		
Property	ASTM Method	LDPE	LLDPE	HDPE	РР		
Specific Gravity 0.90-0.94	D792	0.900-0.925	0.918-0.93	5	0.941-0.955		
(y/IIII) Tensile Str. (nci)	D638	600-2300	1000-/000	3100-5500	4500-6000		
% Flongation	D638	90-800	100-950	30-1300	100-600		
Tensile Modulus	D638	0.14-0.38	0.38-0.75	0.6-1.8	1.65-2.25		
(10⁵psi)							
Impact Str.(ft-lb/in)	D256	No break	1.0-9.0	0.5-20	0.4-1.2		

Property	ASTM Method	LDPE	LLDPE	HDPE	PP
1/8" notched Izod Hardness (Rockwell M or R, Shore A or D)	D785	R10 D41-46	-	D60-70	R80-102
$T_{\alpha}$ (°C)	D3418	-126	-126	-126	-20
T <sub>m</sub> (°C)	D2117	106-112	125	135	168-175
Heat Deflection T (°C)	D648				
66 psi 264 psi		38-49 32-41	6-66 -	60-88 43-54	107-121 49-60
Thermal Conductivity	C177	8.0	-	11.0-12.4	2.8
Specific Heat @ 23°C (cal/g°C)	-	0.55	-	0.55	-
Thermal Expan. Coeff. (10 <sup>-5</sup> in/in°C)	D696	10-20	8.9-11	11-13	8.0-10
Dielectric Constant	D150				
60 Hz 1 kHz		2.25-2.35 2.25-2.35	2.3 -	2.30-2.35 2.30-2.35	2.2-2.8
Dissipation Factor	D150	2.20-2.30	-	2.30-2.35	
60 Hz 1 kHz 1 MHz Electrical		0.0005 0.0005 0.0005	0.0005	0.0005 0.0005 0.0005	0.0005- 0.001
Resistivity (ohm-cm)	D257	1016	1016	10 <sup>16</sup>	1016
Water Absorption (%/24hr-1/8" thick bar)	D570	0.015	0.01	0.01	0.01-0.03

Property	ASTM Method	PC	TPU
Specific Gravity (g/ml)	D792	1.2	1.11-1.28
Tensile Str. (psi) % Elongation	D638 D638	8000-9500 100-130	1500-11000 110-1000
Tensile Modulus (10 <sup>5</sup> psi)	D638	3.0-3.5	0.33-1.5
Impact Str.(ft-lb/in) 1/8" notched Izod	D256	12-18	-
Hardness (Bockwell M or B	D785	M73-93 B115-125	A50-95 D46-80
Shore A or D)			010 00
T <sub>g</sub> (°C)	D3418	150	-51
T <sub>m</sub> (°C)	D2117	-	Varies
Heat Deflection T (°C)	D648	122-142	Varias
00 μ31		102-140	vai 165

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262	Property	ASTM Method	PC	TPU			
	264 psi		130-140	Varies			
	Thermal	C177	4.6	-			
	Conductivity						
	(10 <sup>-4</sup> cal/sec cm°C)						
	Specific Heat @ 23°C	-	0.3	-			
	(cal/g°C)						
	Thermal Expan.	D696	6.8	-			
	Coeff.						
	(10⁻⁵ in/in°C)						
	Dielectric	D150					
	Constant						
	60 Hz		2.97-3.17	-			
	1 kHz		3.02				
	1 MHz		2.92-2.93				
	Dissipation	D150					
	Factor						
	60 Hz		0.0009	-			
	1 kHz		0.0021				
	1 MHz		0.0100				
	Electrical						
	Resistivity	D257	2 x 10 <sup>16</sup>				
	(ohm-cm)						
	Water Absorption	D570	0.015-0.18	0.3			
	(%/24hr-1/8" thick bar)						

# > GLOSSARY OF TERMS USED IN EXTRUSION TECHNOLOGY

**Accelerator:** A chemical added to speed up a reaction; sometimes associated with cross-linking.

Accumulator: A device used to store hydraulic fluid under pressure or a part of the processing equipment (for example, in blow molding) where melt is stored.

**Activator:** A chemical added to activate or start a reaction; usually associated with cross-linking where an activator is used with an accelerator.

**Adapter:** Also spelled adapter. Connects the extruder to the die and directs the melt into the die. Used to attach the dies to the machine. May also change the direction of melt flow. May have angled crosshead, and offset adapters.

Adaptive Control: A control system that automatically changes settings in response to changes in machine performance, so as to produce products of the specified quality. A machine is said to have adaptive control if it 'adapts' itself to meet a change.

Adiabatic Extrusion: After the extruder has been warmed up no external heat is supplied to the machine. The heat necessary to plasticize the polymer therefore comes from conversion of the mechanical energy used to rotate the screw.

**Air Gap:** The vertical distance between the die lips and the nip (for example, in extrusion lamination).

**Air Shot:** If the contents of an extruder are shot, or expelled, into the air, then this is referred to as an air shot or, as an air purge.

**Airflow Switch:** A switch fitted on the exit side of the cooling circuit of an electric motor which prevents motor overheating if the air flow fails; useful where powder is processed.

Amorphous: Usually refers to a plastic material that has no regular molecular structure (is not crystalline). These materials are usually transparent (for example, PS or PC).

**Anglehead Adapter or Adapter:** During anglehead extrusion, the adapter swings the melt through 45 or 600.

**Annealing:** Heating of a product to condition it and/or to relieve stresses. **Anti-aging:** Chemicals added to hinder degradation, for example, by oxidation. **Anti-block Agent:** An additive used to stop blocking, or sticking, of two surfaces. Usually associated with film. May be dispersed in the resin before extrusion or dusted on after extrusion.

Antioxidant: A chemical added to hinder the chemical reaction known as oxidation (oxygen attack). Usually associated with materials such as PE, or PP that readily degrade in this way.

Antiozonant: A chemical added to hinder ozone attack. Usually associated with materials such as natural rubber which readily degrade in this way.

Antistatic Agent: Additives added to reduce static problems. May be a dispersed ionic compound that blooms to a surface (e. g. of a film) and forms a conductive layer.

**Apparent Density:** This is the weight in air of a unit volume of material. **Apparent Viscosity:** Defined as the ratio of the shear stress to the shear rate obtained for a non-Newtonian fluid, such as a plastics melt, assuming that it behaves like a Newtonian material.

Appearance Surface: The surface that is seen in use and, therefore, has to be of a specified standard.

**Applesauce:** A rough, wavy appearance of the extrudate; also referred to as orange peel, shark skin or flow patterns.

**ASTM:** American Society for Testing and Materials.

Autoflex Die: Sheet die in which the lip opening is controlled by the expansion of thermal bolts that respond to thickness sensors.

Automatic Screen Changer: Unit which automatically removes one screen pack from the melt stream path and replace it with another when the measured pressure, reaches a pre-set value.

**Axial Grooved Bushing:** A bushing that contains a number of grooves (for example, six) which run parallel to the screw. This grooved bushing, fitted in the feed section of an extruder, improves the feeding characteristics of the system. The bushing is usually 3 to 4 D (where D is the diameter of the screw) in length and the axial grooves taper out as the material progresses along the bushing. Temperature control of the bushing is important and it is held at of 70 °C by intensive circulation of water. To decrease heat generation, a zero compression screw may be used.

**Barrel:** The extrusion cylinder that contains the screw and the plastic material. Generally referred to as the extruder barrel.

**Barrier Layer:** A distinct layer of material in an extrudate that stops, or hinders, the passage of a liquid or gas. The barrier layer may be a plastic material or a metal.

**Barrier Screw:** A two-start screw that is designed to continually separate the melt from the un-melted plastic material. It subjects the un-melted material to high shear forces. The two melt streams that result are combined and passed through a mixing head to ensure that the melt is of uniform temperature.

**Beta-ray Gauge:** Also known as a  $\beta$ -ray gauge. A thickness measuring device in which  $\beta$  ray absorption is measured. It is directly proportional to product thickness, for a given formulation.

**264 Bi-axial Orientation:** A process for the improvement of tube or sheet properties by stretching the material in two directions at right angles: along and across the extrusion direction.

**Bi-axial:** Usually refers to directional orientation along two different axes (for example, biaxial orientation).

**Bleeding:** Migration of a colorant into from one material into another. **Blocking:** The tendency of sheets or films to stick together under light pressure making separation difficult. Reduced by using non-gloss surfaces or by the use of anti-block agents.

**Blooming:** Term used when a colorant or other material migrates to the surface of a product and forms a dusty layer. Dyes are more susceptible to this than pigments.

**Blow Molding:** A process used to produce hollow enclosed components, such as bottles, by expanding an extrudate (a parison), by air pressure, inside a mould.

**Blow Up Ratio:** The ratio of the diameter of the bubble to the die diameter in blown film extrusion.

**Blowing Agent:** An additive that expands a plastic melt by generating a gas. It is often a solid chemical which decomposes at extrusion temperature (for example, an azo compound).

**Blown Film Extrusion:** A process for making plastic film (also known as tubular film or, lay-flat film) by extruding a tube and using air pressure to blow it up to several times its extruded diameter.

**BOPP:** Bi-axially orientated polypropylene.

BPF: British Plastics Federation.

**Breaker Plate:** A disc, or plate containing a number of holes oriented in the extrusion direction through which a plastic material flows. It is usually used to support screens and is placed between the end of the extruder barrel and the die holder.

**Bridged Screw:** A screw that will not transport material because an arch, or bridge, of material has formed in the hopper.

BSI: British Standards Institution.

**Bulk Density:** The weight per unit volume of a material including voids that are inherent as tested.

Burn Mark: A region of burned or degraded material.

**Bushing:** A removable cylindrical lining used to limit the size of the opening, resist abrasion, or act as a guide.

**Buss CoKneader:** A family of reciprocating-type mixing extruders originally made by the Swiss firm of Buss AG. A single-screw continuous compounder in which each turn of the screw is accompanied by a back-and forth movement of the special screw. Kneading pins, or teeth, protrude from the barrel into three gaps, or slots, running along the length of the screw. To smooth out the pulsating output from such a machine, the mixed material may be discharged into a separate discharge extruder that forms the polymer into strands which are cut into pellets. It is widely used in the polyvinyl chloride (PVC) industry as both the screw and barrel are available with liquid heating (which gives accurate temperature control).

**CAD/CAM:** Computer Aided Design and Computer Aided Manufacturing. **Calender:** A machine used to produce film or sheet at very high speeds and high quality by passing hot resin through a series of rolls.

**Calenderette:** A term sometimes used to denote a series of rolls which are used as the haul off in sheet extrusion or a series of rolls used to produce sheet.

**Capacitive Sensor:** A sensor that uses the extrudate as a dielectric in a circuit to indicate thickness or the proximity of something to the extrudate.

**Capstan:** Large drum device used to pull extrudates, such as wire or cable, by wrapping the extrudate around the rotating drum.

**Cartridge Heater:** An electric resistance heater in the form of a circular rod that may be inserted into a die block.

**Cascade Control:** The output of one controller is used to control, or feed, another.

**Cascade Extrusion:** A type of twin screw machine where two extruders are connected so that the melt output from the first feeds the second.

Devolatilization may be done between the two extruders.

**Cast Film:** A film made by depositing a layer of plastic, either molten, in solution, or in dispersion, onto a surface where it is solidified and then removed.

**Casting:** A flat film process whereby a film is extruded into water or, against a water-cooled roll.

**Cellular:** The density of the resin is reduced by expansion with a gas or vapor. **Chalking:** Surface degradation of the plastic that exposes the pigment. May be confused with blooming.

**Check Valve:** The valve usually used in foam extrusion to control back-flow of the blowing agent in the gas injection method of foam extrusion.

Chill Roll Casting: A plastic film is extruded from a slit die directly onto a roll, which is cold compared to the melt temperature.

**Chill Roll:** The 'cold' roll against which a plastic film is cast after being extruded from a slit die. The chill roll temperature may be relatively high, for example.  $175^{\circ}$ F or 80°C. but much it is lower than the melt temperature.

**Choke Bar:** The portion of sheet die that is used to selectively restrict the polymer flow to even out variations in sheet thickness. It forces the material out to the die extremities.

Choke Plate: A single hole unit that is inserted between the end of the extruder barrel and the die holder to produce a controlled pressure drop in the melt.

**Clam Shelling:** An extrusion defect caused by die body deflection at the center of the die, which causes more material to be present at the center of a flat sheet die.

**Cling Film:** A name given to a film which can either cling, or stick, to itself or to other objects, which is widely used in packaging. Thin, soft films based on soft PVC or EVA show this property.

**Closed Loop:** Control terminology used when the control system checks to see if its commands have been obeyed and then enforces them if they have not.

**Closed Loop, Dimension Control System:** A system that feeds back information on the product dimensions and then adjusts parameters, such as line speed, to correct for dimension shifts.

Coating: Coating is the process of applying a molten polymer to a moving substrate.

**Coextrusion Capping:** The extrusion process used to produce a plastic product which is topped or capped with another plastic.

**Coextrusion:** An extrusion process where two, or more, melt streams are combined in the die to make an extrudate of two, or more, layers of plastic. One of these layers is often a barrier layer.

**Cold Flow:** Refers to the flow of material at less than the optimum melt temperature.

**Colorant:** A material added to impart color that is usually a dye or a pigment. **Compound:** The result produced by the melt mixing of a plastic resin, additives and/or fillers.

**Compounder:** An extrusion machine used specifically for melt mixing. **Compounding Extruder:** An extrusion machine used specifically for melt mixing. **266 Compounding:** The process of melt mixing used to produce a compound. **Compression Ratio:** The ratio of the volume of one screw flight, at the feed end of a machine, to that of one flight at the discharge end. The channel depth often changes along the length of a screw used in plastics processing. It is deepest under the hopper and shallowest at the screw tip. Thus the screw has a compression ratio which is there to compensate for the effective reduction in volume that occurs on melting the plastics granules. The compression ratio is related to channel depth so if the depth is 0.373" under the hopper and 0.125" at the tip, then the compression ratio is said to be 3:1. Typically general purpose screws have a compression ratio of approximately 2.0/2.5:1 and a length to diameter ratio (L/D) of about 20:1.

**Compression Zone:** A compression zone is present on many extruder screws (at the discharge end) where its presence results in melt pressure generation. It is a parallel portion of the screw, with a shallow depth, which produces a stable melt in terms of melt temperature, melt uniformity and melt output. In blow molding, it is that part of a mould which is also called a dam: the dam forces material back into the pinch area, on mould closing, and thus leads to localized thickening in this weak area.

**Computer Control:** A mode of machine operation where the extruder and line are under the control of a process computer that sets the parameters for the operation.

**Conical Transition Section:** Also called a conical tapered section. It is a transition section of a screw in which the root of the screw increases uniformly in diameter so that the section is of conical shape.

**Constant Depth Screw:** Also known as a parallel screw, it is a screw in which the channel depth is constant along its length. Such a design is sometimes used in blow molding machines as such a design minimizes heat generation.

**Continuous Compounding Machine:** A machine that is designed, or operated, to give a continuous output of well-dispersed material. A compounding extruder is the most common type of continuous compounding machine. However, for example, for very heavily filled materials, other systems may be preferred. Such a machine is often a modular compounding machine.

**Continuous Kneading and Extrusion Machine:** A modular compounding machine comprised of dosing, kneading, conveying and discharge sections. The dosing and kneading sections are housed in a steel block that contains eight inter-connecting chambers each containing a pair of kneading tools. The polymer plus additives are fed in at one end and additional materials may be added to the chambers as the mix progresses through the system. The dwell time can be controlled in each chamber by means of adjustable baffles. To obtain a continuous output, the mixed material is discharged through a (10 D) twin screw extruder.

**Control Loop:** The signal circuit that provides feedback information for closed loop process control.

**Control System:** The instruments, and power controlling units, which are used to maintain the machine temperatures, pressure, rate, and other extrusion parameters at set values.

**Controllers:** The individual, or dedicated, instruments used to control temperature, speed, and pressure in the extrusion operation.

**Cooling Fixtures:** Devices, with air or water cooling, for holding and setting the extruded shape.

**Co-ordination:** This means that a group of controllers are connected together so that they may all be changed, at the same time, from a single point. It is often applied to drive systems. For example, the speeds of the feeder, the extruder and the haul-off may all be ramped up or down simultaneously. **Copolymer:** A polymer, or resin, made by polymerizing two or more monomers together (For example, EVA.). The second monomer is added to improve properties such as adhesion, impact, HDT, etc.

**Core Pin:** The center unit, of a tubing die, used to form the inner wall of the extrusion: also known as a torpedo.

**Corona Discharge:** An electrical treatment applied to the surface of plastic products to enhance printing or adhesion.

**CPU:** Central Processing Unit: part of a computer (see microprocessor).

**Cracking:** Can mean the production of simple chemicals from complex ones by heating or the appearance of fine cracks, or crazes, in a product.

**Crammer Feeder:** A hopper mounted unit that forces plastic material into the feed throat of the extruder.

**Crazing:** Apparent fine cracks at or under the surface of a plastic: See Cracking.

**Creep:** The time-dependent part of strain, or deformation, resulting from an applied stress, or force.

**Cross Laminate:** A laminate in which some of the layers of material are oriented at right angles to other layers. Used to make strong film for packaging. Cross-flow: Flow in an extrusion die at right angles to the primary flow direction.

**Cross-head Adapter:** In cross-head extrusion, the adapter turns the melt direction through 900.

**Cross-head Extrusion:** An extrusion process wherein the extrudate comes out of the machine at right angles to the barrel axis. Used in wire covering processes and in some tubing lines.

**Cross-linking or Cross Linking:** A chemical reaction where plastic molecules are linked or tied together to form molecules with increased the molecular weight and viscosity. Many gels result from the cross-linking in plastics.

**Cryogenic Internal Bubble Cooling:** An internal bubble cooling system that can improve the output by approximately 50% (over conventional internal bubble cooling (IBC). The chilled air (approximately 10°C), which is used to cool the internal surface of the film during IBC production, is replaced by much colder nitrogen gas (at B150 °C). Before the cold nitrogen is used, the entire system is purged (with warmer nitrogen) to remove any moisture-laden air which will cause condensation on the film.

**Crystalline:** Refers to plastics that contain ordered molecules, resulting in crystalline areas or zones of crystallinity. Such materials are also referred to as 'semi-crystalline thermoplastic materials' since their structure is not completely ordered.

**Crystallinity:** The amount (usually expressed as a percentage) of a polymer which is ordered or consists of crystalline zones or areas.

Cuff Heater: Another term for heater band.

**Cut Off Units:** Devices such as saws, shears, flying knives, and other devices for cutting extrudates to predetermined lengths.

Cylinder: Another word for barrel.

D: See Derivative. Also may be the symbol for diameter, as in L/D.

**Dancer Roll:** A roll used on a sheet extrusion line to control the sheet (line) tension that compensates for line speed variations.

DC: Direct current.

**DC Drive:** Direct current drive. It is the standard, electric motor, drive system for feeders, extruders and pullers since they give good speed consistency.

**Dead Band:** That part of a temperature controller range in which the control is not affecting the temperature. The power is off and the temperature is allowed to drift.

**Dead Fold:** The ability of a film or foil to be creased without heat and to retain that crease as shown by metal foils.

**268** Deckle Rods: Metal inserts that are used to close off the ends of a flat film die to allow the extrusion of narrower product.

Decompression: The action of reducing melt pressure.

**Decompression Zone:** A zone or region in a vented barrel where the melt is decompressed to allow the release of volatile matter (gas). It is used, for example, to dry material.

**Degradants:** Substances added to promote the breakdown of a product. For example, starch may be added to film so that it degrades when discarded. **Degradation:** A breaking down (or reduction) of the chemical structure, physical properties, or appearance of a plastic.

Delamination: The separation of a product into layers.

**Denier:** A unit of weight used for textiles (an abbreviation is den). Used to measure the fineness or coarseness of fibers. The weight, in grams, of 9,000 meters of the fiber or yarn. To convert to kg/m multiply by 1.111 x 107. To convert to tex multiply by 0.111.

**Density:** Mass per unit volume. Apparent density is the weight in air of a unit volume of material. Bulk density is the weight per unit volume of a material including voids which are inherent as tested.

**Depolymerization:** The breakdown of the polymer into its monomers. The opposite of polymerization.

**Depth:** Screw terminology meaning the perpendicular distance from the top of the screw thread to the root.

**Derivative:** Referred to as 'D'; this term is added to a controller in order to prevent overshooting.

**Desiccant Drier:** A type of hot air drier in which the moisture is removed from the air by means of a desiccant such as silica gel.

**Desiccation:** The removal of moisture.

**Devolatilizing Extruder:** An extruder designed, or operated, to remove volatiles or moisture from a polymer when it is in melt form.

**Die Air Vent:** A passage in a pipe or profile die used to permit the passage of air into the interior of the hollow extrusion.

**Die Cart:** A trolley that supports the die and holds it at the correct height for machine connection.

**Die Entry Angle:** The angle of convergence of melt entering the extrusion die. **Die Gap:** The distance, or separation, between the die lips.

Die Land: The straight section through an extrusion die.

**Die Spider:** A legged unit used to support the torpedo (The die section in the melt stream that forms the interior of a hollow section.).

**Die Swell:** The increase the dimensions of an extrudate over those of the die through which it was extruded. DS = part dimension/die dimension.

Die: An orifice used to shape a plastic melt.

**Digital Setting:** The setting of a parameter to a precise numerical value.

Usually done by means of thumb-wheels or a key pad.

**DIN:** A German test method or standard issued by the Deutscher Normenausschuss.

**Discharge Extruder:** An extruder that accepts the output from another machine (usually a compounding machine). It is used, for example, to smooth out a pulsating output from a Co-Kneader. The mixed material is discharged into a short, single screw extruder, or discharge extruder, which forms the output into strands that are cut into pellets.

**Dispersion Coating:** A plastic, dispersed in a liquid, is applied to a film and the liquid is evaporated to leave a coating. This method is used when the coating resin cannot be melt processed or dissolved.

Distortion: An unwanted change in extrudate shape.

**DNA:** Deutscher Normenausschuss; the organization responsible for issuing DIN standards.

**Doser:** A device that meters/doses a resin, and/or additives, into the extruder barrel (Usually called a feeder).

**Double Parallel Screw:** (Also known as a stepped screw or as a nylon screw) A screw in which two parallel section (feed and metering) connected by a tapered transition zone. This tapered transition zone is usually very short for nylon 66 extrusion since the polymer has a very sharp melting point. It is claimed that the rapid decrease in channel depth (step) acts as a barrier to forward flow of non-melted granules.

**Downstream Equipment:** The auxiliary units used in an extrusion line, after the die, which are used to cool, shape, control and, if necessary, cut the extrudate.

**Drag Flow:** Flow of molten polymer in an extruder caused by the relative motion of the screw rotating in the barrel. Mechanism by which material is caused to flow in a single screw extruder and build up pressure. The polymer must wet both screw and barrel to produce drag flow effects.

**Draw Down (See Drawdown):**The stretching of an extrudate to produce a product of the correct size.

**Draw Ratio:** The die gap thickness divided by the thickness of the final plastic layer. The degree to which an extrudate has been drawn in a particular direction.

**Draw Resonance:** A form of draw down failure characterized by the wandering or wavering of the edge of a molten film or by the loss of thickness uniformity across the width of a coating.

**Drawdown:** A process for stretching the extrudate after it leaves the die by it pulling faster than it is being extruded. In extrusion coating the resin is drawn down from the die gap thickness to the coating thickness.

Drawing: The stretching of an extrudate to produce orientation.

Dribbling: See Drooling.

**Driven from the Back:** An extruder fitted with an axial grooved bush. An extruder fitted with a gear pump is said to be driven from the front.

**Driven from the Front:** An extruder fitted with a gear pump. An extruder fitted with an axial grooved bush is said to be driven from the back.

Drooling: An undesirable loss of melt from an extruder die.

Droop: See Offset.

**Dry Blend or Dry Blend:** A dry compound prepared without heat fluxing or the addition of solvent. It is also known as a powder blend and is usually prepared in a high-speed mixer.

**Dual Durometer Extrusion:** Extruding a shape with a soft and a hard material. Sometimes based on PVC materials. A form of co-extrusion.

**Dual Strand Extruder:** An extruder fitted with more than one die to obtain high outputs without speeding up the rate of extrusion (for example, window profile). If extrusion rates are increased problems associated with dimensional tolerances, shrinkage, surface finish and plate out become more severe. The extruder is generally fitted with two mirror image profile dies.

**Dulmage Mixing Section:** A dispersive mixing section. The barrier section is a cylindrical shearing section, of approximately three screw diameters (3 D) in length, which forms the screw tip. It consists of a series of semicircular grooves machined in the screw tip which run, on a long helix, in the same direction as the screw flight. An extrusion screw, may have more than one Dulmage mixing section separated by conventional screw sections.

**Durometer:** A device for measuring the hardness, or resistance to indentation, of a material.

**Dye:** A chemical colorant that is soluble in resin and which can, therefore, produce transparent colors.

Early Flow Tuning: (Abbreviated EFT) A technique used in the extrusion of flat

270 sheet to control the melt flow profile in the pre-land section of the die. A flexible, stainless steel membrane is incorporated in the pre-land section of the die that can be finely adjusted to control the melt flow across the die.

**Edge Bead:** The build up in thickness at the edges of an extruded film that is caused by neck in. The cross section of an edge bead is teardrop shaped and tapers into the film. Internal reduction rods may be used to minimize edge beads.

**Edge Tear:** Partial tearing, or ripping, of a film along its edge that is also known as 'nip in'. The line speed and the extruder output rate, at which edge tear occurs, determine the minimum coating thickness.

Elastic Melt Extruder: An extruder, based on twin discs, which uses polymer melt elasticity effects to convey the polymer melt through the system and out of the die.

**Elastomer:** Also known as rubber. A macromolecular material that at room temperature returns rapidly to approximately its original dimensions and shape after substantial deformation by a weak stress. In doing so, it releases that stress and springs, or snaps, back.

**Electro-Discharge Machining (EDM):** A method of shaping metal by a process of spark erosion.

**Environmental Stress Cracking:** (Also known as ESC) Failure or cracking of a material or product under stress while in a certain environment. The stress may be external or internal (developed as a result of processing) while the environment is often a liquid (such as a detergent or other chemical agent). **Ethylene Plastic:** Plastics based on polymers or copolymers of ethylene; the

ethylene being in present in the greatest amount by weight. Extrudate: The shaped material exiting from the extrusion die.

**Extruded Foam:** An extruded product produced when a plastic mass is expanded by the formation of gas cells.

**Extruder Barrel:** Section of extruder in which the extruder screw turns. It is also known as the 'extrusion cylinder' or 'barrel'.

**Extruder Feedback Control System:** A method of controlling extruder output by the adjustment of speed or back pressure to maintain a constant output rate.

**Extruder Screw:** The unit used to propel plastics through a plastics extruder and to generate pressure on the melt.

**Extrusion Blow Molding:** A blow molding process in which the parison, or tube, is produced by extrusion.

**Extrusion:** Process for making a product by forcing material through a die. **Extrusion Valve:** An adjustable restriction in the melt stream that is used to control back pressure in an extruder.

**Feed Port:** The entry hole in an extruder barrel through which the plastic material is introduced.

**Feed:** Usually applies to the material being introduced into to the extruder. It is often a mixture of virgin and regranulated material.

**Feed Zone:** The barrel zone adjacent to the location of the hopper. This term is also used to describe the first zone of a screw that conveys the material from the feed port or hopper, through the feed zone of the barrel. **FFS:** Form-fill-seal.

Filler: A material, usually a solid, which is added to plastics in order to enhance their properties or to cheapen them.

 $\ensuremath{\textit{Film:}}$  Thin sheet. Usually applies to thicknesses of 0.010 inch (0.25 mm) or less.

Fines: Plastic material (resin) which is in a fine powder form.

Finite Element Analysis: A mathematical procedure used to quantify flow and stress effects by dividing the analyzed space into discreet "finite" elements

that are related to each other by constitutional equations. The method uses computer techniques to analyze stresses and flows in the absence of exact solutions.

**Fish Eyes:** Defects in an extrudate (usually film), which look like the eyes of a fish, which are caused by small particles of un-melted or cross-linked resin.

They are small globular masses that have not blended completely into the surrounding material.

Flame-Retardant: A substance added to hinder the burning of plastic materials.

Flaming: The action of applying a flame to a plastics surface to make that surface more receptive to printing inks.

Flash: Excess material around the edge of an extrusion or molding.

Flight: The space enclosed by the screw thread and the surface of the screw root, in one complete revolution or turn.

Fogging: The increase in haze of a film due to condensation.

Foil: Term sometimes applied to very thin, but very stiff, sheet.

Form and Fill: A packaging process in which the container is formed and the contents inserted, on line. It is used in blow molding, for example, to get high speed production by the rapid cooling which results when cold liquid is injected into the bottles.

Form-Fill-Seal: (Also known as FFS) A packaging process in which a film is formed into a container, filled and then sealed.

Forming Box: A die extension, which is cooled, used for sizing and cooling products such as rod. Melt is pumped into the forming box and solid rod slowly emerges.

Free Extrusion: Extrusion into a cooling unit without the use of forming, controlling fixtures, or sizing devices.

Freeze Line: The location on a blown film bubble where a semi-crystalline, thermoplastics material (usually a polyolefin) starts to crystallize, as it cools. Frictional Heating: Heat generated mechanically within the extruder by turning the screw.

Frost Line: (See Freeze Line).

Frost Line Height: (Abbreviated as FLH.) The distance measured between the frost line and the die.

**Gate:** A valve, for example, the slide at the base of the hopper used to stop and start the flow of material or part of the die. A hinged gate may be used to attach an adapter to the extruder.

**Gauge Bands:** (Also known as 'piston rings') Regions of increased thickness on a roll of film produced through winding thicker film in the same place or on the same part of the reel. May be eliminated by rotating the die.

**Gauge or Gage:** An instrument in which a measured value is displayed or a measure of thickness (100 gauge is 1 mil or 0.001 in).

**Gear Pump:** A type of extruder where the melt is conveyed (pumped) by intermeshing gears. It may be fitted to the discharge end of a conventional extruder to give a more stable output.

Gear Pump Extruder: An extruder which uses intermeshing gears to pump molten plastic or resin.

**Gel:** A semi-solid consisting of a solid network in which liquid is held. In extrusion the term gels usually refers to lumps seen in film which result from resin degradation or cross-linking.

**Gelation:** A term used in PVC technology to define the point at which PVC particles will gel, or fuse, under the combined effects of heat and pressure. The term is also used to describe the setting (or hardening) of a PVC plastisol on heating.

**Gelled:** Term used to describe PVC after the particles have been fused under the combined influence of heat and pressure.

**272 Glass Transition:** A reversible change in an amorphous material to or from a solid to a rubbery (or brittle to flexible) state. Also occurs in the amorphous regions of a semi-crystalline thermoplastic material.

**Glass Transition Temperature (Tg):** The approximate mid-point of the temperature range over which the glass transition takes place.

**Godet Unit:** A multiple roll drive unit that is usually used in the production of monofilaments and strip. The extrudate is drawn along by friction between the extrudate and the rolls. It is commonly used to orient monofilament.

**Graft Copolymer:** A copolymer in which side chains of one composition have been attached (or grafted) to a polymer of a different composition.

**Granulator:** A machine that grinds up plastic for re-use. It does not necessarily produce regular granules.

**Granules:** Plastic raw material supplied in the form of small pellets approximately 0.12 in. or 3 mm.

**Guide Tip or Guider Tip:** Part of a torpedo through which wire passes and emerges into the melt.

**Gum Space:** The distance between the guide tip of a torpedo and the die. **Gusset:** The folded-in portion of flattened tubular film. Also, a piece of material used to give additional size or strength to a component.

Halogen: A group of chemical elements including fluorine, chlorine, bromine or iodine.

Halogen Containing: A material, plastic or additive that contains halogen atoms.

**Hang Up:** A problem associated with irregular, or uneven, flow through a die. It may be caused by dirt, uneven temperature or poor die design (For example, by a lack of streamlining).

**Haul Off:** A system that pulls an extrudate away from the die. It is usually a series of rolls or a caterpillar haul off.

**Haze:** The scattering of light due to occlusions, crystallinity, microvoids, cracks, gel particles, or suspended matter that reduces the transparency of film to visible light.

Head and Gate End: The die end of an extruder.

Heat Sealing: Technique for sealing film by the application of heat.

**Heat Stabilizer:** A material that hinders thermal degradation. Frequently associated with PVC formulations.

**Heater Band:** An electrical, heating element in the form of a sleeve or cuff. **Helix Angle:** The angle between the screw thread and the transverse plane of the screw.

HF: High (or radio) frequency.

**High Frequency Welding:** High frequency vibrations transmitted into plastics are used to melt and fuse parts together. This process can be used to weld many plastics, but is peculiarly suited to PVC, as this resin is polar.

**Hold-up Time:** (Abbreviation: HUT) The amount of time, usually measured in minutes, for which a material is resides within a barrel. It may also be referred to as barrel residence time. In extrusion and injection molding, the barrel may be heated to relatively high temperatures. The HUT is important as the use of such high temperatures can easily cause oxidation or degradation of a thermoplastic material. Therefore, it is important minimize residence times in the barrel, particularly at high melt temperatures.

Homopolymer: A polymer made from one monomer.

**Hopper:** The material holding device, attached to the extruder at the feed port, used to hold, and then feed, plastic material into an extruder.

**Hub:** The part of the screw that functions as a seal to prevent plastic from leaking back into the drive. It has the same diameter as the flight.

**Hunting:** Control terminology that means that an actual value is moving around a set point. It is usually applied to on/off temperature control.

**Hydraulics:** The study of the mechanical properties of liquids and their application to engineering.

**Hydrocarbon:** A chemical compound based on the elements carbon and hydrogen.

Hygroscopic: Means that a material absorbs water from the air.

**IBC:** An abbreviation for internal bubble cooling.

**Impact Modifier:** An material that is added to a plastic resin to improve its impact strength. For example, MBS resins are added to PVC to produce bottles of adequate strength.

**Injection Blow Molding:** A blow molding process in which the parison, or tube, is produced by an injection molding operation.

**Interlock:** Applies to safety guarding on machinery. For example, when two machine guards are linked together in such a way that machine operation is not allowed, unless both guards are closed.

Internal Bubble Cooling: (IBC) A cooling technique used in tubular film extrusion to improve output and/or, to modify film properties by changing the air within the expanded tube or bubble. In conventional tubular (layflat) film extrusion the air, which expands the extrudate, remains captive within the bubble and reaches relatively high temperatures. This means that film cooling largely occurs from the outside. Changing the internal air improves heat transfer and therefore cooling. Cryogenic internal bubble cooling can improve the output by, approximately, a further 50%.

**Internal Cooling Mandrel:** Tube sizing system that has a cooled mandrel to size the inside diameter of the tubing.

International Standards Organization (ISO): An official body, like the ASTM, which issues test standards or procedures.

**Jig Cooling:** Cooling a product after production on or in a jig to obtain a desired shape.

**Jig:** Usually refers to a device for holding a component. For example, while it is cooling.

**JIT:** Just in time production means that components are not produced until they are required.

**K:** Refers to temperature measurement in degrees Kelvin (for example, 2050K) or alternatively to the K value of PVC which is a measure of molecular weight.

**Kelvin:** Refers to a temperature measurement in degrees Kelvin (The Kelvin temperature scale) which is obtained by adding 273 to the temperature in °C. Also referred to as degrees absolute.

**Key:** That part of the screw that permits the turning motion of the drive to be transmitted to the screw.

**Keying:** The modification of a surface to make it more receptive to inks. Techniques include corona discharge or flaming.

L/D Ratio: Length to diameter ratio.

Lace Cut: Refers to granules that have been produced by chopping strands of circular cross-section.

Laddering: Defective surface finish caused by melt fracture at the die.

Laminates: Layers of different film, or sheet, plied together to form one structure.

**Land Length:** The ratio between the length of the opening in the flow direction and the die opening, for example 10:1.

Land: See Die Land.

**Lay:** A lay is the length of twist produced by stranding filaments, such as wire, fibers, or roving.

Lay Flat or Lay-Flat: Another term used to describe the tubular, or bubble, process used to produce film.

Lay-Flat Width: A measure of the size of tubular film and is the width of a roll

of double thickness film. A tube with diameter D gives a lay-flat width of D/2 or 1.57 D.

**Lead:** The horizontal distance travelled by a material in one revolution of the screw.

**Length to Diameter Ratio:** Commonly called L/D and given as two numbers, for example, 10/1. This means that the length is ten times greater than the diameter. The minimum L/D ratio for a screw for a thermoplastic material is approximately 15:1, while 20:1 is more usual and 24:1 preferred. The higher the ratio the more stable the output and the more uniform the melt temperature. However, a longer screw absorbs more energy and a limit is set by the torsional strength at the feed end of the screw.

**Limit Switch:** A switch set at the limit of movement of any part of a machine. For example, it may initiate the cutting of the extrudate.

**Liquid Colorant:** A concentrated liquid coloring system or masterbatch. Longs: Long strands of material produced during the compounding stage when the cutter, or chopper, does not cut small enough.

**Lubricant:** A material added to ease flow (internal lubricant) or to prevent resin adhesion to the extrusion machinery (external lubricant).

**Lumpiness:** A roughness in the bore, or inside diameter, of a pipe or parison. Frequently associated with poor mixing of material along the screw, for example, in UPVC extrusion.

Machine Direction: The length-wise or longitudinal direction of an extrudate. Mandrel: A term used to describe a sizing element either, in a die or, in a sizer that controls one dimension of an extrudate. This dimension is usually the inside diameter.

**Manifold:** A distribution system within a die whose function is even out the flow of material across the width of a die.

**Masterbatch:** A concentrated mixture of an additive or additives in a carrier system. The carrier system may be a plastic material or a universal carrier system such as a wax-type material.

MD: Machine direction.

**Melt Application Compression:** (Abbreviation: MAC.) Also known as the melt strand process. In this molding process, a decorative material is first placed in a mold. Then a melt strand is formed over the opened mold cavity by an extruder fitted with a sheet die. The mold is closed to shape the part. After cooling the decorated component is removed. In this process, as melt temperatures are low, the decorative material is not subjected to high thermal stresses.

**Melt Coating:** A plastic film is extruded onto a pre-formed film. This is necessary when the material processing conditions are too dissimilar for coextrusion.

**Melt Flow Rate:** The flow rate obtained from extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of time temperature and load. The conditions are specified in ASTM D1238 or ISO 1133. The larger the number the easier the material flows.

**Melt Fracture:** Instability in the melt flow through a die starting at the entry to the die. It leads to surface irregularities on a finished article such as a regular helix or irregularly spaced ripples.

**Melt:** Polymer, or resin, at its processing temperature is commonly called melt.

**Melt Pressure:** The pressure on the melted plastic material at any point in a process.

**Melt Strength:** The strength of a plastic while in the molten state. Reflected in, for example, the resin melt 's ability to hold shape and not sag after it leaves the die.

**Melt Temperature:** The temperature of the molten plastic material as it is within or as it leaves the extruder die.

**Melt-Strand:** A wide strip, or sheet, of melt produced by an extruder and used as a feedstock for the melt-strand process. See melt application compression. **Metallizing:** The technique used to build up a layer of metal (usually aluminum) onto a plastic surface.

**Metering Zone:** The section of an extruder screw that has a uniform flight depth that conveys the melt and controls the rate of flow through the extruder into the die.

MFI: Melt flow index. Now called melt flow rate or MFR.

MFR: Melt flow rate.

**Micron:** A millionth of a meter, that is,  $10^{-5}$ : there are 25 microns (0.025 mm) in 0.001 in.

**Microprocessor:** A component of a computer. It is the data processing part of the central processing unit (CPU).

**Microprocessor Control:** A type of control system for a machine which is based on microprocessor(s). Such machines usually have a visual display unit (VDU) and keypad entry.

Mil: One thousandth of an inch. (0.001" or, 0.001 in.)

Millimeter: One thousandth of a meter, or 10<sup>3</sup> m. One millimeter (mm) equals 0.039 in.

**Mirror Image Extruder:** A type of extruder system. Such extruders are installed in pairs, facing each other to save floor space. They are often used with crosshead dies for processes such as wire covering.

**Mirror Image Profile Die:** A type of extrusion die used with a dual strand extruder. Two such dies are used with one being the mirror image of the other. The melt may be fed through a homogenization device and then divided into two streams before being fed into the two dies (Each producing, for example, window profiles).

**Mixing Section:** A section of a screw that is designed to improve melt mixing. Ideally, such a mixing section should first produce dispersive mixing and then distributive mixing. These two mixing processes may be repeated more than once.

**Modular Compounding Machine:** A compounding machine which is built on a modular principle, so that it may be configured to suit a particular material or compounding operation. Such machines may be based on a single shaft system or on a double shaft system. A twin screw extruder may also be considered as a modular compounding machine in some cases.

**Monoaxial:** In films, or tapes, it refers to single direction orientation produced by drawing in the machine direction.

**Monofilament:** Single strands of plastic used as brush bristles, fishing line or as rope (when twisted together). Diameter range 0.005 to 0.06".

**Monomer:** The starting material, molecule, or unit from which a plastic is produced (or polymerized).

NC: Numerical control.

Neck In: See 'neck-down'.

**Neck-Down:** Reduction of extrudate width caused by drawing down of sheet during extrusion. It is measured as the difference between the die width and the width of the extrudate.

**Necking:** A localized reduction in cross-section that may result from the application of a tensile stress.

Nip: A nip, or pinch, is formed between two rolls.

Nip In: See 'edge tear'.

**Non-Newtonian Flow:** The flow of a polymeric material characterized by a non-linear relationship, or non-proportionality, between shear rate and shear stress.

## 276 Nylon Plastic: Another name for polyamide plastics.

Nylon Screw: See Double Parallel Screw.

**Offset:** Means that a setting is consistently different from that which is set. In temperature control this is minimized by using an integral (I) term.

**Olefin Plastics:** Plastics based on the polymerization of olefin monomers (eg ethylene, propylene). At least 50% of the resin must be olefin.

**Open Loop Control:** A type of machine control whereby a parameter is set and is not adjusted by feedback information.

Orange Peel: An extrusion defect - See "Applesauce".

**Orientation:** An extrusion process that increases the strength and stiffness of a plastic material by stretching or rolling.

**Overcoating:** Used in extrusion coating or laminating. It means that the plastic coating is wider than the substrate. The excess plastic is trimmed and reused.

**Overshoot:** Applied to temperature control. Means that the temperature 'shoots' past the set point. This often happens during warm-up unless the controller is fitted with derivative (D) control action.

**Oxidation:** A common form of degradation for plastics that occurs when a resin is heated in the presence of, and reacts with, air or oxygen. When this occurs, the resin is said to be oxidized.

**Parallel Laminate:** A laminate in which all the layers of material are plied, or oriented, in the same direction.

**Parison:** A softened tube of polymer, produced by extrusion or injection molding, used to make a blow molded product.

**Pellets:** A plastic feed form supplied as cylinders (approximately 0. 118 in. or, 3 mm, in length and breadth), spheres, or lace cut granules.

**Permeability:** Refers to the ability of a plastic to allow diffusion through itself. Many plastic bottles will lose gas, from carbonated drinks because they are permeable to carbon dioxide.

**Permeability Resistance:** The resistance of a film to diffusion of gases or vapors: the reciprocal of permeability.

**Pigment:** An insoluble coloring system that gives opaque colors in a resin. Pinch Draw Rollers: A set of rollers used as pullers in the extrusion process: one form of puller unit.

Pitch: The horizontal distance between corresponding points of two successive lands on a screw.

Plain Extruder: A smooth bore extruder.

**Plain Screw:** A conventional three-zone screw as used in a smooth bore extruder. A screw that does not incorporate mixing sections or mixing pins. **Plastic:** A material that contains as an essential ingredient one or more high molecular weight polymers. It is generally solid at room temperature and is capable of flowing, under heat and pressure, to form a final shape.

**Plastication or Plastification:** The process of turning a plastic material into a melt.

**Plasticize:** To soften a material and make it plastic or formable, either by means of a plasticizer or the application of heat.

**Plasticizer:** A chemical, usually a liquid, which is added to a plastic or resin, in order to lower its glass transition temperature, which makes it softer and more flexible.

**Plastisol:** A suspension of PVC in a plasticizer, which is liquid at room temperature, and will gel or fuse into a solid at higher temperatures.

**Plate Die:** A simple die made by machining a hole in a metal piece and bolting it onto the die holder.

**Plate Out:** When an additive migrates through the plastic during processing, and builds up on the die.

**Plucking:** An extrusion defect that appears at intervals along an extrudate that looks like a long needle has been stitched along it.

**Plug Flow:** A flow condition that means that the velocity across the melt stream is virtually constant. It applied to the flow of most polymers, which are non-Newtonian fluids.

**Polymer:** A chemical compound made up of a large number of repeating molecular units.

**Polymerization:** The chemical process whereby a monomer is turned into a polymer or resin.

**Polyolefin Plastics:** (Also known as olefin plastics.) Plastics based on olefin monomers or repeat units (e.g. ethylene). At least 50% of the resin must be olefin.

**Poor Surface Finish:** Undesirable dullness on the surface of a product. **Porosity:** An extrusion defect that appears as voids.

**Preheater:** Device used to preheat resin before it is fed to the extruder. Also a device used to preheat wire before it is fed to the extruder.

**Preheating:** The process of heating a resin or wire before it is fed into a production machine. It is used to increase output, quality and/or dry the material.

**Pressure Die:** A wire coating die in which the guide tip stops short inside the die to allow the melt to make contact with the wire inside the die. Pressures of 5,000 psi (345 bar) are common.

**Process Capability:** A measure of variation within a process relative to a set specification range ( $C_{P}$ = Specification Range/Process Variation).

**Process Timer:** A timing device used to switch on an electrical circuit at a preselected time.

**Processability:** The ease with which a plastic material can be extruded. For example, at a given screw speed, reduced amps and pressure characterize improved processability.

**Profile Dies:** Dies that produce parts with complex cross sections by extrusion. For example, a window frame section.

**Profile:** The structure that is seen when an extrudate is cut at right angles to the direction of extrusion. This usually refers to a complex cross section.

**Proportional Band:** ('PB') A temperature band or range (for example, 10% of set point) over which power is proportioned, or reduced, as the set point is approached.

**Proportional Control:** A control action in which the power supply is progressively reduced within the proportional band as the set point is approached. **Proportioning:** Often used interchangeably with Proportional.

**Pseudoplastic:** A material is said to be pseudoplastic if its viscosity decreases when it is sheared at a higher rate. That is, it exhibits pseudoplasticity or shear thinning.

**Pseudoplasticity:** This means that the viscosity of a material (its resistance to flow) decreases the faster it is sheared (It demonstrates "shear thinning"). **Puller:** Device used to remove the extrudate from the die region at a controlled rate. It is usually either a pair of rollers or two belts (caterpillar) which trap the extrudate between them.

**Purge Compound:** A compound specifically designed, or used, to assist purging or, machine cleaning. Also known as a purge, a flushing, or cleaning compound. Before such purge compounds are used it is advisable to remove the die and head assembly as many purge compounds do not melt, or flow, like ordinary resins.

**Purging:** The action of cleaning an extrusion cylinder or barrel by running material through the barrel.

**Pushing Flight:** That face, or edge, of the screw flight that drives the plastic towards the die. The face nearest the die.

278 Pyrometer: An instrument used to measure temperature. QC: Quality control.

**Quality Control:** Techniques or schemes that seek to maintain a consistent quality of production.

Ram: Another word for plunger or piston.

**Ram Extruder:** An extrusion machine that uses direct piston pressure, rather than a screw, to drive extrusion.

**Reactive Processing (Reactive Extrusion):** Term used to describe a process, usually extrusion compounding, where the plastic is chemically reacted, or modified, as well as being shaped.

**Reclaim:** A reclaimed plastic material. Usually a reclaimed thermoplastic material, which is commonly known as, regrind.

**Reclaimed Thermoplastic Material:** Also known as reclaim. A material that has been recovered from scrapped, or unwanted, output. The re-use, by melt processing, of a molded component is only possible with thermoplastic materials or thermoplastic elastomers (TPE). One of the most common additives used with thermoplastic materials is reclaimed material. The feed is usually a mixture of virgin (new) material and reclaimed material. However, many machines run on 100% reclaimed material.

Reclamation: The process of reclaiming plastic material from unwanted product.

**Recycled Plastics Material:** A thermoplastic material, prepared from discarded articles that have been separated by type, cleaned, and ground.

**Regranulation:** The process of regrinding scrapped output.

**Regrind:** The term used for a material that has been reclaimed by grinding. **Reinforcing Filler:** A filler that strengthens, or reinforces, a polymer. One of the best known examples is glass fiber that is widely used to make rigid plastics.

**Relay:** A mechanical/electrical device in which a small electrical signal is used to initiate the switching of a much larger electrical supply.

**Release Agent:** A material painted or sprayed onto a substrate in order to prevent adhesion.

**Reprocessed Plastic Material:** A thermoplastic material prepared from industrial scrap produced by other than the original processor.

**Restrictor Bar:** A device used to even out flow along a sheet die. It is used to promote the production of sheet of uniform thickness.

**Retractable Melt Thermocouple:** A type of thermocouple whose depth of immersion can be varied during operation. It is used to study the distribution of melt temperatures across a melt stream, for example, in extrusion.

**Reworked Plastic Material:** A thermoplastics material prepared from rejected production components that has been reprocessed in a fabricators plant after having been processed in that plant.

**RH:** Relative humidity. A measure of how much moisture is present in the atmosphere.

**Rheology:** The science of flow. The study of the deformation and flow of matter in terms of stress, strain and time.

Rheometer: A device used to measure the flow properties of materials.

**Ringing:** An extrusion defect that takes the form of concentric rings of a thicker or thinner wail section caused by screw pulsation or, by slipping in the haul off or, by the extrudate sticking in the sizing box.

**Ripple:** An extrusion fault that takes the form of a steady undulation or wavelike appearance. Sometimes found in PVC extrusions that are non-uniform in temperature.

**Rodding:** Process used to force material to flow through an extruder. A rod of plastic (the same as that being extruded) is pushed onto the screw until the feedstock is gripped and feeds by itself.

Root: The root is the central shaft of a screw.

Rotating Die: Used in lay flat film extrusion to hide thickness variations and eliminate gauge bands.

Rotometer: Instrument for monitoring and regulating water flow.

**Rupture Disc:** A rupture disc is a hollow, threaded plug which has a thin metal diaphragm welded to it that is generally located between the screw tip and the breaker plate. If the melt pressure exceeds the strength of the diaphragm, it bursts and reduces the extrusion pressure by venting the material from the machine.

Safety Gate: An access port to moving machinery that will only allow the machine to work if it is closed.

Sample: A small part or portion of a material, or product, intended to be representative of the whole.

Scorex: A term used for shear controlled orientation extrusion.

**Scotch:** A mechanical restraint used, for example, on the safety gates of some blow molding machines. When the guard is opened the scotch prevents the mold from being closed.

**SCR Drive:** A motor drive system that controls the speed of a DC motor by the use of rectified pulses of power. This means that the AC supply is converted to appropriately sized DC pulses as dictated by a speed measuring device. **SCR:** Stands for silicon-controlled rectifier.

Screen: A piece of wire gauze used as a mesh or sieve; usually made into a screen pack.

**Screen Pack:** A collection, or group, of wire-mesh screens supported by the breaker plate that are used to remove contamination and/or to improve mixing by building up pressure. The coarse screen (20 or 40) is placed next to the breaker plate followed by finer screens (60, 80 or finer).

**Screw Channel:** The open section between the flights of the screw. **Screw:** The screw consists of a round bar of steel, into which is cut a helical channel (leaving the screw thread). It rotates inside the extrusion barrel and melts and conveys a plastic material from the hopper to the die.

Segmented Extruder: An extruder in which the screw and barrel are built up using segments or sections. Such a construction is used in extrusion experiments to study, for example, the effect of changes in screw design on melt mixing. Both segmented single screw extruders and segmented twin screw extruders are commercially available.

**Self-Tuning:** Some instruments are self tuning to avoid over-response or overreaction to a change.

Semi-Automatic: This means that a machine, for example a blow molding machine, will do one cycle of operation and then stop until the operator starts it again.

**Semi-Crystalline:** Usually applies to a semi-crystalline, thermoplastic material. The material is not 100% crystalline. - See crystalline.

**Semiconductor:** A material whose electrical resistance is midway between that of an insulator and that of a conductor.

Set Point: A setting on an instrument. If a temperature controller is set at 200°F or 93°C then the set point is 200°F or 93°C.

Setting: The preparation of a machine for production.

**Shank:** The shank of a screw is the rearward end. It is the part that fits into the drive and which contains the drive key.

Shark Skin: A defect caused by melt fracture. See 'Applesauce'.

**Shear Heat:** Heat generated as a result of shearing or deformation of the polymer: for example, by the screw within the extrusion barrel.

**Shear Rate:** The rate at which a material is undergoing deformation, or movement, in response to a shear stress; usually expressed in reciprocal seconds, that is, in sec<sup>-1</sup>. 280 Shear Stress: The force applied to a material to cause flow. It has the dimensions of force per unit area.

**Sheet:** An individual piece of sheeting. Extruded sheet usually has a thickness greater than 0.01 in. (0.25 mm).

Sheet Extrusion Die: Die used to make plastic sheeting.

**Shrinkage:** The reduction in dimensions that a component suffers after production.

**Shrink-Wrap:** A packaging technique that utilizes stretch orientation. Components are placed inside a bag that is heated cause shrinkage (tightening) of the plastic film.

Silage Bale-Wrap Film: A type of extruded polyethylene film used for the wrapping of silage. Such film is often coextruded, with a central layer of linear low-density polyethylene sandwiched between a UV-resistant black layer and a self-adhesive inner layer. The self-adhesive inner layer is a tacky polyethylene film.

**Single-Screw Extruder:** An extruder that uses a single conveying screw to melt and pump the plastic material.

**Single-Shaft Kneading Extruder:** A modular compounding machine. Such a machine may be based on a central shaft onto which are fitted rotors (that interact with barrel mounted stators) and threaded sections (which convey material between the rotor/stator pairs). The material is heat-softened, in an extrusion section, passed into the mixing section and then into another screw section. This process may be repeated several times to obtain mixing and devolatilization.

Sink Mark: An unwanted depression on the surface of a component.

**Size (of an Extruder):** The size of an extruder is expressed in terms of the external diameter of its screw: for example, 2 inch or 50 mm.

Sizing Plates: Used in tube or pipe production to control size or external diameter. They are metal plates with machined holes of the required size. Sizing Rings: Used in tube or pipe production to control size or, external diameter.

**Skiving:** A process for the production of film or sheet by cutting from a block or log of material. It is used, for example, to make PTFE tape.

**Slip Agent:** A substance added to promote sliding action. For example, a fatty acid amide, erucamide, is used widely in film extrusion.

Slip: The ability of materials to slide over each other.

Smooth Bore Extruder: A smooth barrel extruder or a plain extruder. A conventional single screw extruder that does not contain an axial grooved bush. See Grooved Bush Extruder.

**Softener:** An additive, usually a liquid, which is added to make a polymer softer. Oils are used in elastomers/rubbers for this purpose.

**Solenoid:** An electromechanical device in which the flow of an electrical current through a coil causes an iron core to move. It may be used to open or close valves.

**Solution Casting:** Similar to dispersion coating except that the polymer is soluble in a suitable liquid. See Solvent Casting.

**Solvent Casting:** A solution of polymer is cast onto a metal plate or rolls and the solvent is evaporated, leaving a film.

SPC: Statistical Process Control.

**SPE:** The Society of Plastics Engineers.

Specimen: A piece or portion of a sample used to make a test.

**Spider Lines:** An extrusion defect that is caused by the plastic melt being divided by the legs of the spider.

Spider: That part of a pipe or wire coating die assembly that supports the torpedo. Spreader: Another word for torpedo.

**Sputtering:** An electrical process for depositing a metal coating onto a surface.

**SQC:** Statistical Quality Control.

Square Pitch: When the pitch of a screw is equal to its diameter it is called a square pitched screw. It has a helix angle of 17.70 and the number of turns is equal to the L/ D ratio.

**Standard Deviation:** A measure of spread or dispersion. It is defined as the square root, of the mean of the squares of the individual deviations from the average.

Start: The number of separate threads that can be traced along the screw. This number is usually one; so most screws are single start screws.

Static Mixer Blender: An in-line unit that mixes the plastics melt stream to make it uniform in temperature and composition. It does this by cutting and recombining the melt stream by making it flow over many blade-type obstructions.

**Static:** The electrical charge generated, and retained, on insulated surfaces: generated on film surfaces when they are separated.

Statistical Process Control: The application of statistical techniques to production processes to keep them under control and thus improve productivity and uniformity.

Statistical Quality Control: The application of statistical techniques to the production process to improve quality.

**Stellite:** A hard and non-corroding alloy made from cobalt (35 to 80%), chromium (15 to 40%), tungsten (10 to 25%), molybdenum (0 to 40%) and iron (0 to 5%).

Stenter Frame (also called Tenter Frame): A device for imparting biaxial orientation to extruded film. The film is gripped by the edges and drawn sideways at the same time as it is pulled in the machine direction.

**Step:** A rapid decrease in channel depth (over one turn or less) which acts as a barrier to forward flow of un-melted granules. It, therefore, fixes the gel point at the step. See Double Parallel Screw.

Stepped Screw: See Double Parallel Screw.

Surging: The unsteady flow of a plastic melt through the extruder.

**Tapered Extruder:** An extruder that contains a tapered screw or screws. See Tapered Twin-Screw Extruder.

**Tapered Parallel Screw:** An extrusion screw which has a gradually tapering root (the channel depth gradually changes) in the compression zone, while the feed zone and the metering zone are parallel and of constant depth. Probably the most common screw type. Unlike the double parallel screw, the velocity of the un-melted portion of the melting material bed is maintained and there is less risk of polymer stagnation (because of the absence of a step).

**Tapered Screw:** A screw that has a gradually tapering root: i.e., the channel depth gradually changes. Usually the diameter of the screw root increases steadily from the hopper end to the die end of the extruder.

**Tapered Twin Screw Extruder:** A type of twin-screw extruder that contains two screws the outside diameters of which gradually reduce from the feed zone to the discharge end. By tapering the screws, large thrust bearings can be incorporated which permits high outputs. Shear at the screw tips is also reduced because of the reduced surface speed of the screw at that point.

**Temperature Profile:** The temperatures set on the zones of a machine. During melt processing it is common to have a lower temperature at the hopper-end of a machine and a higher temperature at the die-end. The temperatures of the zones in between are increased fairly uniformly. This is a normal or "uphill" temperature profile. However, one can also have a reverse temperature profile.

282 With some materials, for example high melting point nylon, the extruder may be started with an uphill profile and then be switched to a downhill profile once extrusion is safely underway.

Tenter Frame: See Stenter Frame.

Terpolymer: A polymer made from three monomers.

**Thermocouple:** A heat sensor that consists of two wires of different metals joined at each end. It has two junctions, one hot and one cold. Because of this temperature difference a small electric current flows and its value is proportional to that difference. Also called the Seebeck effect or, the thermoelectric effect.

**Thermoplastic:** A plastic material that can be repeatably melted and solidified by being cooled. This makes thermoplastics recyclable.

Thermoset: The short form for thermosetting plastic.

**Thermosetting Plastic:** A plastic material in which the initial monomers react to form a three-dimensional cross-linked network. It is essentially one large molecule. Therefore, it cannot be re-melted or reprocessed.

**Thixotropy:** Means that the viscosity of a material increases drastically when the shearing force inducing the flow is removed.

**Thread Up:** The procedure of starting an extrusion line by leading the extrudate through the downstream equipment.

**Three Term Control:** Associated with temperature controllers, it means that the instrument has circuits, or terms, for proportional, integral, and derivative control. It is a PID controller.

**Three-Roll Stack:** Set of three metal cooling rollers used to cool and size plastics sheet in a sheet extrusion line.

**Thrust Bearing:** The main bearing that takes the load (or thrust) generated by the screw in an extruder. It is designed to last at least 100,000 hours at the extreme thrust load of the screw.

**Thrust Load:** The load generated by the turning action of the screw that is assumed to be twice the head pressure value. For example, if a 3.500 in. (88.9 mm) machine is operating against a maximum head pressure of 5,000 psi (350 bar) the direct back force is 50,000 lbs. (223 kN). The extreme thrust load is twice this or, 100,000 (446 kN).

**Timed Acceleration:** Limits the rate at which an electric motor comes up to speed to prevent excessive loads that may damage the screw or the drive. **Total Quality Control (TQC):** Means that everyone in the factory is committed to quality production.

TQ: Tubular quench.

Trailing Flight: The rear part of the screw flight.

**Transducer:** A device that changes energy from one form to another. For example, a force may be changed to an electrical output as in a pressure transducer.

**Transition, First Order:** A change of state associated with a discontinuity in properties such as the crystal to liquid change that occurs in the melting of polymer crystals. The melting point of a semi-crystalline, thermoplastics material is an example of a first order transition.

**Transition, Second Order:** A transition characterized by a shift in the rate of change of a property, such as heat capacity verses temperature. An example is the glass transition temperature  $(T_o)$ .

**Transverse Direction:** The direction across the extruder (i.e. at right angles) to the machine direction.

**Tubing Die:** A die used to produce tubing. This die has an outer bushing and an inner pin. The plastic melt flows in between the two to form the product. **Tubing:** Small bore pipe with an internal diameter of 0.5-in. (12.7 mm) or

less. The term may also be applied to larger bore pipe if such pipe is flexible and has thin walls.
**Tubular Film:** Film produced by extrusion. A tube of softened plastic is inflated to the required diameter, cooled, and collected on a reel.

**Tubular Quench:** A tubular film process in which the bubble is quenched with water to improve the clarity of films made from semi-crystalline thermoplastic materials such as polypropylene.

**Twin-Screw Extruder:** An extruder that uses two screws to convey a plastic material. The two screws are parallel and may be intermeshing or non-intermeshing and co-rotating or counter-rotating.

**Two Term Control:** Associated with temperature controllers. Refers to the fact that the instrument has circuits, or terms, for proportional and integral or proportional and derivative control. That is, it is either a PI controller or a PD controller.

**Ultrasonic Welding:** A joining process for plastics that uses the friction induced in components by ultra sound to melt the material at the interface of the parts.

**Underwater Pelletizing:** Another name for the "die face cutting" process used to produce pellets from molten strands by cutting them off, underwater, at the die face,

**UV Stabilizers:** Chemicals added to plastics to hinder decomposition caused by UV light.

UV: Ultra violet.

**Vacuum Calibrator:** Equipment used to set the dimensions of an extrudate (e.g., the external diameter of pipe or tubing) by expanding the material against sizing rings by application of a vacuum.

**Vacuum Sizing:** Technique used to set the dimensions of an extrudate by expanding the material against a sizing sleeve, or against sizing rings, by means of an applied vacuum.

Variance: A statistical term obtained by squaring the standard deviation. VDU: Stands for visual display unit.

**Vent:** A hole, or port, in the barrel though which material can be withdrawn from or introduced into the molten plastic material.

**Vented Extruder:** An extruder which contains a port, or vent, in the barrel through which material can be withdrawn, or introduced, into the molten plastic material. It is often used to extract volatiles (devolitize) from a plastic material.

Venting: The process whereby volatiles escape, or are removed from, a molten plastic material.

**Venturi Cooling Ring:** A unit used to cool film, by using a primary air stream to draw in additional air by the venturi effect. It is also used to stabilize, or size the film while it is cooling.

**Vertical Extrusion:** A process in which the barrel of an extrusion machine is mounted vertically. (I.e., at right angles to the ground.)

Virgin: In extrusion refers to material that is new or unused.

**Viscosity Coefficient:** The shearing stress necessary to induce a unit velocity flow gradient in a material. Generally expressed in Pascal-seconds (Pa-sec). **Viscosity:** The resistance to flow of a material. The ratio of the shear stress and shear rate. Viscosity is constant for a Newtonian fluid, but variable for polymers. It is expressed in Nsm<sup>2</sup>, Pa-sec, or lbs. sec. in<sup>2</sup> and given the Greek letter  $\eta$  (Apparent viscosity is the value obtained for non-Newtonian fluids, e.g. polymers, assuming that they behave as Newtonian materials).

**Voids:** A defect found in thick sections that develop during cooling. They are unwanted cavities.

**Volatiles:** A general term for a substance that is a gas at processing temperatures.

**Warping:** Distortion of a product that generally occurs upon cooling. The dimensions of an extrudate may distort as a result of uneven cooling or

284 because it was hauled off unevenly. May result from orientation in filled (e.g., glass) extrudates.

Water Treeing: The breakdown of cable insulation caused by moisture in the presence of an electric field.

Web: A name given to film under tension during production.

Weir: An aperture on the end of a water tank that is used to control the water overflow into the drain.

Wettability: The relative tendency of a fluid (for example a plastics melt), to adhere to and spread on a substrate. The wettability of a plastic coating is typically dependent on surface tension, viscosity and chemical compatibility.

Wheatstone Bridge: A divided electrical circuit used for the measurement of resistance.

Wind-Up: Roller system used for collecting film or wire.

Winders: Units used to collect extrudates on spools or reels.

**Window:** An area of transparent material in an otherwise translucent, or opaque, part. This defect could be due to uneven cooling or, to un-melted granules that have passed through the machine.

Wire Covering or Wire Coating: An extrusion operation for the application of a plastic coating onto a wire. It is usually done by crosshead extrusion. Yardage: The length of product on a reel.

Yield: The area of film at a given thickness produced from a given weight of resin.

**ZD:** Zero defects. Refers to the goal of producing perfect product all the time. **Zero Start:** The speed control must be turned to zero before the drive can be started.

**ZI:** Zero inventory manufacturing - see JIT.

**Zone:** A section of an extruder barrel. For ease of control, the barrel of an extruder is divided into zones or regions. Each zone has its own heating elements and controls. That is called zoned heating.

**Zoned Heating:** The separate heating, and temperature control, of each zone of an extruder. The barrel of a small extruder will usually have three zones, while larger machines may have, for example, twelve. A temperature sensor and associated electronics (for example, a microprocessor-based, three term (PID) controller) control each of these zones.

## ► BOOK LIST

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