

Qenos

06

ROTATIONAL MOULDING

—
TECHNICAL GUIDE



Alkatuff®



Front Cover

The standard for UV performance for PE Water Tanks specified in AS/NZS 4766 PE Tanks for the Storage of Chemicals and Water is 8,000 hours of uninterrupted exposure to an intense and specifically developed UV light source. Qenos exhaustively tests the long term UV performance of its Rotational Moulding Resins under conditions of controlled irradiance, chamber temperature and humidity and repeated rain cycles. *Alkatuff 711UV* resin achieves a class leading UV performance exceeding 20,000 hours against the required standards, ensuring that tanks made from *Alkatuff 711UV* are *Tough in the Sun*.

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ROTATIONAL MOULDING

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INTRODUCTION

Rotational moulding provides a means of producing polyethylene articles of a size and shape that cannot be manufactured economically in any other way, and is particularly suited for relatively large, hollow, seamless parts. Such articles include large industrial chemical or agricultural tanks, shipping containers, marine floats, traffic bollards, canoes and small boats, rocking horses, pedal cars – indeed almost any hollow, enclosed or open-ended shape.

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INTRODUCTION

For many years, low density polyethylene (LDPE) powders were used for the rotational moulding process. However, since the early 1990's, linear low density polyethylenes (LLDPEs) have come to the fore in rotational moulding applications, because of their outstanding physical properties (excellent environmental stress crack resistance and impact performance) and ease of processing. *Alkatuff* LLDPE powders are eminently suitable for rotational moulding for these reasons.

GRADE SELECTION FOR ROTATIONAL MOULDING

The *Alkatuff* grade range is designed to provide optimum performance in moulding and durability. The grades provide a range in flow, balanced with excellent crack resistance.

Alkatuff LL711UV is a premium tank grade providing high rigidity along with excellent processability. This material is known for its proven performance and broad processing window.

For smaller mouldings where rigidity is of less importance, the higher flow *Alkatuff* LL705UV, again with excellent crack resistance may be suitable.

Both *Alkatuff* LL711UV and LL705UV contain premium UV and antioxidant packages and meet the requirements of the tank standard AS/NZS 4766. Both are supported with technical data to enable Finite Element Analysis (FEA) of designs.

For parts requiring higher flow and sharpness of detail, but allowing greater flexibility, the higher flow grade *Alkatuff* LL710UV may be considered.

Effect of Melt Flow Index and Density

The end-use properties of the rotationally moulded product will depend on the density and Melt Flow Index (MFI) of the polyethylene used. Increasing the density leads to increased stiffness as well as a higher softening temperature, surface hardness and abrasion resistance. A higher MFI is desirable for ease of flow but low MFI leads to better toughness and environmental stress crack resistance.

Polyethylenes with an MFI between 2 and 20 g/10min can be processed by rotational moulding, but normally grades with an MFI of approximately 3 to 5 g/10min are most suitable.

The effects of changes in MFI and density on the properties of polyethylene rotational mouldings are shown in Figure 1.

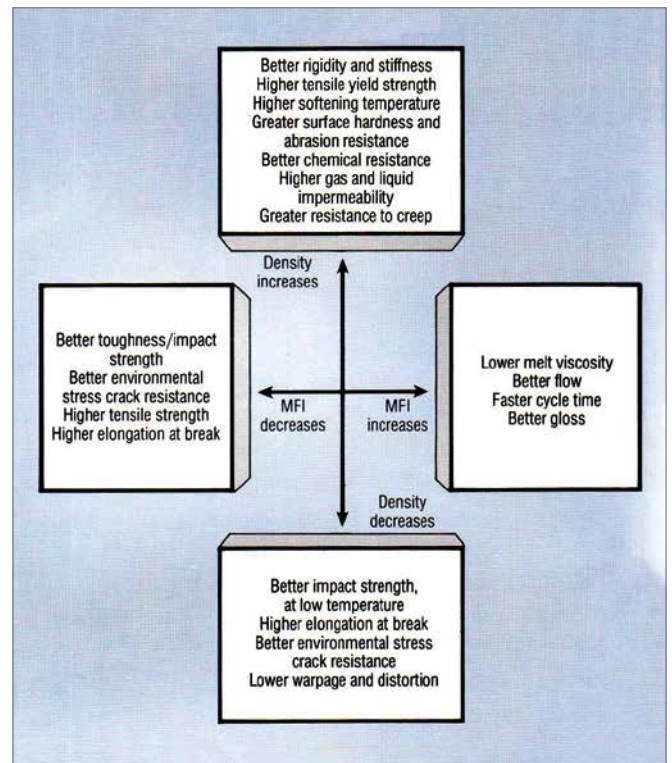


Figure 1: The Effects of Changes in MFI and Density on Properties of Polyethylene Rotational Mouldings

Effect of Comonomer on Crack Resistance

When subjected to stress over a period of time most materials commonly fail by cracking. Polyethylene may also suffer this mode of failure. It is important to understand that different grades of Polyethylene can exhibit vastly different performance with respect to crack resistance. The crack resistance should be a major consideration when selecting a grade of polyethylene for a particular application.

The crack resistance of a specific grade of Linear Low Density Polyethylene is governed largely by the size of its molecules (indicated by its Melt Index) and the type and level of comonomer employed in its manufacture. Comonomers such as butene (C4), hexene (C6) and octene (C8) are reacted into the polymer chain in order to produce small side chains which in turn create a deviation, or change of angle in the molecule chain. The effect of this change of angle is somewhat akin to the effect of a knot in a piece of wood.

The Polyethylene becomes more difficult to split. The size and quantity of comonomer molecules introduced into the polymer chain determine the degree to which the crack resistance is improved. For example, the side chain created by the incorporation of hexene is twice the size of that

created by the incorporation of butene. As a result, the chain deviation caused by hexene is significantly greater than that caused by the incorporation of butene and results in the hexene LLDPE exhibiting a crack resistance approximately 10 times better than the butene LLDPE. The incorporation of octene into the polymer chain creates only a slightly larger deviation than that created by the incorporation of hexene and the change in crack resistance is less significant.

Alkatuff LLDPE rotational moulding grades utilize hexene comonomer resulting in excellent crack resistance.

Weather Resistance

When polyethylene is exposed outdoors, the ultra-violet component of the sunlight causes photo-oxidation of the polymer, resulting in surface crazing and a marked deterioration of the mechanical properties with time. Stabilisers are incorporated into the polyethylene to provide weather resistance for outdoor applications. Qenos' UV stabilised rotational moulding grades contain an efficient additive package which enables extended outdoor life spans. Qenos exhaustively tests the long term UV performance of its Rotational Moulding Resins under conditions of controlled irradiance, chamber temperature and humidity and repeated rain cycles. *Alkatuff LL711UV* achieves a class leading UV performance exceeding 20,000 hours of uninterrupted exposure to an intense and specifically developed UV light source. See Figure 2. This far exceeds the minimum requirement of UV8.



Figure 2: UV performance testing to >20,000 hours against AS/NZS 4766

Thermal Stability

When unstabilized polyethylene is exposed to heat it will decompose or degrade quite quickly. The material will turn yellow/brown in colour and lose its strength and ductility. During the degradation process the material undergoes two reactions. One is crosslinking, where polymer chains are joined to form larger molecules and the other is chain scission, where the chains are broken into smaller fragments. Both reactions occur at the same time, crosslinking having the predominant effect initially. This may be noted as a change in the melt flow index of the material. Initially the melt flow index may decrease as crosslinking increases the viscosity of the polyethylene. As exposure to heat continues the melt flow index will increase as chain scission decreases the viscosity of the polyethylene. In an air environment oxygen plays a major role in the degradation reaction. The oxygen reacts with polyethylene breaking the chains and creating shorter molecules which contain oxygen end groups. The material is said to have been oxidized.

In order to arrest the rate of thermal decomposition, stabilizers, typically called antioxidants, are added to the polyethylene used for rotational moulding. The antioxidants are compounded into the polyethylene by first mixing them with polyethylene powder and then passing the mixture through a heated extruder and pelletiser. The extruder contains screws specifically designed to melt and homogenise the polyethylene and antioxidant mixture. The result is that the antioxidants become finely dispersed throughout the polyethylene, enabling them to provide access to potential oxidation sites.

Antioxidants are necessary to provide protection to polyethylene over a number of stages. The first is during extrusion conducted by the polyethylene manufacturer. The second, for coloured materials, is another extrusion stage where pigment is added via a colour masterbatch (concentrate) and the material is pelletised again. During both these extrusion stages the material may be exposed to temperatures of the order of 230°C. The third stage is during milling (grinding), however the temperature the material is exposed to in this process is relatively low and only for a brief period of time. The final processing stage is of course the moulding process, where the material can be exposed to high temperatures for long periods of time. After processing the mouldings may also be exposed to moderately elevated temperatures for long periods of time during service.

In order to provide adequate protection, the additive package needs to be robust and will normally include two different types of antioxidant added at generous levels.

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Colouring

Pigments play a major role in many rotationally moulded articles. Indeed there would be a much lower demand for products if they could not be provided in a multitude of colours. One of the benefits of polyethylene is that it can be readily pigmented to provide a very wide range of colours. Apart from aesthetic appeal, pigments can play a vital role by nature of their ability to absorb and reflect light and UV radiation from the sun. The ability to block light is essential for applications such as water tanks. A tank produced from unpigmented polyethylene would allow the transmission of light and provide a haven for algae to grow. Pigments added at sufficient levels can block light sufficiently to stop algae growth. As different pigments will allow different levels of light to pass through polyethylene, the quantity or loading of the pigment will need to be different to achieve the same level of blocking.

This is true also when considering the absorption of UV radiation. Most pigments used in the colouration of polyethylene for rotational moulding will assist in resistance to weathering by absorbing UV radiation. It is UV radiation that attacks polyethylene and causes it to become brittle, leading to cracks. Some pigments can have quite a significant benefit. The best example of this is carbon black, the pigment typically used to colour polyethylene black. The right type of carbon black when added at a rate of 2 – 2.5% and well dispersed can enable UV life spans in polyethylene of greater than 20 years. Other pigments in combination with UV stabilisers can provide UV life spans beyond 10 years.

For a pigment to be suitable for rotational moulding it needs to possess certain properties. It needs to be safe to use. This is particularly important when considering articles that come in contact with food or water. There is also the question of environmental effect when the article has reached the end of its useful life.

During processing the pigment needs to be thermally stable, and in the final application it needs to resist fading. They need to be “light-fast”.

Pigments may also have negative effects upon polyethylene. If they are poorly dispersed the resulting concentrated areas of pigment may cause embrittlement of the polyethylene.

Due to their nature, pigments can affect the rate at which the material heats and cools. The result is that optimum cooking and cooling times may differ from colour to colour. Different pigments may also affect shrinkage to varying degrees.

This may be an important factor when tight dimensional tolerances are required. Some organic pigments increase the crystallization rate by acting as nucleating agents. They may, as a result, contribute to warpage and brittle behaviour.

The ability of pigments to absorb radiation from the sun may have another unwanted outcome, a change in dimensions. Radiation absorbed by a pigment may be converted in to heat and raise the temperature of a moulding. This in turn leads to expansion. Such expansion may lead to increased stress in a moulding and reduce its useful life. Other unwanted outcomes may be reduced rigidity and increased creep.

The choice of pigment used for colouring polyethylene can play an important role in the performance of mouldings.

Pigments can be incorporated in to polyethylene in a number of ways. Dry blending of powder with pigments can give good results, however this practice is not recommended as the pigment may not be properly dispersed and this will reduce the impact resistance and tensile strength of the moulding. High-speed blending in, for example, a Henschel-type mixer, will give better results than simple tumble blending of the powder components.

Coloured powders obtained by grinding compounds into which the pigment has been incorporated will lead to mouldings with the best balance of physical properties. This is particularly so for mouldings which will be subject to stress during their use.

In the melt compounding process the pigment is premixed in a masterbatch form, which is then mixed with the polymer granules as it passes through an extruder which melts and mixes the two materials together to form a uniform coloured melt.

An extruder consists of a long screw which rotates inside a steel barrel. The barrel is heated by electrical heater bands which circle the barrel, and each heater is controlled separately so we can vary the amount of heat in each one. There is a hole in the top of the barrel at the back end, to which the hopper containing the material is fixed. The barrel is open at the front allowing the melted plastic to be pumped out. Attached to the front of the barrel is a die with holes in it and the plastic material passes out of these holes. Typically it is cut into pellets with an underwater chamber called a pelletiser like shown in Figure 3.

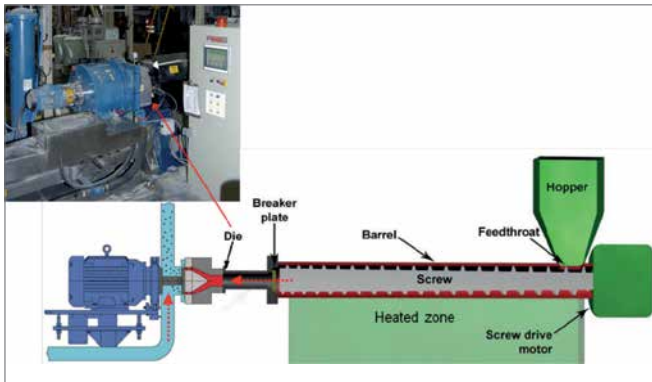


Figure 3: Extruder with Die Face Cutting

The pellets are then ground to the required particle size powder using a Grinding or Pulverising Mill (Figure 4).



Figure 4: Grinding Mill

Other specialised additives such as antistatic and fire retardant may be used. They are normally added by the supplier of the plastic powder, (not the polymer manufacturer) after discussing the requirements with the rotational moulding company concerned.

POWDER PRODUCTION AND PROPERTIES

Polyethylene is ground in to powder for rotational moulding for two major reasons. The first is to enable an even coating of material to be laid down on the internal surface of the mould. The second is to enable fast heating of the material which has an inherently low thermal conductivity.

Polyethylene powder for rotational moulding is produced by passing pellets through a mill which fragments them in to fine particles. The mill consists of two circular steel plates with multiple lines of cutting teeth which typically radiate outwards like spokes in a wheel. One of the plates is rotated at high speed and the other, stationary plate, contains a hole in the middle through which the pellets are fed. The gap between the plates is greater towards the centre and as the material is ground it makes its way to the periphery of the plates. It then exits and is conveyed to a set of vibrating screens for sorting. Particles which are too big to pass through the screens are directed back in to the mill to be ground smaller.

Powders may be classified by the mesh size used for screening. A 30 mesh screen, which has 30 holes per square inch, has hole sizes 0.6 mm square. This mesh screen is commonly used in Australia. Finer powders may be produced using 35 mesh screens which have hole sizes of 0.5 mm square. No matter the mesh size, there is always a range of particle sizes produced. The range of particle size, or particle size distribution, may be measured using a set of sieves of different mesh sizes. The sieves are set up in order of mesh size in a stack, with the coarsest mesh at the top. A sample of powder is then introduced and the sieve stack is shaken by a vibrating base. Table 1 details the sieve analysis results obtained for a commercial sample of powder in Australia.

Table 1: Sieve Analysis Results

Sieve Size (microns)	Weight (%)
600	0.3
500	8.4
425	13.7
300	24.0
212	25.9
150	13.7
Pan	14.0

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It can be noted that the sample contains 14% of “fines” which fell through to the pan. The fines play an important role as they are the first particles to melt and stick to the mould surface. It is important to have sufficient fines to coat the entire mould surface. They form the first layer to which larger particles can adhere and melt. Fines are also important for their ability to flow into mouldings which contain fine detail.

The other key point to note is that this sample of powder contains only a small percentage of particles over 500 microns (0.5 mm) in size. Larger particles take longer to heat and necessitate longer cook times. As the larger particles lay down last, the quality of the internal surface finish is largely dependent upon their size and number. Large particles may form a crazed internal surface which may compromise the impact strength of the moulding.

Particle shape and size distribution can be varied by changing conditions in the mill. Two of the key parameters are the gap between the plates and the rate of feed of the material in to the mill. Whilst a smaller gap produces a smaller average particle size it will also result in the generation of more frictional heat. This is also the result of a faster feed rate. Both these parameters need to be balanced to produce a suitable powder temperature.

The temperature of the powder in the mill plays an important role in determining the shape of the particles produced. Too low a temperature can result in particles with “tails” and too high a temperature will cause the particles to melt together. Tails are a significant issue as they can reduce the flow of the powder and in the worst case tangle to form balls.

The ease with which a powder flows can be readily measured by timing how long it takes for a set quantity to pass through a funnel. Indeed this forms the basis of the ARM International Dry Flow test. The test measures the time taken for 100 g of powder to pass through a funnel of the following dimensions (see Figure 5).

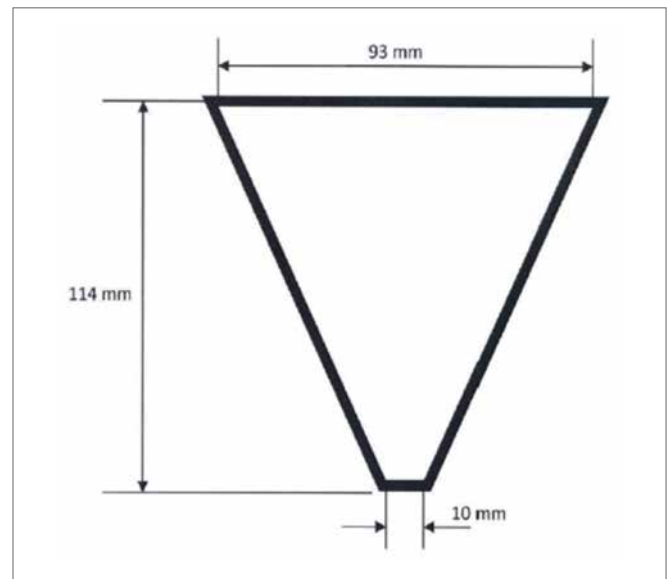


Figure 5: Dimensions of Dry Flow Funnel

Desirable dry flow results are typically between 24 and 30 seconds.

In order to further define the properties of the powder the discharge from the funnel is collected in a cup. The cup is 100 ml in volume and the excess or overflow material is wiped from the top of the cup with a straight edge. The cup is weighed prior to and after collection of the powder sample. The weight of powder is determined by the difference between the two. The weight of the powder per 100ml is a measure of its bulk density. A powder with a very low bulk density is undesirable as it is likely to trap a lot of air as it melts. Desirable bulk densities are in the order of 340 to 380 grams per litre.

ROTATIONAL MOULDING PROCESS TECHNOLOGY

In the rotational moulding process (often called rotomoulding), polyethylene powder is heated in a mould of the shape required to form a hollow moulded article. While the mould and powder are being heated, the mould is rotated simultaneously about two perpendicular axes to distribute the powder evenly over its internal surfaces and to cast a uniform coating of the molten polymer. After cooling the mould, the moulding is removed and the cycle repeated.

There are four basic stages to this process, viz. mould charging, mould heating, mould cooling, and demoulding. However, it is the heating and cooling steps which mainly affect the quality of the production (see Figure 6).

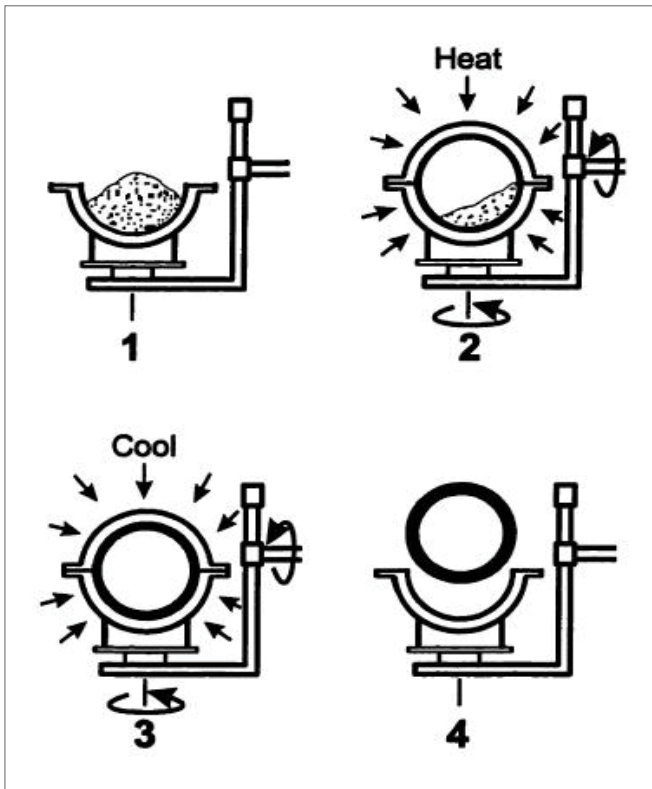


Figure 6: The Four Stages used in the Rotational Moulding Process

Stage 1 – The Mould Charging or Loading Step

In the first stage of the process, polyethylene powder is charged into the stationary bottom section of the split mould. The mould sections are then closed and firmly secured, usually by means of quick-acting clamps or bolts, and the mould assembly mounted in the rotational moulding machine ready for the moulding stage. The vent hole should be checked to ensure that air can enter or escape the mould during the moulding operation.

The quantity of powder used determines the ultimate wall thickness of the moulded part. Charging the mould with too much powder results in unnecessarily thick walls, and the cycle will have to be extended to ensure complete fusion. Too little powder leads to localised thin areas in the moulding wall, with the risk of subsequent product failure. Wall thicknesses between 1.5 and about 25 mm can be readily achieved using the rotational moulding process.

Stage 2 – Mould Heating Cycle

The loaded mould is rotated about two perpendicular axes in an oven heated usually by gas-fired hot air at a temperature between 250°C and 300°C. The axial speeds of rotation are quite slow, e.g. 5 to 20 revolutions per minute, and should be adjustable, in their ratio depending on the mould configuration (see Table 1).

The applied heat conducted through the mould walls gradually melts the tumbling powder, which then adheres to the mould and gradually fuses to form a smooth homogeneous layer of uniform thickness over the inside surface. As the air inside the mould heats up, it expands and the excess escapes through the vent-hole.

After a predetermined heating time, which can typically vary between about 5 and 40 minutes, the rotating mould is transferred from the oven to a cooling station. The heating cycle should be kept as short as possible in order to minimise polymer degradation, but should be sufficient for optimum fusion, i.e. to eliminate porosity in the moulding, to maximise toughness properties and to give a smooth inner surface (see pg. 11).

Various methods are employed to heat the mould in the oven. The most common is recirculating hot air, which is heated by gas burners and distributed evenly throughout the oven using blowers. High air velocity is required for efficient heat transfer. Other methods include the use of direct flame heating and electrical or gas-fired infra-red radiant heating. The temperature of the air in the oven is generally used to control the process.

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Stage 3 – Mould Cooling Cycle

When the rotating mould is transferred to the cooling station at the end of the heating cycle, the rotation is maintained so that the molten polymer retains an even thickness inside the mould and does not flow or sag due to gravity before solidification occurs.

The mould is cooled either, by a forced air blast or by a combination of air and cold water sprayed over the mould, to solidify the molten polymer. When forced air is used, the rate of cooling will generally be much slower, and this may not always be desirable for optimum toughness.

As the air inside the mould cools, it contracts and extra air is drawn in through the vent-hole to compensate for any vacuum. Often cooling water is also drawn in but this rarely constitutes a problem although the resulting water-tracking may be undesirable in some mouldings.

Stage 4 – Demoulding or Extracting the Moulding

The cooled mould is moved to the unloading station where rotation is stopped. The mould is opened and the finished moulding removed only after it has been cooled sufficiently to retain its shape. Removal is normally easy as the moulding shrinks away from the mould wall during cooling. It is advisable to include a taper of a few degrees (e.g. 3 degrees) in deep-draw mouldings to aid demoulding. Release agents can also be used to facilitate removal of the moulding (see pg. 11).

ROTATIONAL MOULDING EQUIPMENT

For a given part size, rotational moulding equipment is simpler and relatively low in cost compared with equipment for other moulding processes such as injection or blow moulding. Several different types of machines are used for rotational moulding.

Types of Equipment

The most common type of rotational moulding machine is the so-called Carousel-type three-arm machine which is shown in Figure 4. This consists of a central hub or capstan, from which three horizontal arms or spindles protrude at 120 degrees to each other. The moulds are mounted on the ends of the arms. The capstan is indexed through 120 degrees at a time, so that any one arm with its attached mould will see in turn the heating oven, the cooling station and the unload/load station.

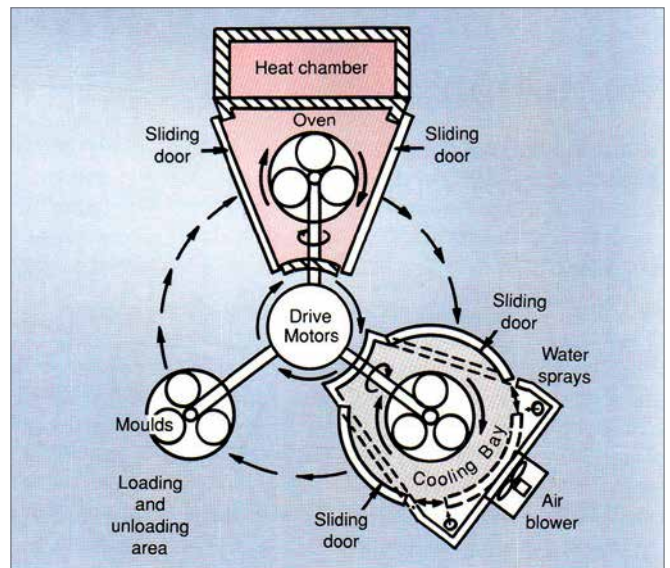


Figure 7: Three Station Rotational Moulding Machine Plan View

Other types include:

- Batch-type machines;
- Straight-line shuttle-type machines where the mould is mounted on a carriage which is moved between the three stations via a track;
- Multi-arm machines similar to the carousel type where production rates can be increased by having a two- or three- stage heating cycle and two-stage cooling; and
- Open-flame “rock and roll” machines which use a combination of uniaxial rotation in one direction and a rocking motion in the other with heat applied directly from a gas-fired burner (see Figure 8).
- Rock and roll oven machines, in which the mould is uniaxially rotated inside a rocking oven
- Uniaxial machines with direct mould heating and cooling



Figure 8a: Multi-Arm Rotational Moulding Machine



Figure 8b: Open Flame “Rock and Roll” Rotational Moulding Machine

Rotation Speeds

Typical rotation speeds for both the major and minor axes are kept low, between 5 and 20 rpm, to prevent the development of strong centrifugal forces which would cause large variations in wall thickness.

However the ratio of the speed between the major and minor axes must be chosen to obtain a uniform distribution of the polyethylene powder on the mould surface, and hence a uniform wall thickness. The ratio of rotation speeds depends on the shape and mounting position of the mould, and also on the relative size of the powder charge to mould volume. It has been found that a ratio of 4:1 for the major-to-minor-axis speeds is satisfactory for many regularly-shaped mouldings and where the longest dimension of the moulding is mounted parallel to the major axis. A ratio of 1:1 will tend to generate areas starved of powder. The optimum ratio is generally determined by practical tests combined with previous experience. Some guidelines developed by machine manufacturers are summarized in Table 2.

Table 2: Suggested Speed Ratios

Speed Ratio Major: Minor Axes	Shapes
8:1	Horizontally mounted oblongs
4:1	Cubes, balls, boxes, etc.
2:1	Toroids, round flat shapes
1:2	Parts which show thin sides at 2:1
1:5	Vertically mounted cylinders

Mould Construction

The moulds used for rotational moulding are generally of simple construction and hence are relatively inexpensive compared with those for injection or blow moulding. This is because the pressures involved are negligible, no water cooling channels are required, and the plastic melts are free-flowing.

The most common materials used in the manufacture of moulds are cast aluminium, sheet metal or, less commonly, electro-formed copper or nickel. Aluminium moulds are usually about 6 mm thick in order to be sufficiently robust to resist distortion during the frequent heating and cooling cycles. Cast aluminium moulds are particularly suitable for moulding complex shapes, but they should be free from any porosity since this will lead to porosity in the moulding. Because mild steel has relatively poor thermal conductivity (about one quarter that of aluminium), thinner gauge sheet of about 2 mm thickness is used. Copper and nickel are used for mouldings where fine surface detail and high gloss are required, but these moulds are more expensive.

Because the quality of the inner surface of the mould is accurately reproduced on the outer surface of the moulding, it is important that this mould surface is free from any roughness, weld-lines, etc. which will mar the appearance of the product.

The mould is constructed of two or more sections for easy removal of the finished product. Parting lines must give a close water-tight seal when the mould sections are clamped together, in order to prevent entry of cooling water into the mould and to minimise flash (see Figure 9).



Figure 9: Preparation of an Aluminium Rotomould to Ensure a High Quality Internal Surface Finish

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Mould Venting

Most rotational moulds require a venting system to maintain atmospheric pressure inside the mould during the process. This is to prevent pressure buildup in the mould during the heating cycle and formation of a partial vacuum during the cooling cycle. The size of the vent will depend on the mould size. Since vents leave a hole in the moulded article, correct placement is essential, eg. in an area which may be cut from the product or where it does not impair appearance or performance. Vents should also be located or shaped to prevent cooling water from entering the moulding; this may cause a water-track mark on the inside of the hot article which in some products may be undesirable.

Modified Moulds

Ideally a moulding of uniform wall thickness is required. However, if it is desirable to have areas of thinner section, then this can be achieved by either increasing the mould wall thickness for these areas or partially shielding the outside of the mould with an insulating material such as “Teflon” to reduce the heat transfer at these points.

For parts designed with large flat surfaces, stiffening ribs should be used for additional structural support and internal air pressure maintained in the mould during cooling to prevent warping. These stiffening ribs should have generous radii for an acceptable moulding; deep or narrow ribs will lead to under filling or bridging, respectively. Inserts for attaching to other items can be moulded into the rotational moulding. These inserts should be made either of a material with a high conductivity such as aluminium, or of a high-melting point plastic such as nylon, and their surface should be knurled to give better anchorage of the polyethylene. Integrally moulded-in threads are also possible (see Figure 10).



Figure 10: Large Rotomoulded Storage Container Showing Ribbing to Increase Stiffness and Reduce Warpage

Mould Release Agents

Since many complex rotational mouldings are not easily removed from the mould, it is usually necessary to use some form of release system. Release agents are often used to treat the mould surface.

Mould release agents for polyethylene should be selected with care, to ensure that no serious environmental stress crack hazard is introduced. The most effective release agents are silicone based, and these can be applied to the clean inner surface of the mould by spraying, brushing or wiping with a fine cloth. It is essential that only air-curing and bake-on silicone resins are used; free silicone liquids (oils and emulsions) migrate into the moulding and cause severe stress cracking in a very short time (the order of minutes). The coating of release agent can be force-dried or cured by passing the mould through the heated oven for a short cycle.

Effective mould release can also be achieved by coating the mould surface with a thin layer of “Fluon” or “Teflon” PTFE. A correctly-applied release coating should retain effective release properties for fifty to several hundred mouldings, although use of high temperatures or long cycles will reduce the number of effective releases.

The amount of release agent used depends on the type of polyethylene being moulded; low density polyethylenes require more effective release properties than medium density polyethylenes. The coating should be evenly applied to prevent unacceptable warpage and distortion in the moulding. Insufficient release agent will cause sticking of the moulding to the surface of the mould; too much release agent may lead to warping.

Secondary Finishing Operations

Once the moulding process has been completed, secondary finishing operations may be required to obtain the marketable product. These operations are generally performed manually, are time-consuming and hence expensive.

Such operations include trimming, drilling, cutting to provide openings in the wall section, welding of sections by hot bar or hot gas techniques, attaching fittings, or flame or chemical pre-treatment before painting or decorating.

PROCESSING PARAMETERS

The heating and cooling stages of the rotational moulding process have significant effects on the quality of the mouldings, as measured by appearance, impact strength, tensile properties, stiffness and environmental stress crack resistance (ESCR).

The Heating Cycle

In the heating stage, sufficient heat must be transferred to fuse the polyethylene powder within the mould. The rate of heat transfer is controlled by both the oven temperature and the heating time, often termed the Oven Residence Time. The amount of heat required will depend on the desired wall thickness of the moulding, the complexity of the mould shape, and the characteristics of the powder and of the polymer melt. Too little heat (under-fusion) will give a porous low strength moulding; too much heat (over-fusion) will lead to degradation of the polymer and a drastic drop-off in the physical properties.

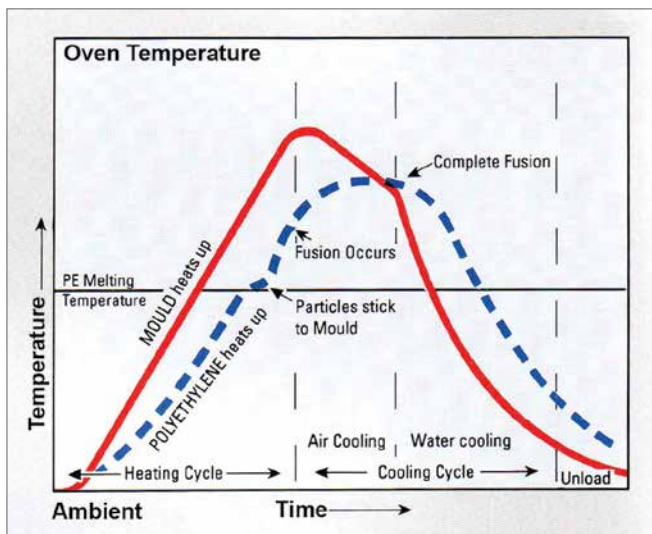


Figure 11: Typical Rotational-moulding Cycle

A typical moulding cycle is shown in Figure 11. After the mould enters the oven, its temperature rises, which heats the tumbling polyethylene powder within the mould. When the melting point of the powder is reached, the powder particles falling against the hot wall of the mould begin to melt and adhere to it until the mould surface is completely coated. Eventually all of the free powder melts, or sinters, the molten polymer then builds up to form a porous coating of uniform thickness.

At this point the coating has a very rough sandy (powdery) inner surface and contains a large number of trapped air bubbles and voids, i.e. it has a high degree of porosity. These observations are indicative of under-fusion and the mouldings are characterised by poor tensile properties, low impact strength and ESCR (see Table 3).

With further heating, most of the air bubbles are released and the coating completely fuses into a homogeneous densified layer of molten polyethylene. Optimum fusion occurs when a smooth dull inner surface is obtained, and impact and ESCR properties are at or very close to their maximum values.

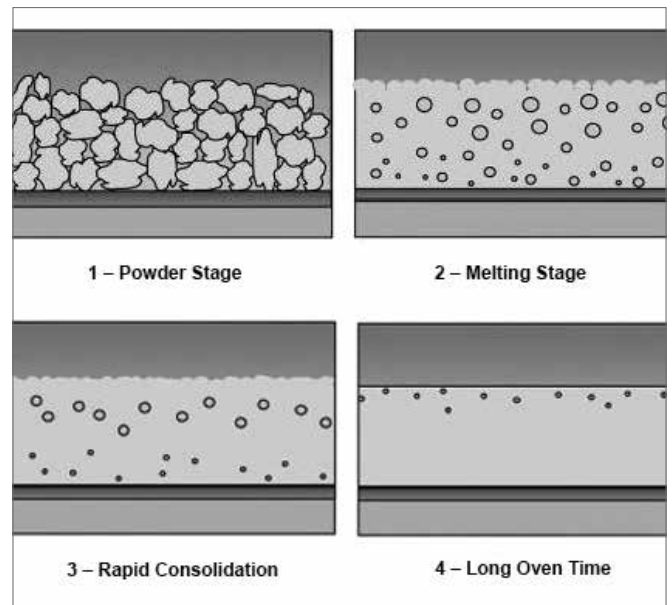


Figure 12: Schematic of the Densification of Polyethylene Powder in Rotational Moulding

The few remaining bubbles will generally have little effect on the final properties. However, when the product is one that will be subjected to severe service conditions, every attempt should be made to minimise the porosity.

If the powder is heated for too long, this results in thermal degradation of the polyethylene. *Alkatuff* LLDPE powders for rotational moulding are stabilised with antioxidants to protect against this effect. Once this antioxidant is consumed, the inside surface of the moulding will begin to oxidise and small cracks can be created. This over-fusion is easily recognised by a glossy inner surface, a yellow discoloration, and a strong odour. As the degradation proceeds, physical properties such as impact strength and ESCR will be seriously impaired.

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If long oven residence times and/or high temperatures are unavoidable, then the air within the mould can be replaced with nitrogen, but this is a costly practice.

The Optimum Heating Phase

The optimum oven residence time can be determined by trial and error, by producing mouldings at a fixed oven temperature for various oven times.

Visual inspection of the internal surface of the mouldings will give a good indication of the state of fusion (e.g. absence of powder residues, smoothness of surface, gloss level, lack of porosity, minimum odour and colour development). This can be backed up, if necessary, by performing mechanical tests on the walls of the mouldings (for example, impact tests), measuring the drop-off in MFI, or using Infra-red Spectroscopy or Thermal Analysis to follow the chemical degradation. Impact tests at low temperature are probably the quickest and most sensitive for checking properties. Figure 13 indicates typically the effect of oven residence time on the impact strength of a rotationally-moulded polyethylene.

In large or complex-shaped mouldings, there may be unequal fusion and hence thickness or property variations in different parts of the moulding. An optimum oven residence time may be difficult to determine in this situation, and the design of the moulding and the mould construction may require modification.

In summary, the effects of fusion time on the properties of the moulding are listed in Table 3 (on next page).

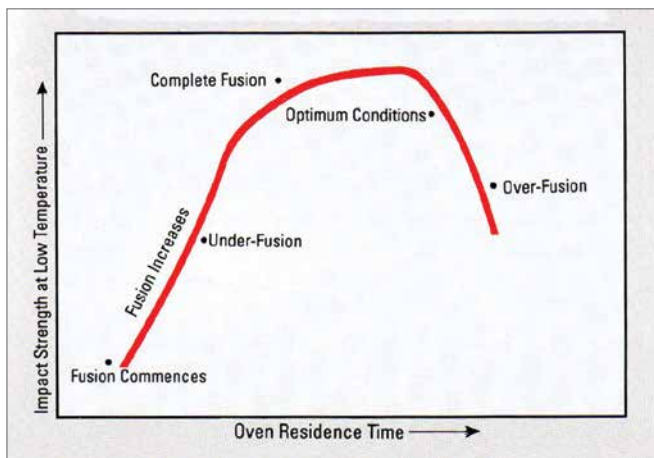


Figure 13: Effect of Oven Residence time on Impact Strength of Rotationally-moulded Polyethylene without Bubbles

The maximum impact strength can be achieved at shorter oven times by increasing the oven temperature, however very high temperatures may be detrimental to properties. *Alkatuff* type LLDPEs are characterised by having a broad latitude in oven residence time and oven temperature conditions for achieving optimum fusion.

Table 3: Effects of Degree of Fusion on the Properties of Rotationally-Moulded Specimens.

Degree of Fusion	Observations	Physical Properties
Severe Under-fusion	Very rough inside surface High porosity Large number of bubbles Powder residues	Low impact strength Low ESCR Poor tensile properties
Some Under-fusion	Smoother surface Some porosity at inner surface	Physical properties almost at optimum
Optimum Fusion	Smooth surface Dull finish No porosity	Physical properties at optimum
Slight Over-fusion	Glossy inside surface Sharp odour	Reduced impact strength Low ESCR
Severe Over-fusion	Sharp acrid odour Very glossy inner surface Yellow discolouration of inner surface	Drastic drop-off in impact strength Low ESCR

The Cooling Cycle

On removal of the mould from the oven, the cooling cycle is commenced. Because of the thermal mass of the mould, heat will continue to be transferred to the polyethylene for a short time to complete the fusion process, even though cooling is being applied.

The rate of cooling markedly influences the final properties of the moulding, as well as the degree of distortion and shrinkage that occur. This is particularly so with medium density, more crystalline *Alkatuff* LLDPE grades than with the lower density grades. Careful control of the cooling cycle must be carried out to obtain the best balance of physical properties and dimensional stability.

Rotational mouldings which are air-cooled experience a very slow cooling rate. This reduction in the rate of cooling promotes crystalline growth of the spherulites in the solidifying polyethylene. Formation of larger spherulites in the polymer results in higher density, stiffer mouldings that have greater resistance to distortion and warpage. Conversely, generation of such higher density mouldings are impacted by a reduction in toughness (e.g. impact strength and ESCR).

On the other hand, fast cooling, or quenching, limits the crystalline growth in the polyethylene. This produces mouldings with decreased density and improved impact strength and ESCR at the expense of stiffness.

Severe warpage may also be experienced because of non-uniform cooling in different parts of the moulding. If the cooling rate is too fast, mould life may deteriorate because of the continued thermal shock to the moulds.

Hence a compromise must be made in determining the cooling rate, to maximise the toughness and yet have sufficient stiffness and avoid warpage of the moulding. For some mouldings, it is advantageous to use a two-stage cooling cycle, by subjecting the mould to forced-air cooling followed by a water spray or atomized air-water spray – distortion can often be minimised by following this technique.

To increase production rates, the moulding can be stripped from the mould while it is still warm and then cooled further outside the mould. However care must be taken that the moulding maintains its proper shape during this subsequent cooling.

Faster cooling cycles can be obtained by using internal cooling, in which air is introduced into the moulding via the venting system; this technique is also advantageous in preventing the moulding prematurely pulling away from the mould wall, leading to excessive warpage and shrinkage. (Care should be taken, however, as to whether the mould can withstand such internal pressures).

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PROPERTIES OF ROTATIONAL MOULDINGS

Stiffness/Rigidity

The stiffness or rigidity of a rotationally moulded article produced from *Alkatuff* LLDPE resins depends upon the density of the polymer being used, the wall thickness of the moulding, and the rate of cooling. Increasing the density leads directly to increased stiffness. Slow cooling will promote a higher moulding density and hence increased stiffness. Additional rigidity can very often be obtained by incorporating ribs or rims in the design of the article.

Toughness (Impact Resistance and ESCR)

The toughness of rotational moulded polyethylene articles, as measured by low temperature impact properties and environmental stress crack resistance (ESCR), depends on the choice of the polyethylene used, the rotational moulding cycle, and the design of the moulding.

These properties are highly dependent on the MFI and density of the polyethylene and, as shown in Figure 1, low MFI and low density are preferred for maximum toughness. Intermediate density grades of high MFI (e.g. above 20 g/10min) can be prone to brittle failure.

As indicated in the previous section, the toughness of the rotational moulding can be maximised by careful control of the heating and cooling cycles of the process. The heating time and temperature must be sufficient for optimum fusion of the powder (see Figure 9). Rapid cooling of the moulding is desirable for lower densities and hence greater ductility.

In most applications, mouldings made from *Alkatuff* LLDPE using the guidelines above are unlikely to break in service. However mouldings may have a tendency to exhibit premature failure under certain conditions if there are notches, sharp corners or rough edges on the moulded part, or if the moulded article is subjected to high stress rates, impact at low temperatures, or stress in the presence of stress-cracking liquids.

Shrinkage

The shrinkage of polyethylene rotational mouldings during the cooling stage is a very complex phenomenon and depends on a number of variables. This makes it very difficult to predict the exact shrinkage that will occur in any one part of a moulding. There is no alternative to the practice of carrying out moulding trials with a mould having dimensions estimated as correctly as possible and then modifying the mould where necessary

The shrinkage of mouldings depends mainly on:

- 1. The grade of powder used.** Shrinkage is greater for polyethylenes of higher density.
- 2. The melt temperature.** The higher the temperature the greater the shrinkage.
- 3. The thickness of the moulding.** Shrinkage increases with the thickness of the moulding.
- 4. The shape of the moulding.** Uniform shrinkage in all dimensions can be expected only with symmetrical shapes, e.g. tubes and spheres. Shrinkage can vary between 1% and 5% in various parts of complex shaped mouldings.

Other factors which have some effect on shrinkage are the rate of cooling and the excessive use of mould release agents.

Distortion or Warping

Distortion or warping can be a problem with the rotational moulding of flat-sided articles, and is the result of residual or moulded-in stresses. The difficulties are greater with higher density polymers and with thicker wall sections (see Figure 14).



Figure 14: Rotomoulded Case with Large Flat Sides – Moulded Ribs Minimise Distortion and Warpage

The most important factors affecting distortion are the rate and uniformity of cooling. Rapid cooling can cause severe distortion in most mouldings because the temperature gradient between the inner and outer layers of the moulded wall section leads to non-uniform cooling rates. Slow cooling rates result in more uniform cooling of the moulding and smaller temperature differences across the moulded section. However slow cooling is not always desirable because it results in higher densities and a proneness to brittle mouldings. The choice of the grade of powder with the best balance of MFI and density is therefore important, so that the rate of cooling necessary to avoid distortion can be achieved without making the moulding brittle.

Adequate venting, a pressurised mould during the cooling process, reduction in the amount of mould release agent used, and the introduction of stiffening ribs to the moulding will also help to minimise distortion.

Chemical Resistance

Although polyethylene resins are used in a large range of end applications, careful consideration needs to be made of the choice of polymer that will meet the demands of the finished product and the environment(s) that it will be exposed to (e.g. oils, fats, alkalis, acids and temperature, etc.). To make the best resin selection, customers are advised to discuss their specific end product requirements with their Qenos Technical Service Representative.

HANDLING PRECAUTIONS

Although *Alkatuff* LLDPE powders are inert and present no toxic hazard, care should be taken when handling the powders to minimise atmospheric dust levels and avoid any risk of a dust explosion. This dust hazard is less than with materials such as flour and starch, but precautions should be taken.

The prime requirement is that no spilt powder should be allowed to accumulate, such that when agitated it can create a dust cloud. Heat sources of more than 350°C can cause auto-ignition of polyethylene. Attention should be given also to the siting and selection of electrical equipment used in the powder handling areas. Working areas should be well ventilated.

In large scale operations, additional precautions are required, since hazards may be introduced by such items as bulk containers, conveying systems and intermediate hoppers. Special attention should be paid to the possibility of electrostatic charge accumulation which could cause spark formation. This could happen with unearthed metal parts or when non-conducting parts are used in the wrong situation.

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APPENDIX 1 – ROTATIONAL MOULDING TROUBLESHOOTING GUIDE

Problem/Issue	Cause(s)	Potential Solution(s)/Action(s)
Air Entrapment	Insufficient heating	Cook to a higher peak internal air temperature
	Powder quality	Powder may be too coarse or contains tails. Consult powder supplier
	Cycle time too short	Increase heating time
Corners do not fill	Design – sharp corners or small gaps between walls	Redesign mould
	Powder has poor dry flow	Contact Powder supplier
Failure during Chemical Storage	Contents not compatible with container	Discontinue or revise service life appropriately
	Design – Stress concentrators present	Redesign mould
	Thickness insufficient	Increase shot weight
	Service temperature too high	Increase shot weight, reduce maximum exposure temperature
Flashing	Contamination in part line	Ensure good cleaning practices
	MFI of polymer too high	Change to a lower MFI grade
	Unevenly clamped mould	Check clamping mechanism
	Poor venting	Check vent is not blocked and is of sufficient size
Insufficient Rigidity in Moulding	Wall thickness too thin	Increase shot weight
	Density of Resin too low	Trial higher density resin
	Design	Design stiffening features or ribs may be required
Odour, shiny yellowish internal surface	Overcuring	Reduce temperature or heating time. Reduce peak internal air temperature
Parts Stick in Mould	Insufficient mould release due to inadequate application	Reapply mould release
	Mould release diminished with number of shots	Reapply mould release
	Design	Check for undercuts
	Mould damage	Inspect mould and repair if required
	Poor mould finish	Inspect mould and repair if required
Poor Impact Properties	Design	Review Design, minimising any sharp corners or areas of stress concentration
	Undercure – Small voids are present in wall sections	Cook to a higher peak internal air temperature
	Overcure – Internal surface is shiny	Cook to a lower peak internal air temperature
	Choice of Polymer	Lower Density and/or lower MFI grades may enable increased impact resistance
Wall Thickness Variation	Poor powder quality. Tails and poor flowability	Contact Powder supplier
	Incorrect rotation	Alter rotations
	Variable heating of the mould walls	Check for thickness variation of the mould wall. Check for shielding effects
Warpage	Uneven Cooling due to large thickness variations	Alter rotation ratio. Review design
	Uneven Cooling due to premature separation from mould surface	Reduce mould cooling or apply internal cooling. Reduce mould release
	Poor Venting	Check vent is not blocked and is of sufficient size

Disclaimer

The proposed solutions in this guide are based on conditions that are typically encountered in the manufacture of products from polyethylene. Other variables or constraints may impact the ability of the user to apply these solutions. Genos also refers the user to the disclaimer at the beginning of this document.

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