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Removal of pinholes and bubbles from rotationally moulded products

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The rotational moulding processing method for plastics is steadily expanding its product range and market areas. However, a drawback of the process has always been the surface pinholes and internal bubbles which occur in the products due to the inherent nature of the process. In an attempt to alleviate this problem, a programme of research has been carried out to investigate the factors that affect the formation of the bubbles and more importantly influence their removal.

This paper describes how bubbles in rotationally moulded products can be removed by pressurizing the inner atmosphere of the mould. It has been shown that bubbles were successfully removed from MDPE (medium density polyethylene) by introducing a low pressure (0.5 bar) inside the mould after the polymer had melted. The removal of bubbles using pressure was shown to increase the impact properties (25 per cent) and the tensile properties (5 per cent) of the moulding while also reducing the cycle time. Pressure was successfully used to remove bubbles from other rotational moulding materials, such as polypropylene, Nylon 12, polycarbonate, ethylene vinyl acetate and ethylene butyl acetate.

Key words: rotational moulding, plastics, pinholes, bubbles, pressure, rotomoulding

1 INTRODUCTION

Rotational moulding is a method used to produce hollow plastic articles. It is also known as rotocasting or rotomoulding and is unique among plastics moulding processes because the heating, shaping and cooling of the plastic all take place inside the mould with no application of pressure. The concept is simple. A predetermined charge of cold plastic powder is placed in one half of a cold mould—usually sheet steel or cast alu-

minium. The mould is then closed and rotated biaxially in a heated oven as shown in Fig. 1. As the metal mould becomes very hot, the plastic powder tumbling inside starts to melt and coat the inside surface of the mould. When all the powder has melted, the mould is then transferred to a cooling environment. The biaxial rotation continues until the plastic has solidified. At this point the mould is opened and the product is removed. The article being produced need not be hollow since finishing operations such as cutting or sawing can be used to make, for example, right- and left-handed articles (1, 2).

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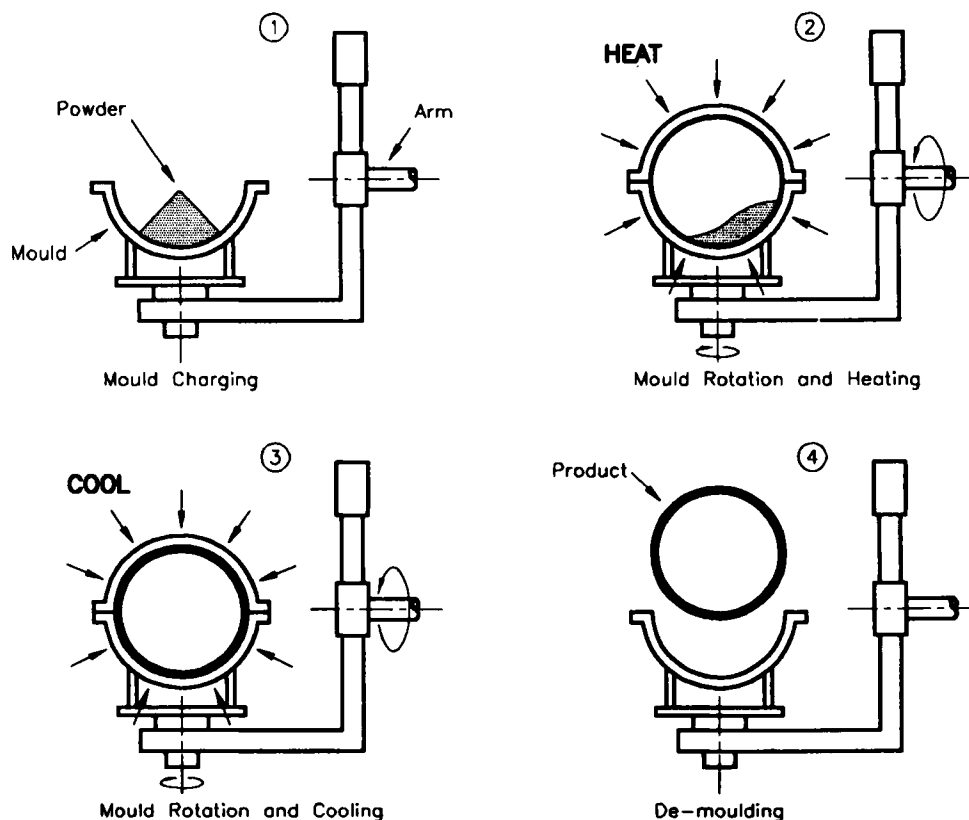


Fig. 1 Principal stages in rotational moulding

Polyethylene tends to dominate the market as a rotational moulding material due to its high thermal stability and the wide range of grades which have been developed specifically for the process. However, other thermoplastics such as polyvinyl chloride (PVC), acrylonitrile butadiene styrene (ABS), polycarbonate (PC), polyester, polyamides (Nylons) and even some thermosets can be rotomoulded successfully. Although one of the less well known plastics processing techniques, rotational moulding offers tremendous design capabilities. Several years ago the rotational moulding industry primarily consisted of moulders producing tanks and drums. However, recent technological developments have given the industry a new lease of life, coupled with an enthusiastic attitude towards the future (3–5).

The need for process control, which was so apparently lacking in the past, has recently been provided by the Rotolog diagnostics system (6). The Rotolog system, which monitors the internal air temperature of the mould, provides the moulder with precise information necessary for controlling the cycle. Designers are now beginning to realize the advantages of the process, producing more intricate products. Decorating rotationally moulded parts has also become increasingly sophisticated with mould-in graphics now available. With this technique, graphics are literally embedded into the resin during the rotational moulding process, enhancing the overall quality of products by promoting brand names or decorating parts. Recent advances in rotational moulding machinery have given the industry a necessary productivity boost. Computer control has helped to automate the process, increasing efficiency (7, 8).

Although rotational moulding appears simple, the plastic material undergoes complex heat transfer and flow situations while it is in the mould. These are, of course, influenced by the process conditions such as rotational speeds, oven temperature, oven time, cooling methods, etc., but their precise effects are unclear. Experienced moulders are able to produce quite complex shapes based on past knowledge, but they openly admit that they are being stretched by the ever-increasing technical demands which are being placed on rotomoulded articles. One area where there is a lack of understanding is the mechanism of bubble formation in the wall section and surface porosity of such articles. The presence of internal bubbles is a characteristic feature of products manufactured by rotational moulding. Bubbles in mouldings are generally undesirable since they reduce stiffness and impair the appearance of a product or cause hygiene problems in some products if they occur at the surface. The reasons why these bubbles and pores occur are not completely clear and their nature, distribution, mobility and removal are matters of continuing speculation. During this work a number of parameters were identified as contributing to the formation and/or removal of bubbles in rotomoulded products. These included:

1. Powder—particle shape, size and distribution
2. Viscosity of melt
3. Additives (for example pigments)
4. Mould surface
5. Temperature
6. Time

7. Atmosphere inside the mould
8. Surface tension
9. Vacuum
10. Pressure

This paper presents some of the results from the research carried out on bubbles in rotomoulded products. These results concentrate specifically on the use of gas pressurization of the mould and the application of a vacuum to remove bubbles and surface pores from rotationally moulded products.

2 HISTORICAL BACKGROUND OF BUBBLES IN ROTATIONAL MOULDING

The problem of bubbles in rotationally moulded products has been recognized for the last 30 years. In 1965 the United States Industrial Chemicals Company published a paper on rotational moulding of polyethylene powder (9). They suggested that the bubble problem might be solved by raising the oven temperature, increasing the heating cycle, using a material with a high melt flow index or using a material with a lower density. Since then, a number of authors have targeted particular areas of the rotational moulding process as being responsible for the appearance of surface pores and bubbles within the walls of rotationally moulded products.

One such area which appeared to affect the existence of bubbles was the geometry of the particles being rotationally moulded. McKenna (10) of Union Carbide explained that bubbles are caused by air entrapped between powder particles. If the powder particles have irregular, long thin protrusions, this will give rise to a low packing density, and hence more bubbles will be trapped in the powder pool.

In 1972 Rao and Throne (11) also related particle size to the concentration of pores on the surface of the moulded part. An additional amount of finer particles in the powder mixture will reduce porosity, improve heat transfer to the plastic and help the powder flow by lubricating the movement of the larger particles. Ramazzotti (12) pointed out that larger particle sizes, when used with a high-viscosity material, result in poor surface reproduction and a tendency to trap air in the form of bubbles when the part is formed.

Moisture content was recognized by several authors (13–15) as a cause of bubbles and pores in moulded parts. Both the quality and surface finish are impaired, and the strength of the product is considerably reduced when hygroscopic raw materials pick up excess moisture in transit and storage. The grinding of these resins greatly increases the surface area that is available for moisture pick-up. The moisture can vaporize during moulding and create bubbles and surface pitting of the part. The addition of carbon black pigmentation can also increase the rate at which moisture is picked up (16). The problem of moisture does not apply singularly to polyethylene, indeed it is more widely associated with materials such as polycarbonate, Nylon and ABS. It is recommended that these latter materials be dried prior to moulding. Polycarbonate in particular is known to produce mouldings with a high proportion of bubbles and pores. It has been suggested (16) that the problem could be alleviated by increasing the internal pressure using nitrogen gas.

Other, more specific areas that referred to the problem of bubbles included Carrow's (17, 18) work with cross-linked polyolefins. Carrow stated that the size and thickness of the part magnify the bubble problem inherent in moulding cross-linked polyolefins and he also suggested that bubbles are caused by the decomposition of the cross-linking agent. Harkin-Jones (19) performed a detailed investigation of the problem of bubble formation in PVC plastisols. This work showed that the rate of viscosity increase will affect the number of bubbles that form. Howard (3) discussed the problem of bubbles and surface pores which were caused by adding a percentage of regrind material with a virgin polymer. He found that if more than 10 per cent of regrind material was used, the surface porosity and bubble content of the product increased.

2.1 Advantages of removing bubbles

There are many advantages to be gained from removing bubbles and surface pores from rotationally moulded parts, the most obvious improvement being to the aesthetic quality of the product. When surface pores in particular are removed, products are much more suitable for outdoor applications and also applications where hygiene is important. The removal of surface pores also improves the general appearance of the product. Another major advantage is the increase gained in mechanical strength when bubbles and pores are removed. McKenna (10) quoted some mechanical properties of the plastic, indicating that a void-free part has improved low-temperature impact strength and a higher elongation at failure, relative to a sample tested with a large distribution of bubbles.

Extensive research has been done in the area of impact strength (20–23). Crawford and Nugent linked the optimum impact strength to cycle time (20). They concluded, as have others (24, 25), that it is best to slightly under-fuse rather than over-fuse the product. This is to prevent oxidation of the inner surface. Therefore, recommendations to increase the oven temperature or increase cycle time to remove bubbles will have an adverse affect on the impact properties of the part. Kelly (26) has shown this to be the case.

Kelly has perhaps performed the most extensive work, relating the presence of bubbles in the parts wall to a detrimental effect on physical properties. He suggests this is because bubbles prevent stresses imposed on the part from being uniformly distributed throughout the wall. In a series of tests, Kelly investigated the impact strengths of various samples moulded at optimum conditions. His results indicate that samples with a high distribution of bubbles had a reduced impact strength (25 per cent), compared to samples with very few bubbles. Examination of the impact samples at the point of initiation showed that the failure crack propagated through the centres of a series of bubbles in the wall always followed the path of least resistance. He concluded that a high concentration of bubbles in the wall reduces both the energy for crack initiation and propagation. Kelly also investigated the theory of increasing cycle time to remove bubbles. He discovered that additional heating time caused thermal degradation of the polymer which seriously weakened the structure. For an extra 2 minutes over the optimum cycle

time, the impact strength dropped by as much as 75 per cent.

2.2 Bubble formation

It is apparent from the beginning of this section that a large number of authors have referred to the presence of bubbles in rotationally moulded products. However, only a few researchers have made a serious attempt at explaining how bubbles are formed and how they are removed. In an initial attempt at this problem, Rao and Throne (27) drew extensively from the theories of metal sintering and glass densification. It was suggested that the formation of a homogeneous melt from powder particles involves two distinct steps:

1. The particles stick or fuse together at their points of contact. This fusion zone grows until the mass becomes a porous three-dimensional network, with relatively little density change. This is referred to as *sintering* (see Fig. 2).
2. At some point in the fusion process, the network begins to collapse, and the void spaces are filled with molten polymer. This is referred to as *densification* (see Fig. 3).

Ten years later, Progelhof *et al.* (28) further developed the powder densification theory. Their updated theory suggested that as the powder is heated, the particles become sticky and adhere to each other, and upon further heating, the particles fuse together or densify to form a unitized structure. From observing hot plate experiments, Throne and his associates concluded that as the heating process continues, the solid/melt interface moves upwards, the top of the free surface of the powder drops and with time the powder completely melts. Throne and co-workers also observed the forma-

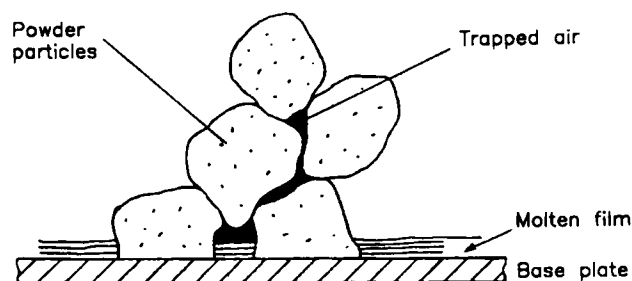


Fig. 2 Model for sintering concept

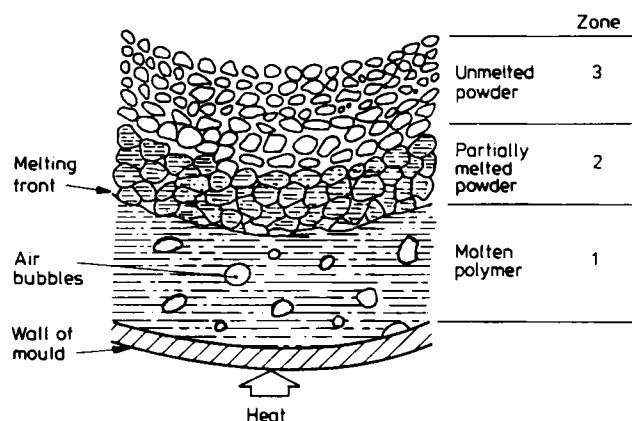


Fig. 3 Densification of powder into molten polymer

tion of voids. The voids appeared to be a result of a bound inclusion of the space between individual particles, and the most striking point was the slow movement of voids to the free surface. There appeared to be some coalescence of voids, but with time the voids diminished in size.

Another person to consider powder densification was Kelly (26) of Du Pont Laboratories, Canada, in an unpublished paper. Kelly suggested that air bubbles are trapped in the polymer during melting and decrease in diameter as the melt temperature increases. The high viscosity of the melt prevents movement of the bubbles. At a high enough melt temperature, the air in the bubbles begins to dissolve into the polymer. Oxygen has about twice the solubility of nitrogen in polyethylene. At high temperatures, the oxygen is further depleted by direct oxidation reactions with polyethylene. The depletion of oxygen reduces the bubble diameter. At thermodynamic equilibrium, the pressure inside a bubble of radius R exceeds the pressure in the fluid by an amount $2\gamma/R$, where γ is the interfacial tension. This pressure differential forces the nitrogen to dissolve in the polymer; thus the bubble diameter is further reduced and this chain of events repeats until the bubble disappears. As cycle time increases, the size and quantity of bubbles decrease. At very long cycle times, the moulded part will have no bubbles. However, the impact strength of the moulded item may be reduced due to oxidation.

Crawford and Scott (29, 30) carried out hot plate tests on powders, using video equipment to record and examine the processes of melting in detail. They based the conditions on earlier measurements made during actual moulding trials. Vacuum-dried powders confirmed that solvent residue or moisture was not involved in the formation of bubbles. The video recording allowed the formation and subsequent dissolution of bubbles to be recorded and modelled. They described the melting and collapse of the powder as heat is applied from the base as two bulk movements: firstly, the progression of the melt front through the powder mass to the free surface and then the collapsing of powder particles into the melt. It is the latter that tends to trap air. A force analysis on a typical bubble shows that the apparent viscosity of molten polyethylene is so large that the buoyancy forces acting on it are insignificant. They also stated that the trapped air will diffuse into the surrounding polymer mass and produced bubble measurements to support this. The initial size of the bubble has a significant effect on the rate at which it dissolves, as the surface area–volume ratio is inversely proportional to the diameter. They found that this initial size was dependent on the size of the particles and that the number of bubbles was dependent on the particle size distribution and the MFI (melt flow index) of the material.

Crawford and Xu (31) complemented Scott's earlier work on bubble analysis. They derived a semi-empirical relationship between the bubble diameter ratio, temperature and time. From this relationship the bubble diameter was predicted as a function of time and melt temperature.

2.3 Mould pressurization

Internal bubbles and external surface pores on plastic products are unique to the process of rotational mould-

ing. Other plastic processes such as injection moulding and blow moulding do not have this problem. Another novelty with the rotational moulding process is that mouldings are manufactured in an unpressurized atmosphere, which produces relatively stress-free articles. However, the application of pressure has been successfully used to remove bubbles in other processes.

High pressure (300 bar) was used by Letterrier and G'Sell (32) to remove voids from thermoplastic matrix composites at 200 °C. Cylindrical voids as large as $\varnothing 9.5$ mm \times 4 mm were successfully removed using compression techniques. The removal of a void was found to be a two-stage process. Firstly, the void was reduced in size by the compressive force exerted on the matrix. Secondly, the high pressure provoked dissolution of the gas. However, high pressure is not suitable for the process of rotational moulding due to the practical implications. Theory governing the deflection of plates (33) dictates that even a moderate pressure of 0–10 bar would destroy the majority of mild steel fabricated moulds in the rotational moulding industry today. Aluminium cast moulds with 10 mm thick wall sections are more suited to pressurization (1–2 bar), provided the mould surface area is small.

There are a number of references concerning pressurization of the mould during rotational moulding. Smith (13) recommended sealing the vent port to help ensure a bubble-free part. He suggested that with the vent port sealed a partial vacuum would be created inside the mould during the cooling cycle. This partial vacuum would tend to draw the bubbles to the inner surface of the moulding, whereupon they would burst.

The use of pressure during rotational moulding was also investigated by Nugent (34) during his PhD research. Nugent performed a series of tests using air pressure from 0.5 to 2.5 bar piped through the arm of the machine into the mould via the vent port. The air was free to circulate within the mould and pass back out through the vent port. This meant that the actual internal pressure inside the mould was considerably lower than the inlet air pressure. From these experiments using two different materials, Nugent experienced no significant change in bubble density or in the time required for bubbles to disappear.

Sharp (35), back in 1969, made reference to mould pressurization as a future development and way forward for the rotational moulding industry. He stated that further sophistication of the process would involve internal mould access, in order to allow for internal mould pressurization, for example, to hold the cooling part against the mould wall. Sharp's prediction has come true. Leaversuch (36) points out that McNeil Akron (a machine manufacturer) and others now introduce an inner gas pressure of 0.07–0.14 bar into the mould. This gas pressure is used to hold the part against the walls of the mould in order to reduce warpage and shrinkage.

The Rotational Molding Development Centre (RMDC) at the University of Akron has conducted extensive research (37–39) on mould pressurization as a method to reduce warpage and shrinkage. The RMDC found that warpage in rotationally moulded parts was associated with rapid cooling coupled with easy separation of solidified parts from the mould walls. Mould pressurization was used to simply hold the solidified

polymer against the mould wall and prevent it from separating and being able to distort, until it has sufficiently cooled, so that its modulus and related mechanical properties become sufficient to resist the thermal quench stresses.

3 MATERIALS, EQUIPMENT AND EXPERIMENTAL METHODS

A Caccia RR 1400A shuttle-type machine was used for all moulding tests during this work. This machine has an offset arm, capable of carrying moulds up to 175 kg with a maximum diameter of 1400 mm. The oven is heated by a gas-generated flame, with burner capacity of 77 000 kcal/h which allows a maximum operating temperature of 400 °C. A central pipeline through the arm of the machine provides a means to pressurize the mould. In order to provide accurate control of the pressurization system, a control panel was designed, manufactured and fitted to the rotational moulding machine. The panel allowed a pressure line, in the form of air or any other gas, to be connected. The pressure was then piped through a relieving regulator, a $\phi 100$ mm pressure gauge, and then through a solenoid-operated control valve, to the machine arm and mould. A pressure connector was designed and fitted to the mould to allow pressure to be applied, and a thermocouple probe via the same port.

During the rotational moulding trials a cast aluminium mould was used for all experiments involving pressure. The mould was approximately cubic with 260 mm sides \times 10 mm thick, and contained a 100 mm cube inverted corner. The mould was split along its centre-line with each half having a 3° draft angle. All internal corners of the mould had a 25 mm radius. Sealing was achieved using an 'O' ring. Typically a 1 kg shot size was used to produce a 3 mm thick part. The majority of the experimental work was carried out using standard rotational moulding grades of polyethylene, which accounts for 90 per cent of the materials used by the rotational moulding industry (5). However, other less commonly used materials, which experience similar bubble and pinhole problems, were also used (see Table 1 for details).

During the course of this work a test circuit was designed and manufactured to enable pressure experiments to be conducted using a hot plate. The circuit included a hot plate, a controlled pressure line and a sealed hot plate rig. The hot plate rig incorporated an

internal melting chamber and a detachable back door, to facilitate the removal of the test specimen. The test rig also included a sealed viewing port to observe the powder as it melted and compression-fitted thermocouples to record the melt temperature. This apparatus was used to observe the effects (using a video camera) of different pressure levels on bubbles contained within the melt. The measurement of bubble diameters from videotape was achieved using a computer hardware package called VIDI PC 24. The system which operated within 'Windows' was linked directly to the video recorder. Bubble images were transferred from the video to the PC screen where they were analysed and their sizes recorded in pixels. By video recording an object of known size, a pixel to millimetre scaling factor was established. The size of each bubble was then converted to millimetres.

Some mechanical properties in the form of low-temperature impact energy (-20°C) and tensile strength were determined. The total energy required to fracture moulded samples was determined in accordance with ISO 6603-2 using a Rosand IFWIT7U computer-controlled drop weight impact tester. The tensile strength, modulus and percentage elongation were determined in accordance with ASTM D638M-89 using a JJ Lloyd L6000R computer-controlled machine.

4 DISCUSSION

4.1 Pressurized hot plate experiments

The effect of pressurization was initially investigated using NCPE 8017 (natural polyethylene, MFI 3.2 g/10 min), with the aid of a sealed test rig and a hot plate set at 300 °C. From these tests, it was observed that there was little or no difference experienced in the diffusion rates of bubbles in atmospheric pressure, compared to the diffusion of bubbles in a pressurized atmosphere. For both sets of conditions, bubbles take between 15 and 25 minutes to diffuse at a relatively high melt temperature (190 °C). These initial observations were similar to what Nugent (34) experienced using pressure. Nugent found that the application of pressure caused no significant change in bubble density or in the time required for bubbles to disappear completely.

The reason why these initial pressure tests had no effect on bubbles or pores became apparent after considering the formation of bubbles in an unpressurized atmosphere. For a melt containing bubbles whose pressure is the same as that of the surrounding fluids, then

Table 1 Materials used during rotational moulding trials

Product code	Supplier	Type	MFI (g/10 min)	Density (kg/m ³)	Colour
NCPE 8017	Neste	Medium density polyethylene	3.2	934	Natural
NCPE 8113	Neste	Medium density polyethylene	3.2	934	Sky blue
NCPE 8605	Neste	Medium density polyethylene	8.0	935	Black
8107	Dupont	Low density polyethylene	5.0	924	Black
NCPE 8019	Neste	Ethylene butyl acetate	7.0	924	Natural
108VN5	ATOchem	Ethylene vinyl acetate	8.0	940	Natural
MA 3950	ATOchem	Nylon 12	—	1040	Natural
PPFC/RM	Tecmalen	Polypropylene	7–8	900	Natural
Markolon 2400	Bayer	Polycarbonate	17	1200	Natural

at some stage the bubbles will begin to contract to the radius at which the bubble pressure reaches $2\gamma/R$, plus the fluid pressure. If pressure is introduced at the beginning of the melting process, then the relationship between the pressure inside the bubble and the pressure of the surrounding melt remains the same, as does the rate of change of bubble radius.

It was postulated that for mould pressurization to be more effective, a pressure difference had to be created between the pressure inside the bubble and the pressure exerted by the surrounding melt. This was achieved by first allowing the polymer to melt under atmospheric conditions. Then, secondly, when the powder had completely melted, the pressure was introduced. This method of pressure application was used for a new series of hot plate tests. During these tests, the effect of 0.5, 1, 1.5 and 2 bar pressure on the diameters of bubbles in a polymer melt (natural polyethylene, MFI 3.2 g/10 min) at 190°C was investigated. Figure 4 illustrates how the diameters of bubbles that formed under atmospheric pressure conditions were influenced by increasing the applied pressure level. From the illustrations, it can be seen that pressure, if applied after the polymer has melted, can successfully remove bubbles from within the polymer melt in a matter of seconds. It would appear that increasing the applied pressure level increases the rate at which bubbles are removed.

The effect of pressure on the surface porosity of the part was also gauged using the enclosed hot plate rig. During this series of tests the rig/melt interface was heated to 200°C and then the pressure was applied for a set period of time, for example 1 bar for 10 seconds, then relieved and opened to atmosphere again. The polymer was then cooled and the number of surface pores contained in a 10 mm square area were counted. The polymer used for these tests was NCPE 8605 (black polyethylene, MFI 8 g/10 min), because surface pores were easier to see on this black material. The overall effect of a range of pressures (0.5, 1, 1.5 and 2 bar) on surface porosity was determined and illustrated graphically in Fig. 5. From Fig. 5 it can be seen that pressure removes surface porosity in a similar fashion to the removal of bubbles contained within the melt. It

appears that pressure removes the smallest surface pores instantaneously, and then gradually forces the air out of the remaining larger pores. Figures 6 and 7 illustrate the visual effect of removing bubbles from the surface of the product.

4.2 Pressurized rotational moulding of polyethylene

Initial rotational moulding trials confirmed that moderate pressure (0.5 bar) could successfully remove bubbles and pores if applied after the polymer had melted. Mould pressurization provided a simple mechanical means by which bubbles and pores could be removed from the product. Therefore it was decided to investigate in greater detail some of the effects of rotational moulding with pressure.

4.2.1 Effectiveness of pressure at different melt temperatures

It has been established (20) that the optimum point to remove a polyethylene moulding from the oven for cooling is when its internal air temperature reaches 200°C. When cooled, tests have shown that a peak internal air temperature of 200°C corresponds to the optimum mechanical properties of the moulding (20). It was decided to investigate whether the optimum mechanical properties of the part could be maintained using lower peak internal air temperatures, combined with pressure. It was hoped that the reduction in mechanical properties due to a lower melt temperature would be counteracted by the applied pressure removing weakness in the polymer structure due to bubbles and pores.

During these tests 1 kg of NCPE 8017 (natural polyethylene, MFI 3.2 g/10 min) was rotationally moulded using a sealed aluminium mould. The oven temperature was set at 300°C and the mould was removed from the oven at various internal air temperatures. While being removed from the oven, a 0.5 bar pressure was applied through the arm of the machine for a set period of time.

From these moulding trials the effectiveness of pressure was highlighted by taking cross-sections of the moulded parts. Figures 8 and 9 illustrate cross-sections

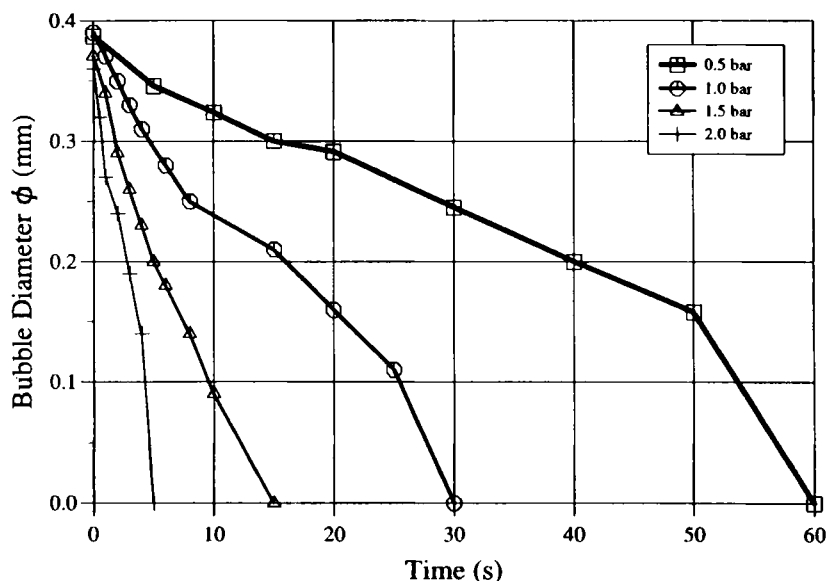


Fig. 4 Effect of pressure on bubble diameter

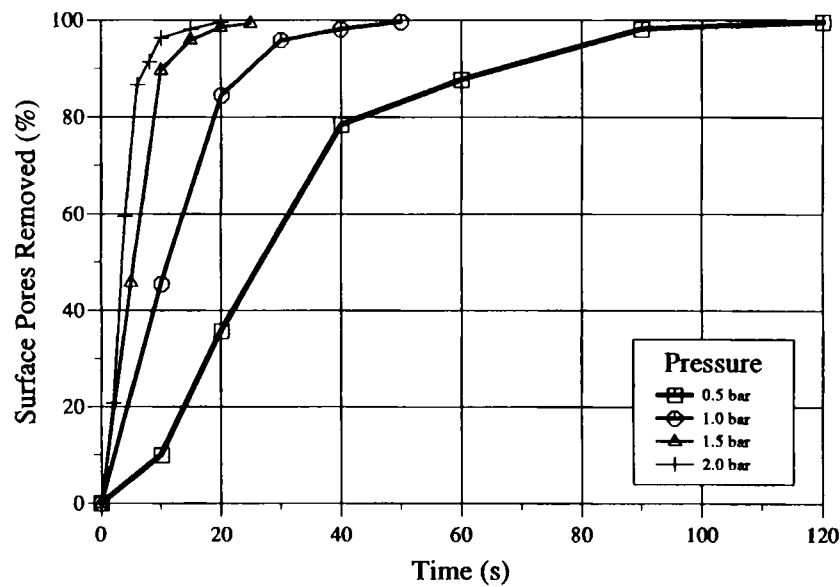


Fig. 5 Effect of pressure on surface porosity

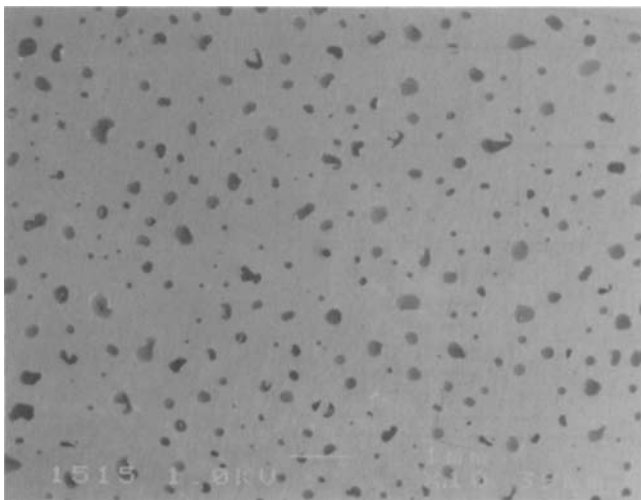


Fig. 6 Normal surface porosity—no pressure applied (magnification $\times 10$)

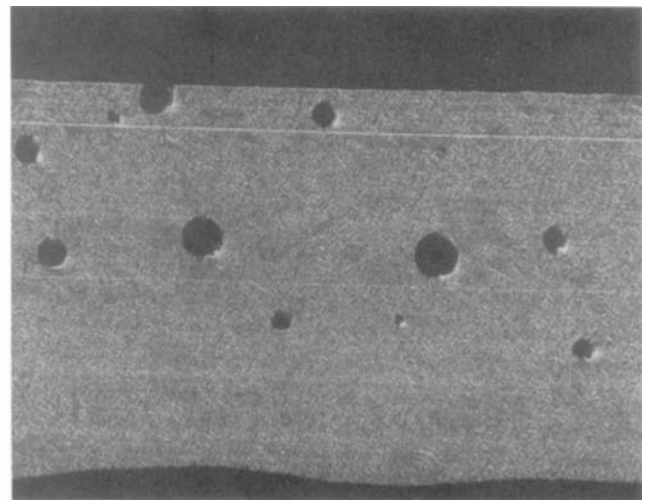


Fig. 8 Cross-section of moulding with no pressure applied (peak internal air 160°C , magnification $\times 29$)

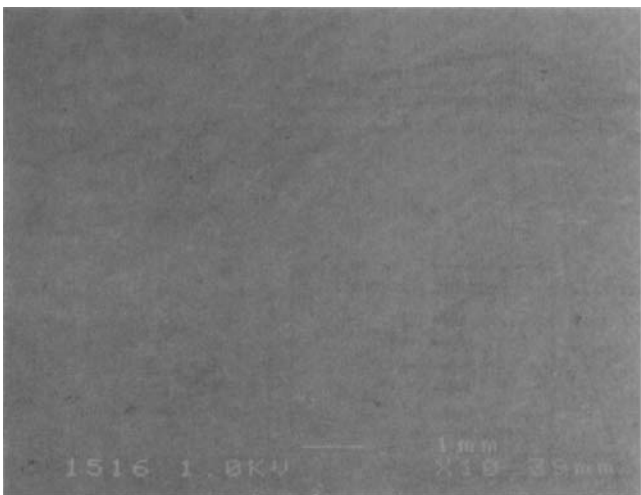


Fig. 7 Effect of 1 bar pressure applied for 60 seconds (magnification $\times 10$)

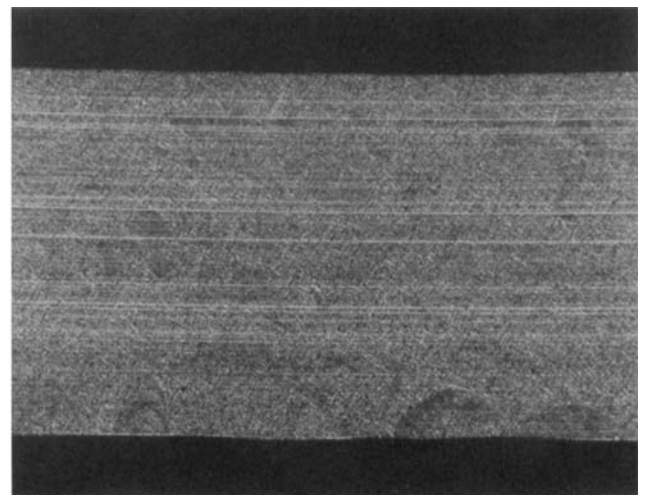


Fig. 9 Cross-section of moulding with 0.5 bar applied for 10 min (peak internal air 160°C , magnification $\times 29$)

using cross-polarized light at $\times 29$ magnification. From these illustrations it can be seen that the density of bubbles is decreasing as the duration of pressure increases. The micrographs also illustrate no apparent change in the crystalline structure of the polymer due to pressure. However, it would appear that pressure causes a levelling effect on the inner surface of the moulding.

Also, from these trials an approximate indication of the bubble density was visually gauged from the moulded parts. From the data represented in Fig. 10, it can be seen that the effectiveness of pressure to remove bubbles increases with increasing melt temperature. This can be related to the polymer viscosity which decreases as the melt temperature increases.

For the series of mouldings some mechanical properties such as impact strength and tensile modulus were measured to see if the use of pressure offered any advantages in this regard. Preliminary trials showed that there was no significant change in flexural properties.

Low temperature (-20°C) impact tests were carried out on samples from each moulding. The data from these tests, which are illustrated in Fig. 11, confirmed that mechanical properties increased when bubbles and pores were removed from the part. With no pressure applied, the bubble count is 100 per cent, which corresponds to the least energy that is required to break the sample. Bubbles weaken the polymer structure by providing crack initiation zones. When bubbles are removed, the impact strength typically rises by about 15–20 per cent, and on some occasions by as much as 25 per cent.

Tensile tests were also performed on a series of samples from each moulding. Again the advantages of using pressure were highlighted. Bubbles in the tensile samples cause weaknesses in the polymer structure, through which cracks form. These weaknesses can be easily seen during testing and are illustrated at $\times 10$ magnification in Fig. 12. During the tensile test, the

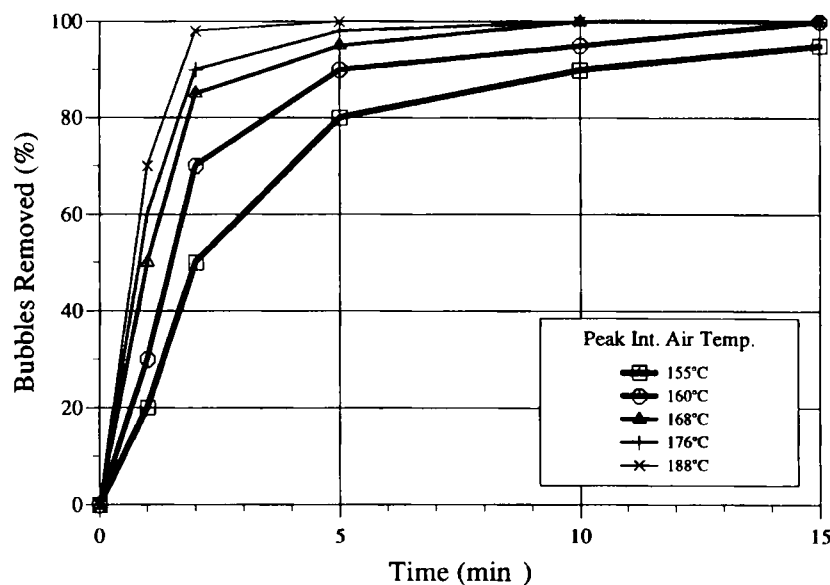


Fig. 10 Effect of 0.5 bar on bubble removal

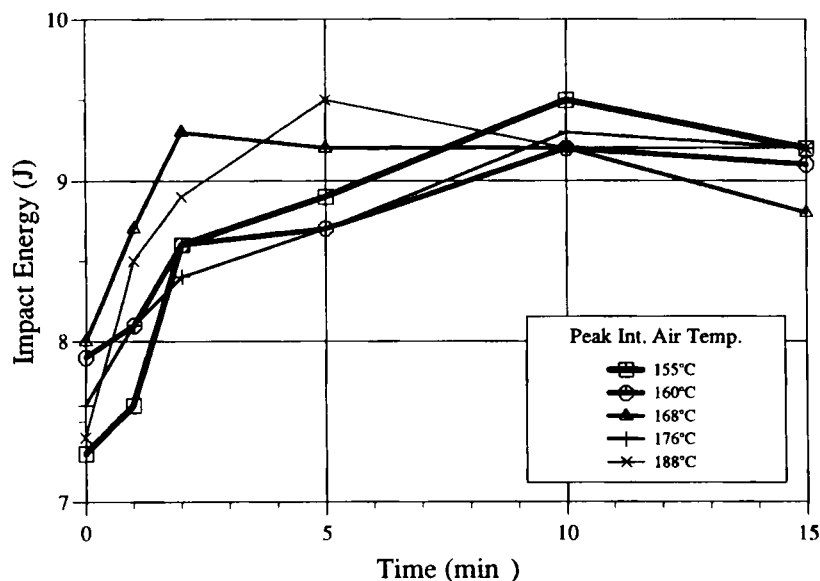


Fig. 11 Effect of 0.5 bar on impact energy

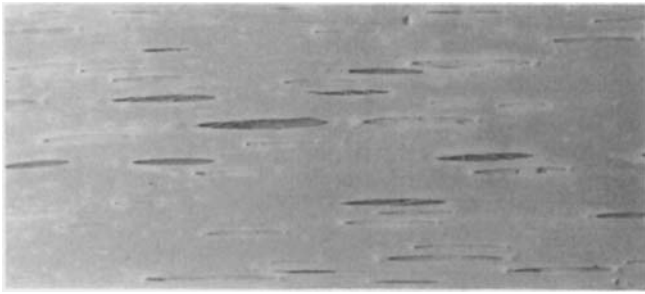


Fig. 12 Tensile sample with bubbles (magnification $\times 10$)



Fig. 13 Tensile sample without bubbles (magnification $\times 10$)

bubbles elongate, increasing in size and eventually they cause the sample to break. If pressure is used to remove bubbles, as is the case for Fig. 13, then the sample has no points of weakness. The tensile data illustrated in Fig. 14 indicate that the removal of bubbles increases both the tensile strength by approximately 5 per cent and the elongation, before the sample breaks.

From the mechanical test results it can be seen that, with the application of pressure, the mould can be removed from the oven at a lower temperature with no adverse effect on the properties of the part. Indeed, part properties increase, as the weaknesses incurred due to bubbles and pores have been removed. Removing the mould from the oven at a lower temperature has an added advantage in that polymer will be less degraded, as it will be subjected to a higher temperature for a shorter period of time.

4.2.2 Effect of pressure level on mechanical properties

The effect of the applied pressure level on the mechanical properties of the product was investigated using NCPE 8017 (natural polyethylene, MFI 3.2 g/10 min). During this series of tests, the polymer was heated to 190°C and then removed from the oven. As it was being removed from the oven, pressure levels ranging from 0 to 3 bar were applied for 10 minutes and then relieved. Following rotational moulding, some mechanical properties were investigated.

Impact tests (at -20°C) again highlighted the advantage of using pressure to remove bubbles (see Table 2). From the impact tests, the three applied pressure levels all removed bubbles and pores completely, and hence increased the parts impact strength. However, there was no noticeable increase in impact strength experienced for each increasing pressure level.

Tensile tests, unlike impact tests, were affected by the applied pressure level (see Table 3). As the applied pressure level increased, the tensile strength and tensile modulus both increased. It is suggested that the tensile properties improved because increasing the pressure level consolidates the polymer structure and improves its resistance to the externally applied forces.

4.2.3 Effect of pressure on part shrinkage

The effect of pressure on part shrinkage was studied in conjunction with NCPE 8113 (sky blue polyethylene, MFI 3.2 g/10 min). During this series of tests, shot sizes of 1 and 2 kg were used to produce mouldings that were 3 and 6 mm thick. Each moulding was heated to 190°C (internal air temperature) and then removed from the oven, during which the desired pressure level was applied. The pressure was maintained until the internal air temperature of the part reached 80°C, when it was released and the part demoulded. Part shrinkage was measured by monitoring a 200 mm square grid, which had been moulded into the part. The grid was first measured with the part still hot (50°C). Shrinkage was then recorded after 1, 5 and 30 days (see Table 4), with each mould being stored in a temperature-controlled environment at 20°C.

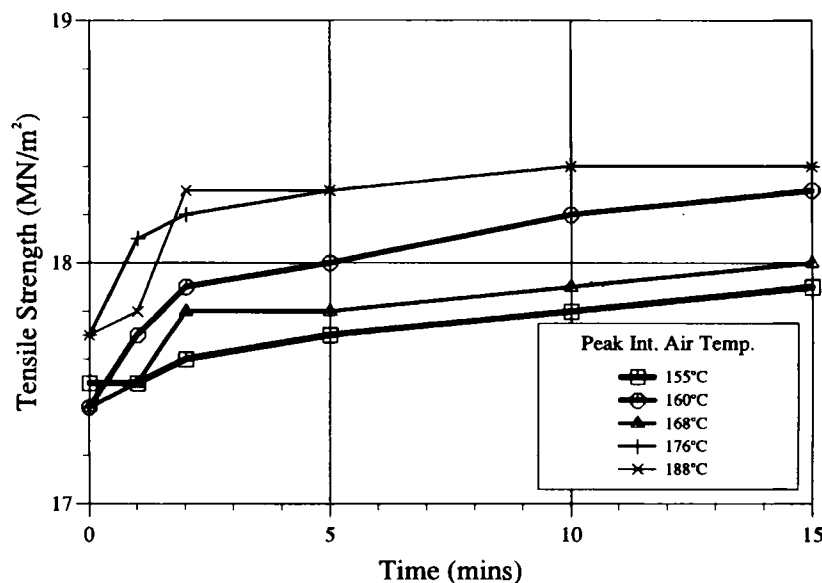


Fig. 14 Effect of 0.5 bar on tensile strength

Table 2 Impact data (-20°C) for increasing pressure levels

Applied pressure	Thickness (mm)	Force (kN)	Energy (J)	Energy/mm (J/mm)	Mode of deformation
0	2.83	1.74	8.16	2.88	Ductile
1 bar for 10 min	2.76	1.86	9.70	3.51	Ductile
2 bar for 10 min	2.75	1.81	9.24	3.36	Ductile
3 bar for 10 min	2.70	1.76	9.42	3.49	Ductile

Table 3 Tensile data for increasing pressure levels

Applied pressure	Tensile strength (MN/m^2)	2% secant modulus (GN/m^2)	Elongation at break (%)
0	17.53	0.323	200–500
1 bar for 20 min	18.05	0.327	1000+
2 bar for 20 min	18.17	0.333	1000+
3 bar for 20 min	18.76	0.347	1000+

Table 4 Effect of pressure on shrinkage

Material thickness (mm)	Pressure	Shrinkage (%)			
		50 $^{\circ}\text{C}$	1 day	5 days	30 days
3	0	2.55	3.16	3.18	3.18
3	1 bar for 20 min	1.55	2.09	2.13	2.14
3	2 bar for 30 min	1.40	2.09	2.10	2.11
6	0	1.87	2.75	2.75	2.64
6	1 bar for 20 min	1.42	2.21	2.30	2.25
6	2 bar for 30 min	1.50	2.38	2.38	2.30

Table 5 Effect of pressure on material density

Material	MFI	Manufacturer's density value (kg/m^3)	Density with bubbles (kg/m^3)	Density without bubbles (kg/m^3)
NCPE 8113	3.2	934	917	933
DuPont 8107	5.0	925	918	923

From the shrinkage data, it can be seen that mould pressurization reduces shrinkage by as much as 1 per cent. Pressure forces the moulding against the side of the mould during cooling. This restrains the contractional forces inside the polymer structure as it cools and solidifies. The data indicate that the majority of shrinkage occurs during the first 24 hours after moulding.

4.2.4 Effect of pressure on material density

Pressure has been shown throughout this section to remove bubbles and pores from the moulded part. However, it is not clearly understood whether or not the air molecules contained in the bubbles are forced out of the polymer or if they are dispersed within the polymer in minute quantities which are undetectable to the human eye. Two materials, NCPE 8113 (sky blue polyethylene, MFI 3.2 g/10 min) and Du Pont 8107 (black polyethylene, MFI 5 g/10 min), were used to investigate this area by considering how the densities of the moulded parts vary when pressure is used to remove bubbles.

During these tests, each material was first rotationally moulded in the normal fashion, and then rotationally moulded using pressure to remove bubbles and pores. Sections from each moulding were then accurately machined to set dimensions and weighed. From the dimensions and mass of each section the density was

calculated (see Table 5). The density calculations confirmed that the air inside the bubbles and pores is forced out of the polymer when pressure is applied. This is apparent from the data, because of the rise in material density when bubbles were removed.

4.2.5 Effect of pressure on thick-walled mouldings

The effectiveness of pressure to remove bubbles and pores from thick-walled polyethylene moulding was investigated using NCPE 8017 (natural polyethylene, MFI 3.2 g/10 min). During these trials, shot sizes of 2 and 3 kg were used to produce parts that were 6 and 9 mm thick. Mould pressurization (0.5 bar) was again shown to effectively remove bubbles and pores. Therefore, it was concluded that the action of mould pressurization to remove bubbles was independent of part wall thickness, at least for thicknesses in the range of 1–9 mm, and there is no reason to suspect that it will not also work for greater wall thicknesses.

4.3 Pressurized rotational moulding of other materials

The application of pressure to the polymer melt was successfully used to remove bubbles and pores from all polyethylene materials used during this research. However, it was not known how effective pressure would be on other rotational moulding materials. During this series of tests, five other rotational mould-

Table 6 Moulding conditions for rotational moulding trials

Material	Product code	Oven temperature (°C)	Oven removal/onset at pressure (°C)
Ethylene vinyl acetate	1080 VNS	300	150
Ethylene butyl acetate	NCPE 8019	300	150
Polypropylene	PPFC/RM	300	195
Polycarbonate	Markolon 2400	350	280
Nylon 12	Rilsan MA 3950	350	240

ing materials were considered: polypropylene, Nylon 12, polycarbonate, ethylene vinyl acetate (EVA) and ethylene butyl acetate (EBA). The individuality of the materials meant that each material required its own specific moulding conditions. Table 6 outlines the moulding conditions used during these trials. During each pressure trial, 0.5 bar was applied during the removal of the mould from the oven and was maintained until the part was removed from the mould.

From the rotational moulding trials it was found that pressure could successfully remove bubbles and pores from the other rotational moulding materials. However, it was found that EBA and EVA required a higher pressure (1 bar) before bubbles and pores were completely removed. This relates to the fact that the EBA and EVA materials used during this work were processed at lower temperatures than the polyethylene materials used.

Impact tests were performed on all of the materials except polycarbonate, which was omitted because of variable wall thicknesses. Impact tests showed that the application of pressure has less of an effect on these materials than on polyethylene. However, it should be noted that the aesthetic properties of some of these materials are just as important as their mechanical properties, for example polycarbonate, which is used in applications where its transparency is critical. For materials like Nylon, which is used to manufacture chemical storage tanks, the removal of voids and pores reduces the risk of possible leakage. In general, the use of pressure to remove bubbles improves the overall quality of the product, which can only be good for the industry.

4.4 Effect of pressure on the mould

It should be noted that the use of pressure has certain limitations. Mould pressurization is extremely dangerous, if care is not taken. It is recommended that pressure should only be used for small moulds (1 m³ or less) that are sufficiently strong to withstand the forces involved. This method for removing bubbles is not suitable for large moulds fabricated from sheet steel, as moderate pressures will cause distortion or even mould fracture.

In general, the use of pressure is not suited to the majority of moulds used in the industry today. This is because their clamping is not sufficiently good to withstand the internal forces caused by pressure. When manufacturing a new mould specifically for use with pressure, the following suggestions are made:

1. Aluminium moulds need to be three times thicker than mild steel moulds to provide equal strength.
2. Flat sections on moulds should be avoided. The design of the mould should incorporate strengthening features where possible.

3. When using pressure, the quality of the moulding is dependent on the surface finish of the mould, as pressure provides better part definition.
4. Pressure will tend to force the molten polymer into the parting line. Therefore, the mould parting line should be machined, if possible, and contain an 'O' ring.
5. The mould, or the pressure connection on the mould, should incorporate a pressure relief valve.

4.5 Automation of mould pressurization

The application of pressure to a mould is reasonably straightforward through the arm of the machine. However, the manual control of the pressure line used during these tests would not be suitable for the rotational moulding industry. Therefore, a portable pressure control box was designed and manufactured to automate the mould pressurization process. During the design of the control box, two principal areas were considered: the pressure line to the mould and the control of the application and release of pressure.

4.5.1 Pressure line

The pressure line was designed to provide the utmost control of the pressure to the mould. The airline pressure entering from the back of the control box passes through a regulator fixed at 2.5 bar. The pressure line can then be regulated to the required pressure level using the second (variable) regulator. At this stage the application of pressure to the mould is controlled by a two-position, three-way, solenoid-operated, spring return valve. The valve allows the mould to vent normally, but when the solenoid is operated, the mould is pressurized. The valve has the inherent safety feature in that if the power fails, the spring automatically returns the valve to its mould venting configuration. The pressure gauge position after the solenoid valve indicates the pressure rise in the mould when pressurization begins. Also incorporated in the pressure line are two pressure relief valves: one in the control box and one at the mould/pressure connector. These pressure relief valves are necessary safety features to ensure that the mould will not be overpressurized.

4.5.2 Electronic control

The application and release of pressure to and from the mould was electronically controlled. A four-position switch, which was normally in the 'off' position, controls the pressure line by three methods:

1. *Manual.* The manual position is provided so that when the switch is closed, the solenoid operates, opening the valve, and continues to operate until the switch is re-opened.

2. *Timed.* The timed position incorporates two timer units. The first timer unit sets the delay before pressure is applied. The second timer sets the duration of the pressure application. When the switch is closed on the timed position, the countdown begins.
3. *Computer control.* A computer-controlled position was supplied for use in conjunction with the Rotolog diagnostic software to provide complete automatic control of the system. The computer-controlled position allowed pressure to be applied to the mould and relieved from the mould at a specific melt temperature corresponding to the Rotolog curve. The control system was triggered using a designated function key on the computer keyboard.

4.6 The use of a vacuum to remove bubbles

It has been shown throughout this section that pressurization of the polymer after it has melted successfully removes bubbles. Therefore, it was postulated that perhaps a vacuum could also be used to provide a difference in pressure between the bubble and the surrounding melt, and so aid bubble removal. During these tests the rig was charged with NCPE 8017 (natural polyethylene, MFI 3.2 g/10 min) and sealed. A vacuum was then applied to the atmosphere contained within the rig. The rig was then placed on the hot plate (set at 300 °C) with the vacuum still being applied.

From the results it was observed that with a vacuum applied the number of voids that formed were significantly reduced. However, the bubbles that did form increased in size as the polymer temperature increased. This was because the air pressure inside the bubble was rising due to the surrounding melt temperature, and so the bubble was able to expand. At 180 °C, the vacuum was removed and atmospheric air was introduced to the rig (see Fig. 15). At this point bubbles contained within the melt disappeared instantaneously. The introduction of atmospheric pressure caused a pressure difference between the pressure inside the bubble and the pressure of the surrounding melt. This pressure difference caused the void to collapse.

These observations confirmed that a vacuum could be used successfully to remove bubbles contained within the melt by creating a pressure difference between the air inside the bubble and the pressure exerted by the surrounding melt. This method to remove bubbles was confirmed by rotational moulding trials. With the vacuum applied during the heating cycle and removed when the polymer powder had completely melted, mouldings were produced without bubbles or surface pores.

However, it should be noted that not all moulds would lend themselves to the application of a vacuum. To facilitate this, moulds would require an 'O' ring for sealing purposes; otherwise suction holes would be created in the parting line. Also, moulds would need to be strong enough to withstand the forces caused by the applied vacuum.

5 CONCLUSIONS

As a result of this work on the rotational moulding of thermoplastics, the following conclusions may be drawn:

1. Mould pressurization successfully removes bubbles from the melt by creating a pressure difference between the inside and outside of the bubble. By first allowing the polymer to melt and the bubbles to form in the normal fashion, a pressure of 0.5 bar then introduced to the mould removes bubbles successfully from polyethylene. The use of pressure was shown to successfully remove bubbles from mouldings up to 9 mm thick.
2. By removing weaknesses in the moulding due to bubbles and pores, the use of pressure was shown to increase the mechanical strength of the moulded product. Impact strength was shown to rise by as much as 25 per cent and tensile strength increased by 5 per cent. Another advantage pressure gives to the rotational moulder is the possibility of reduced cycle times, as pressure can consolidate the melt and remove bubbles at lower temperatures (140–180 °C).
3. The use of pressure was also shown to have additional beneficial effects on the moulded product. For

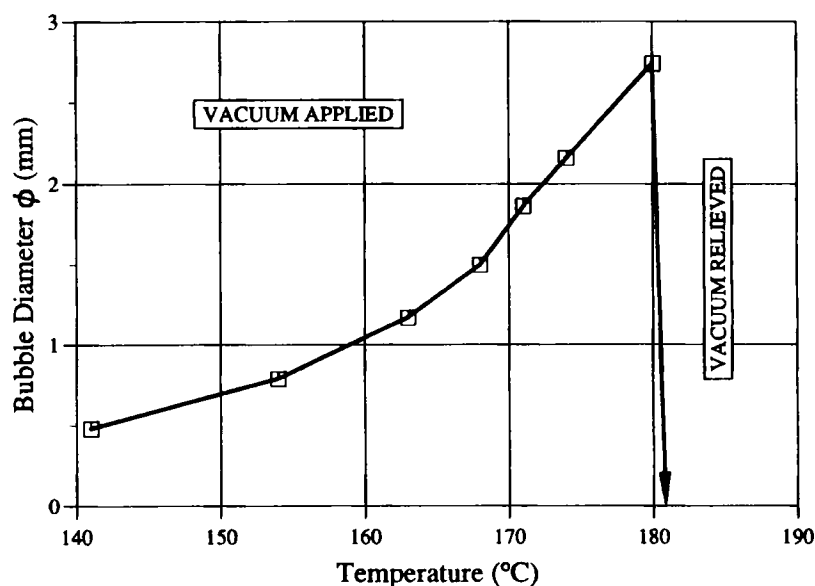


Fig. 15 Effect of a vacuum on bubble removal

example, the shrinkage was reduced when pressure was used. The density of the material in the wall of the moulding was also increased when pressure was used to remove bubbles.

4. Mould pressurization improves the aesthetic quality of the rotationally moulded product by removing surface pinholes and also providing better part definition.
5. Pressure can also be used to increase the wall thickness of rotationally moulded products. By introducing pressure before the polymer has melted and relieving it after the polymer has melted, atmosphere inside the bubble contains pressurized air when formed, which is free to expand when the applied pressure is relieved.
6. The use of pressure to remove bubbles was also shown to be successful for a number of rotationally mouldable materials other than polyethylene. Bubbles have been removed from materials such as polypropylene, Nylon 12, polycarbonate, ethylene vinyl acetate and ethylene butyl acetate using pressure.
7. The application of a vacuum before the polymer has melted and relieved after the polymer has melted was shown to instantaneously remove bubbles contained in the polymer melt. The removal of the bubbles was a result of the pressure difference between the inside of the bubble and the surrounding polymer. This was caused when the vacuum was relieved and atmospheric pressure introduced.

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