Innovative PE100 material improves the quality and productivity of injection moulded fittings

James McGoldrick, Mats Bäckman, David Walton, Susanne Nestelberger.

ABSTRACT

Polyethylene has been in use for the manufacture of piping systems since the sixties and has progressed in its property spectrum not only in terms of resistance to pressure, but also both in terms of resistance to Rapid Crack Propagation and to Slow Crack Growth. In terms of processing friendliness however, further progress has been achieved only in recent years in the form of PE100 materials which exhibit "Low Sag" properties thus allowing the manufacture of very large thick-walled pipes having very constant wall thickness around the whole circumference of the pipe. This paper describes a ground-breaking material technology which facilitates easy manufacture of injection moulded PE100 fittings whilst still fulfilling all the requirements of international standards and outlines also the benefits which this technology can bring to a manufacturer of pipe fittings.

INTRODUCTION

Times have changed - in the past plastics were produced to a specific formulation, and product manufacturers and end-users were faced with selecting the best material to meet their needs. Inevitably this was a compromise since processing materials using the extrusion- or injection moulding processes make strongly contrasting demands on the processing characteristics of the material. However some of today's most modern polymerisation technologies now make it possible to customize materials in order to meet both specific manufacturing- and end-user requirements. Indeed both customer satisfaction and application needs have become a real driving force in the development of new polyethylene raw materials over the last few years.
For many years standard bimodal PE100 pipe extrusion grades have been used also for the production of injection moulded pressure pipe fittings. Since these materials exhibit high viscosities which are desirable for the extrusion process, and a molecular weight distribution which has been optimised for pipe extrusion, there have been some clear disadvantages when using such materials for the manufacture of injection moulded fittings. Such disadvantages have been long cycle times, surface distortion, sink marks, etc. Up till now, no material that has satisfied both the demanding requirements of the technical property spectrum which is needed in a PE100, while simultaneously exhibiting the high flow properties needed for the avoidance of problems in the injection moulding process. However utilisation of the flexibility of the Borstar process has enabled the fulfilment of both the needs for mechanical excellence and processing friendliness in injection moulding.

THE POLYMERISATION PROCESS

In the pipe business the flexibility of the bimodal (or multimodal) process for producing polyethylene materials has provided the greatest scope for producing "tailor-made" materials. The choice of catalyst, comonomer type-, content-, and selective distribution thereof in the polymer chains, and the selection of process parameters in each reactor all effect the development of the polymer structure and the properties of the end product. Changing these variables enable the properties to be optimised for a manufacturing process or end use application.

The bimodal process consists of two polymerisation reactors in series. Figure 1 shows a simplified view of the basic principles of the bimodal process. The Borstar® low pressure slurry loop and gas phase reactor process is illustrated. The catalyst is fed into the first reactor, where polymer is formed as powder particles through polymerisation of the ethylene monomer and suitable amounts of comonomer, continuing in a series mode into the second reactor.

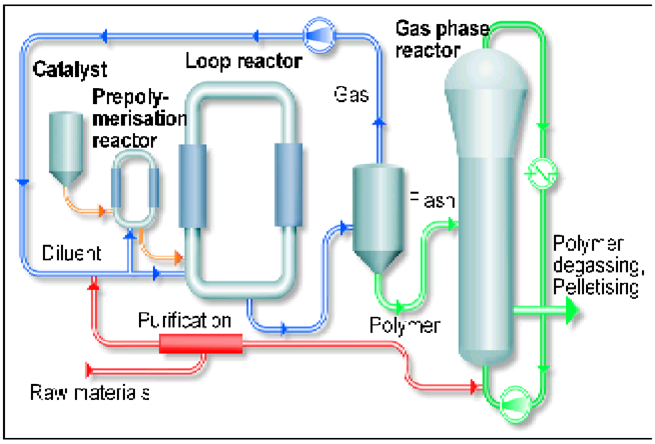

Fig. 1 : Schematic diagram of the Borstar polymerisation process

The principal advantages of the process are:

- Independent control is applied to the reactors to steer comonomer distribution and to tailor molecular weight distribution (MWD).
- Flashing between the reactors guarantees independent reaction mixture compositions. A wide density range, from LLDPE to HDPE can be produced.
- Different comonomers can be incorporated according to needs, e.g. butene or hexene.
- The MFR_2 of the different reactors can be varied across a very broad range, from <<0.1 to more than 1000 g/10min.
- The process offers great flexibility with regard to the type of comonomer which can be incorporated into the correct regions of the polymer. For example, the use of the comonomer hexene in the bimodal Borstar process results in polymers having extremely high resistance to Slow Crack Growth.

DESIGNING A BIMODAL PE100 MATERIAL FOR INJECTION MOULDING

Bimodal materials were initially introduced for higher strength PE100 pipe extrusion materials which were then also used for injection moulded fittings with many of the consequences mentioned above. Subsequently “low sag” PE100 materials were developed which enabled 2 metre (80 inches) diameter pipes to be produced with 120mm (4.8 inches) wall thickness.
The introduction of hexene as a co-monomer in the Borstar process provides further design scope enabling very high stress crack resistance materials to be produced whilst maintaining high strength and stiffness. Conversely it is also possible to significantly improve the flow properties of the material whilst still maintaining all the strength of a PE100 material – *which are exactly the characteristics required for the injection moulding of pressure fittings.*

Extrusion grades tend to be designed with a broad molecular weight distribution and a high molecular weight tail but these characteristics are far from ideal for the injection moulding process. Indeed, the surface distortion and shrinkage is linked to the long relaxation times for the high molecular weight part of the material. High stresses and deformations that do not have time to relax due to the rapid cooling of the surface of the fitting result in high shrinkage and warpage of the component. In order to reduce this effect, it has been necessary to use high processing temperatures, which has led to increased cycle times and as a consequence thereof, to reduced productivity for the fittings manufacturer.

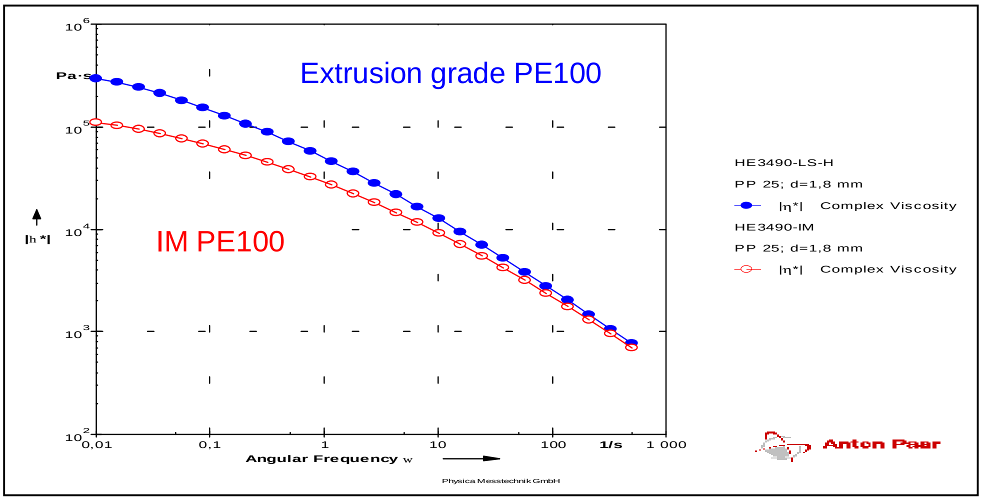

Fig. 2: Rheology curve of extrusion grade and PE100 for the injection moulding process

The high molecular weight material can easily be detected in a rheology diagram as it will show up as a high low shear viscosity as shown in the rheology comparison above. As can be seen, there is a big difference in the zero shear viscosity between the pipe extrusion grade and the injection moulding grade, which stem principally from the changes in broadness and in the high molecular weight tail. However as conformity to the relevant industry standards for fittings made of PE100 material must be retained, the molecular weight distribution has to be correspondingly optimised. The resulting molecular weight distribution is shown in the GPC comparison of figure 2. An important requirement is to balance the high and low molecular weight material so that both excellent flow characteristics can be achieved whilst retaining the performance of a PE100 material in terms of hydrostatic pressure rating (MRS 10) as well as resistance to Slow Crack Growth and resistance to Rapid Crack Propagation.

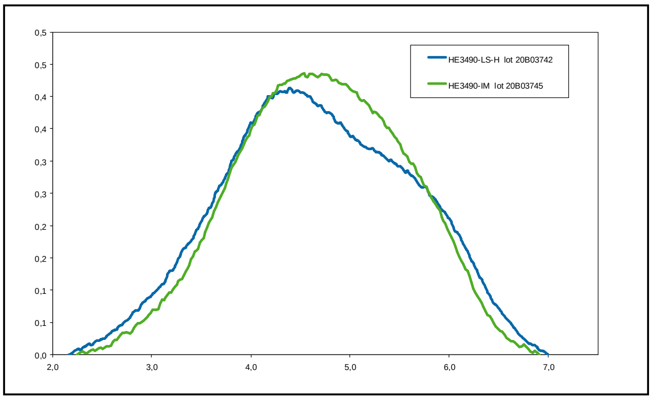

Figure 3. GPC comparison between IM-PE100 and a PE100 extrusion grade

The flexibility of the Borstar process, especially utilising hexene comonomer, makes it possible to fine-tune the balance of low and high molecular weight material in a very precise manner. The use of hexene comonomer is important as this provides greater scope for modifications by improving mechanical properties like slow crack growth and impact. Similar technology will open up the possibility for more modified- and specialised bimodal PE100 materials in the near future as this in combination with the flexibility of the Borstar process opens up new and better routes for material design, especially in the field of PE100.

As shown in table 1 below, all the mechanical properties meet the PE100 requirements whilst a major improvement of the flow properties, and therefore mouldability, of the material has been achieved. Therefore the material provides a better balance in properties for the production of injection moulded PE100 fittings since on the one side, the mechanical demands which are made on the material are fulfilled, and on the other side the easy-flow properties which are essential to the avoidance of processing problems in the injection moulding process have been incorporated into this special grade. Indeed, whilst the hydrostatic pressure rating has been retained at MRS 10 and the resistance to RCP is unchanged, the resistance to SCG has been vastly improved despite a reduction in the mean molecular weight of the material Mw 280000 (Mw/Mn = 23) in comparison to 350000 (Mw/Mn = 38).

Table 1 Key mechanical properties of Borstar HE3490-IM

Test requirement	Method	Value	Requirement
Pressure resistance 5,5 MPa / 80°C (176°F)	ISO1167	3972 h	5,4 MPa >165 h (EN)
Notched Pipe Test 4,6 MPa / 80°C (176°F)	ISO 13479	1880 h	>165 h (EN) > 500 h (PE100+)
PENT	ASTM F1473	2500 h	>100 hours
FNCT	ISO 16770	5500 h	3300 h (DVGW/gas) (highest requirement)
Resistance to RCP , S4 160mm SDR 11 Pc	ISO 13477	>11 bar	$PcS4 > \frac{1.5 \times MOP - 2.6}{3.6}$ (EN 1555 requirement)
Resistance to RCP, S4 110 mm SDR 11 Tc	ISO 13477	-7°C (19°F)	No EN requirement as Tc
Pressure res. T-Piece 180 mm SDR 11 Tc	ISO 1167	>2100 h	5,4 MPa >165 h

INJECTION MOULDING OF PRESSURE FITTINGS

The injection moulding machine consists in essence of a mould locking part and of the injection part.

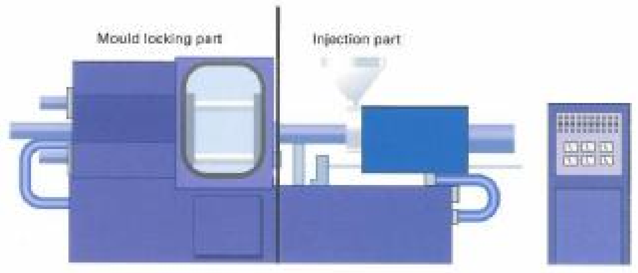

Figure 1. Injection moulding machine

The process of injection moulding is a discontinuous process consisting of three distinct phases, namely injection, packing and cooling phases. In other ways, the process has little in common with the extrusion process especially with regard to the forming stage of the process. While the calibration stage of the pipe extrusion process takes place in a vacuum tank, or in some cases under internal pressure, the forming stage of a pipe fitting in the injection moulding process takes place in a closed cavity into which the prepared melt is injected at a comparatively high speed.

In order to better understand what is happening to the material in the process and in particularly how this effects issues such as shrinkage it is necessary to study the Pressure – Volume – Temperature (pvT) curves for the material as shown in figure 5 below.

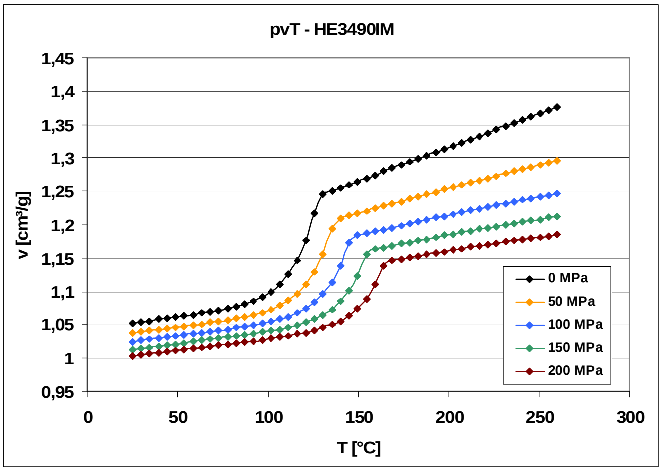

Fig. 5 Pressure-Volume-Temperature diagram for Borstar HE3490-IM

The curves show the relationship between the volume occupied by the material at different temperatures and at a constant pressure in each case. The knee in the curve is the crystallization temperature and this temperature increases as the pressure is increased.
As the material cools inside the mould the volume occupied by the material will decrease. The degree of shrinkage can be calculated from the above figure using a hypothetical example. If the mould cavity has a volume of 1000 cm^3 and the pressure during the cooling process is 100 MPa then the specific volume of the material at 220°C will be 1.230 cm^3/g (equivalent to a density of 813 g/l).This means that the mould will contain 813 g of material under these conditions. At ejection, the temperature will be approximately 100°C and the volume would contract to 854 cm^3, and at 23°C the volume would be 837 cm^3. This means that if no material is pressed into the cavity during the packing phase, then the material used to manufacture the fitting would undergo a volumetric shrinkage of approximately 16% when cooling down from 220°C to room temperature. Such excessive shrinkage would be extremely difficult to deal with during daily manufacturing conditions.
This theoretically high level of shrinkage can be reduced to manageable levels by continuing to press material into the mould during the packing phase, that is, whilst the material in the tooling cools. This material will compensate for the reduction in volume provided of course that the injection gate remains “open” (in the fluid state) during this part of the process. Control of this packing phase is essential to produce good quality products as the packing phase

- has the greatest influence on the shrinkage level.
- can be used to avoid sink marks on the surface of the fitting
- can be used to avoid the appearance of voids in thick sections of fittings

However, the packing phase cannot solve all problems. Indeed if the pressure level used during this phase is excessively high, it can be detrimental to product quality and in particular to the resistance to slow crack growth due to the development of excessively high stresses during the packing phase, and thus in the fitting. This danger can be reduced by employing lower packing pressures that are possible with materials that have easier flowing properties. This results in fittings with lower frozen-in stresses and which in turn exhibit excellent stress-crack resistance. Thus both the concept of the material and the processing parameters both contribute intrinsically to the capability of the fitting to resist against Slow Crack Growth.

As a general rule packing pressure level should be around two thirds of the injection pressure level and it should be reduced slowly (preferably in profile) prior to the ejection of the fitting. Gating design is also critical and significant benefits can be achieved using hot runner systems.

Simulation of the injection moulding process

The use of Computer Aided Engineering and Design (CAED) as a tool to visualise the injection moulding process has grown rapidly in recent years. It can be used to design the injection moulding tool in terms of its construction without costly trial and error process, and to predict a great variety of critical issues such as the placement of the point(s) of injection and the resulting positioning of weld lines. In addition, shrinkage and warpage levels, for example the level of ovality of the fitting spigots, and many other important dimensional characteristics can be predicted.

Also, CAED can be used to simulate the injection moulding process as a whole and thereby optimise the important parameters. For example it is possible to study the influence of the injection temperature on cycle times and thereby the effect on product costs. Practical tests have shown that whilst standard PE100 extrusion grades are usually injected at melt temperatures of 240°C it is possible to inject this injection-moulding PE100 (IM-PE100) at 200 - 210°C. CAED calculations show that this difference in the injection temperature allows the cycle time to be reduced by around 10% and this has been confirmed practically in industrial trials.

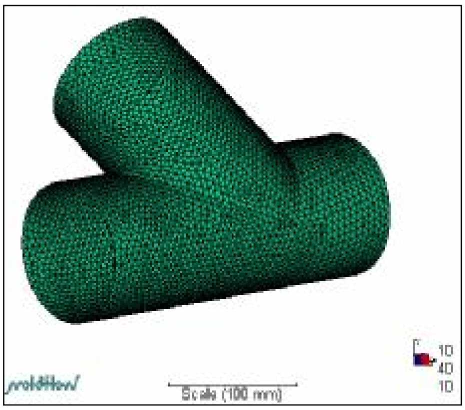

Fig. 6 Transforming product design to CAED coordinates

High molecular weight PE materials exhibit anisotropic shrinkage characteristics, that is, they shrink more in the flow direction than they do in the transverse direction. This occurs because the long molecules orient themselves in the flow direction in the same way as wooden logs do when they are transported on a river. This will be most evident at the end of the spigot where the melt flow in reaching the end is deflected at 90° to the flow. This means that a certain amount of anisotropic shrinkage is inevitable which will lead to ovality. A flow simulation using CAED is shown below in figure 7 below.

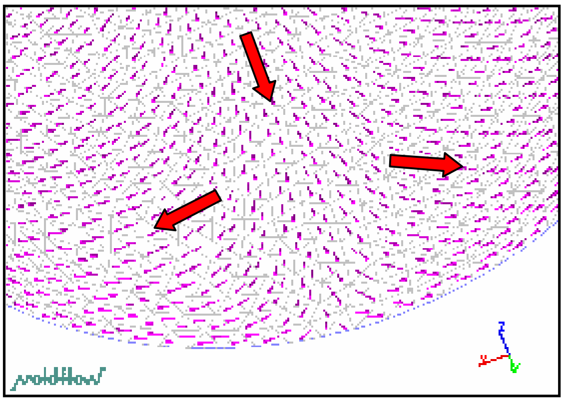

Fig. 7 Flow simulation showing orientation effects using CAED software

Since the anisotropy is primarily due to the orientation of the high molecular weight material, the reduced molecular weight of this IM-material means that the level of orientation and therefore anisotropic shrinkage will also be lower. This means that fittings produced using IM-PE100 will exhibit less ovality than those produced using PE100 extrusion materials. This ovality can be even further reduced using the lower injection temperatures and packing pressures which can be implemented by taking advantage of the property package which this new and innovative material offers.

SOME PRACTICAL CASES THAT DEMONSTRATE THE ACHIEVABLE BENEFITS AFFORDED BY THIS MATERIAL CONCEPT

Production trials conducted by manufacturers of fittings have not only confirmed the expected performance of this material but indeed have surpassed all expectations placed in the whole concept. Here are some examples :

Surface quality

The first improvement which becomes evident is the one in the surface quality of the fittings especially in the case of smaller and more intricate fittings. The surface of fittings made of this new material is smooth and shiny in comparison to fittings produced using PE100 extrusion materials - similar to the appearance of PE80 fittings.

Critical areas in fittings

In most fittings there is a critical area caused by the design of the fitting and/or by the way it is manufactured. Such fittings produced using extrusion PE100 materials are sometimes inferior to the rest of the fitting in mechanical strength. This is due to local turbulence and consequently to the formation of micro flow-fronts caused by the lack of flowability of PE100 extrusion materials. Due to the easy-flow properties of this new IM PE100 material, it has been shown that problems which manufacturers of fittings have been struggling with for years disappear when a purpose-designed material concept is used.

Weld lines

The positioning of weld lines has become less critical through the emergence of this material.
This is also due to the easy flow characteristics of this material which facilitate good bonding between merging flow fronts. This effectively improves the strength of the fitting itself.
Also, the speed at which the melt is injected into the mould also plays an important role here. If it is too high, the effect will be high orientation of the melt and thus high anisotropic shrinkage. On the other hand, if it is too low, cooling of the melt front will be the result and poor weld strength will become apparent in the fitting.

Less rejects

Due to the general easy processing characteristics of this new material, significant reductions in the quantity of rejects have been recorded during industrial trials.

Better quality and lower scrap rates for electrofusion fittings

Increased quality and significantly lower scrap rates have also been recorded in industrial trials using Borstar HE3490-IM (IM-PE100). These factors are again achieved due to the excellent flow characteristics which is responsible for good contact to the wires in such fittings. Also, improved flow characteristics mean that the material follows more closely the profile in the tool. This means for a small diameter electrofusion fitting better profiling of the grooves which in turn gives better lock-in of the electrofusion wires.

High productivity due to low cycle times
This can be achieved due to the lower injection temperatures with which IM-PE100 can be moulded in comparison to PE100 extrusion materials. The achievable advantage here becomes more apparent with increasing wall thickness of the fitting.

The commercial value of a tailor-made injection moulding material

On the basis of data gathered from industrial injection moulding manufacturing, and using a Cost Simulation programme (CostSim, which is accessible via www.borealisgroup.com/borsim) an attempt was made to quantify the commercial value of reducing the cycle time.
Since it has been shown both virtually on a CAED programme as well as in practice that cycle time reductions in excess of 10% are achievable when using Borstar HE3490-IM instead of an extrusion PE100 material, calculations were made on this basis. Since a reduction in cycle time has an effect on manufacturing costs, machinery costs and labour costs, the initial saving in cycle time of 10% increases to 15,5 % using this cost calculation programme in the example shown as illustrated in figure 8 below . Since the price differential between traditional PE100 extrusion material and this new and processing-friendly grade is small in relation to the achievable increase in productivity this material has been very well received on the market.
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Figure 8 : Estimation of total savings due to cycle time reduction

Summary and conclusions

Since the introduction of PE100 at the end of the 1980's, some progress has been made in making these materials more processing friendly. The first was the development of "Low Sag" (LS) materials. This made the production of extruded pipes, having large diameter and large wall thicknesses feasible, because the hot material in the pipe wall no longer moves to the bottom of the pipe during the cooling stage, and thus very large pipes having very regular dimensions can be produced. The quest for a specialised PE100 injection moulding material has shown itself to be more difficult since easy processing in injection moulding means having a material which exhibits good flow properties and this in turn means lower molecular weight. Thanks to the flexibility of the Borstar process and the use of hexene co monomer a true PE100 injection moulding material has now been developed. In process trials this material offers a number of benefits to the pressure fittings manufacturer including shorter cycle times and lower costs.

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