ADVANCES IN HDPE TECHNOLOGY FOR LARGE DIAMETER PE100 PIPE APPLICATIONS

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Abstract

Although bimodal HDPE has been well-established in PE100 pipe applications since the 1990's, continued advances in catalyst and polymerization process technology in recent years have led to further improvements in mechanical properties and extrusion processability. Univation Technologies has commercialized the PRODIGY™ BMC-200 Catalyst System to produce a bimodal PE100 material in a single gas-phase reactor. This new bimodal HDPE material exhibits an excellent balance of mechanical properties along with a unique rheology which delivers advantages in the pipe extrusion process. The molecular weight distribution resulting from this bimodal catalyst system yields processing advantages that include high output, low melt temperature, and high melt strength. The high melt strength of the extrudate provides improved wall thickness distribution, which facilitates the production of large diameter pipes. In addition to mechanical properties and extrusion processability, the quality of joints of pipes and fittings is a critical element of pipe performance and/or integrity. Electrofusion and butt fusion trials were conducted in order to successfully validate compatibility of large diameter pipes produced with the BMC-200 PE100 with other PE100 materials and with commercially-available fittings.

Introduction

PE pipes have been in use for more than 50 years, delivering excellent performance. Today polyethylene is a well-established material for gas and water supply systems, waste water disposal and sewage systems, as well as numerous industrial applications such as transport of slurries in mining and for coal seam gas. PE piping systems, also in larger nominal diameters, have become a common preference in tenders and specifications by civil engineering firms for large scale irrigation projects.

Traditionally, these bimodal polyethylenes have been produced only in multiple reactor configurations (Figure 1). These staged-reactor systems produce excellent products for applications including film, blow molding and PE100 pipe. However, the process is more complicated and requires more capital than a single reactor system.

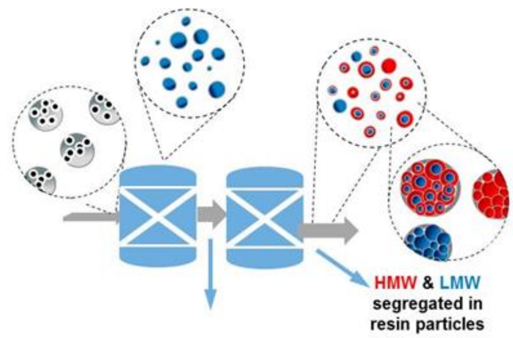

Figure 1. Staged-Reactor Bimodal HDPE Polymerization Process

Recently, Univation Technologies has developed a two component catalyst system that produces a bimodal molecular weight distribution with a desirable comonomer distribution (Figure 2). The two catalyst components produce different, controlled, polymer chains under specific reaction conditions. The first catalyst component (component A) produces high molecular weight polymer chains. Catalyst component B produces low molecular weight polymer chains. Component A has a high affinity for comonomer which is essential for formation of tie molecules and promoting excellent slow crack growth resistance. Component B also incorporates some comonomer, but less than that of component A.

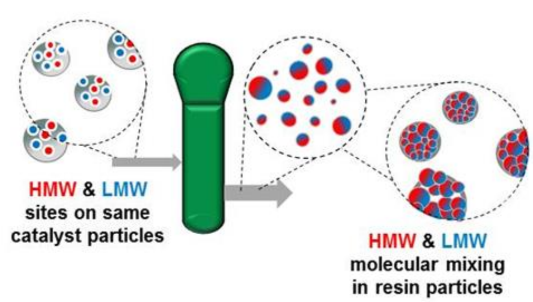

Figure 2. Single-Reactor PRODIGY BMC-200 Polymerization Process

Polyethylene products for various applications are produced by varying the split (ratio of HMW/LMW polymer components), spread (ratio of the molecular weights of the components), the overall molecular weight, and the comonomer content. These polymer attributes are controlled by varying the ratio of the catalyst components, the reaction temperature, and the gas composition in the reactor. The overall molecular weight distribution is relatively broad resulting in good extrusion processability. Figure 3 shows the typical distributions of molecular weight and comonomer for polyethylene produced with the PRODIGY BMC-200 Bimodal Catalyst System. This combination of molecular weight distribution and comonomer distribution results in an excellent balance of stiffness (pressure rating), slow crack growth resistance (SCGR), resistance to rapid crack propagation (RCP), and extrusion processability.

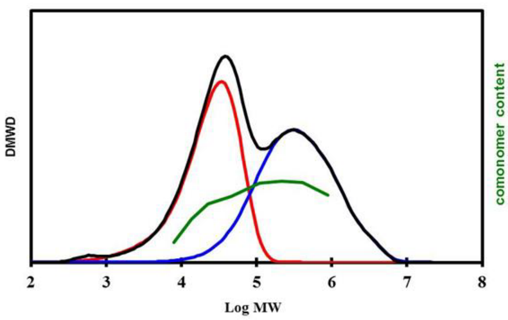

Figure 3. MWD and Comonomer Distribution for PRODIGY PE100 Resin

Polymer Properties

The PRODIGY BMC-200 PE100 resin, when first introduced, had a melt index MFR_5 that was outside the range specified in the ISO water and gas product standards, ISO 4427 and ISO 4437. The 2007 versions of these ISO standards specified the MFR_5 range of 0.2 to 1.4. The melt index for the PRODIGY PE100 product was outside this range because the polymer was designed for the end-use properties and processability by optimizing the individual component Mw's and MWD's, the split, and the comonomer content. However, simply specifying the resin's melt index and density are not sufficient for designing bimodal or multimodal polymer products for demanding applications such as PE100 pipe.

The MFR_5 of the PRODIGY PE100 product is in the range of 0.15 to about 0.18 compared to the lower limit of 0.2 in the earlier ISO standards. Consequently, the melt flow ratio is somewhat higher than that of other PE100 materials. The MFR_5 range in the ISO standards was eventually modified in order to accommodate lower melt index materials such as PRODIGY PE100. This is discussed further later in the paper; there was a significant amount of fusion compatibility testing done in order to support the petition to make the change. Overall, the PRODIGY PE100 material meets or exceeds the critical pipe properties required for PE100 applications, including long-term hydrostatic strength MRS10, slow crack growth resistance, and resistance to rapid crack propagation.

The combination of the low MFR_5 and the characteristic molecular weight distribution gives the PRODIGY PE100 resin some unique properties. In the extrusion of pipes, especially large pipes, the flow characteristics at different rates is important. At high shear rates, it is desirable to have a relatively low viscosity to enable low head pressure and good extruder output. The flow of the polymer as it exits the die is also critical. At the die exit, the shear rate is relatively low. Gravity acts on the extrudate as it flows from the die exit to the first cooling tank. Therefore, it is desirable to have high viscosity at low shear rate in order to prevent sag, a flow of the extrudate from top to bottom. Commercially-available "low sag" PE100 materials exhibit high viscosity at low shear rates.

The rheology of the PRODIGY PE100 exhibits a high degree of shear thinning. The complex viscosity, measured at 190°C, is shown in Figure 4. The competitive PE100 used for comparison is a popular "low sag" grade. At relatively high shear rates or frequencies in the dynamic rheology test, the complex viscosity of the PRODIGY PE100 is similar to that of the competitive PE100. However, at low frequencies, measured down to 0.01 s^{-1}, the complex viscosity of the PRODIGY PE100 is significantly higher than that of competitive PE100 material. Thus, the PRODIGY PE100 exhibits a unique balance of high viscosity at low frequency and desirably low viscosity at high frequency.

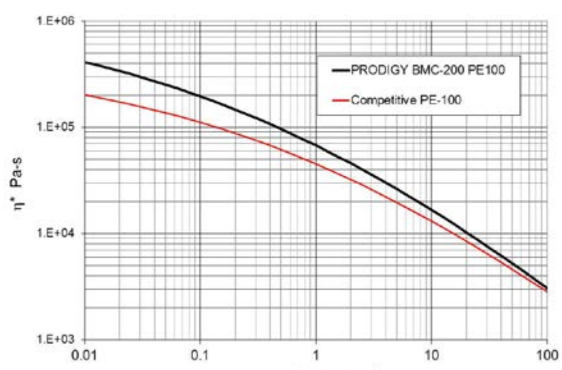

Figure 4. Complex Viscosity Tested at 190°C

The melt flow of the PE100 materials may also be characterized using a Rheotens rheometer to measure the melt strength in the lab. In this test, the extrudate from the rheometer is pulled at a constant acceleration rate while the force in the extrudate is recorded. Figure 5 shows the melt strength tested at 190°C comparing the PRODIGY PE100 to four competitive PE100 product grades, including “low sag” materials. The melt strength of the PRODIGY PE100 is significantly higher than that of the competitive grades. In pipe extrusion, depending on the specific extruder and operating conditions, a given material may be extruded at a range of melt temperature. Therefore, the melt strength test has been conducted at a range of temperatures with the same trends between the materials.

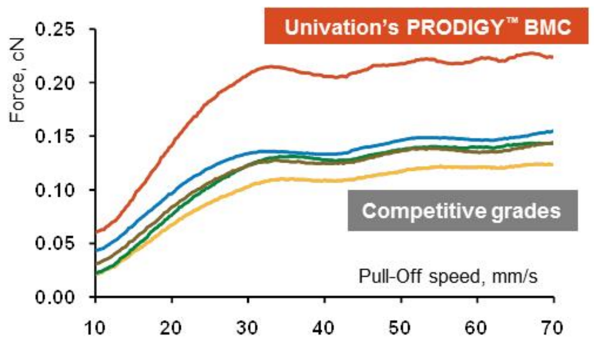

Figure 5. Rheotens Melt Strength at 190°C for PE100 Materials

In the commercialization of the PRODIGY PE100 material, demonstrations of fusion compatibility for both butt welding and electrofusion were done. In these fusion processes, the polymer at the interface is melted and then cooled while in contact with either another pipe or a fitting. Therefore, the melting and cooling characteristics of pipe materials are important to the fusion process. The melting and cooling of polyethylene is characterized in the lab by differential scanning calorimetry (DSC). In the DSC test, the heat flow is measured as the polymer is melted, cooled, and then re-melted. Figure 6 shows the DSC scans for the PRODIGY PE100 and for a competitive PE100 resin. The thermal behavior is similar with respect to positions of melting and cooling peaks as well as the area under the peaks. This data demonstrates that the overall crystallinity and thermal properties of these materials are similar. This is significant because pipes and fittings may be produced from different PE100 materials but the DSC data demonstrates they will heat and cool in a similar manner. This was demonstrated with successful electrofusion of PRODIGY PE100 pipes to fittings produced with another PE100 material.

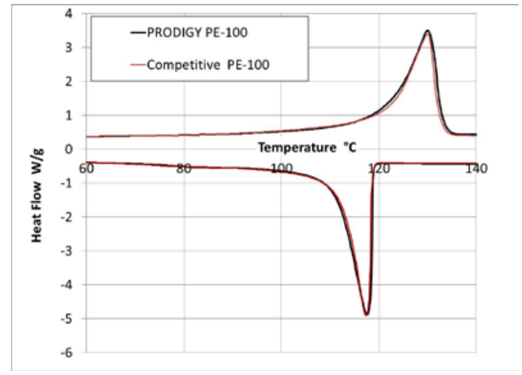

Figure 6. DSC Heating and Melting Behavior

Pipe Extrusion Characteristics

The extrusion performance of PRODIGY PE100 is advantaged, in particular for fast extrusion of large, thick wall pipes. The unique rheology results in a relatively high specific output and low melt temperature and subsequently faster cooling rates. The output of many pipe extrusion operations is constrained by the capacity to cool the pipe downstream of the die. Figure 7 shows the specific output and melt temperature of the PRODIGY PE100 along with 5 other commercial PE100 materials. This data was generated from an extrusion trial run on a 90 mm diameter grooved barrel extruder in commercial operation. This trial produced 250 mm SDR11 pipes. These extrusion characteristics enable good control of the wall thickness and ovality, two properties important for efficient and high quality pipe joining.

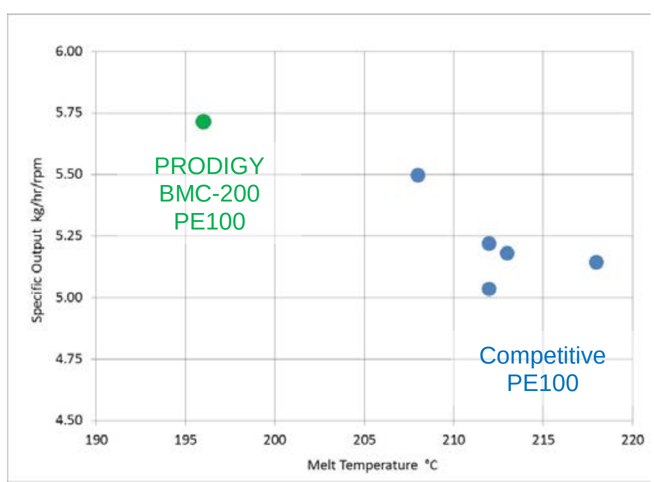

Figure 7. Specific Output and Melt Temperature During Production of 250 mm Diameter SDR11 Pipes

Case Studies of Large PE100 Pipes

In 2011, Hunter Water in Australia evaluated options to replace an old 900 mm diameter steel water pipeline that, because of the age of the pipe and corrosion, required regular maintenance and repair. PE100 pipe was chosen for its flexibility, corrosion resistance, and ability to use advanced installation techniques. HDF145B PE100 material, produced by Qenos Ltd in Australia with the PRODIGY catalyst, was chosen to produce the 1,000 mm diameter PN16 pipes.[1] The new pipeline installation achieved some significant environmental benefits.

The route of the pipeline was through a sensitive wetlands area and the new underground installation minimized disturbance to the local environment. The old pipeline used a bridge to cross a river. The bridge was replaced with the PE100 pipe which was installed under the river bed using horizontal directional drilling (HDD). In order to perform the HDD installation, about 300 meters of the pipe was assembled by butt fusion using standard welding parameters. The welding operation is shown in Figure 8. Figure 9 shows the length of 1,000 mm diameter PE100 pipe attached to a steel puller as it is readied for HDD installation. The toughness of the PE100 material allows this type of installation. The pipe is subjected to high tensile stresses as it is pulled through the pilot hole. Further, the pipe may be scratched or gouged as it is pulled through; the high resistance to slow crack growth ensures that the pipe is still suitable for long-term service after installation.

Figure 8. Butt Welding of 1,000 mm diameter pipes in the field

Figure 9. A 300 meter long length of pipe prepared for HDD installation

Another PE100 installation success story in Australia was achieved in 2012. The heaviest rainfall recorded in the Melbourne area in 133 years caused the breach of a levee and the water from the Morwell River flooded an open-cut coal mine. The coal from this mine directly supplied a power plant on the same site which was supplying a significant portion of the electricity for the state of Victoria. [2]

In order to alleviate the flooding in the mine and power plant, an emergency response team devised a solution to dewater the mine and to divert the flow of the river. Dams were constructed and PE100 pipes (500 mm and 630 mm diameter) produced with HDF145B from Qenos (produced with PRODIGY BMC Catalyst) were used to transport the water out of the mine (Figure 10). The second phase of the project was to divert the entire flow of the river around the affected area using PE100 pipes. This phase of the project used 1,600 mm diameter PE100 pipes to transport the 800 million liters per day of water (maximum flow of 2,800 million liters per day).

Figure 10. PE100 pipes used to dewater the mine

The emergency response team evaluated options to alleviate the flooding and to divert the river flow. Several factors dictated that PE100 pipe would be the optimum solution for the problem. PE100 provides the mechanical properties required for the water distribution application – pressure rating, impact resistance, slow crack growth resistance. The PE100 pipes offer ease of installation relative to other materials, with the ability to handle long lengths of pipes due to its light weight and the ability to butt weld in the field in order to produce leak free joints.

The Qenos HDF145B material, produced with PRODIGY BMC-200 Catalyst Technology, was selected specifically for the application. Delivery time was critical. The ability to extrude with high rates at relatively low melt temperature allowed the fastest possible production of pipes. The excellent extrusion characteristics ensured optimum performance in the field installation. The exceptional melt strength resulted in a very uniform wall thickness distribution and tight control of the ovality of the pipes. In large pipe installations, if wall thickness distribution is not substantially uniform, the pipes need to be routed to match up thicknesses of the pipes being joined. However, because of the successful production of consistent pipe with the Qenos HDF145B material, the pipes were produced within specifications, eliminating the need to field rout the pipes in the fusion process. This greatly facilitated the installation process. Figure 11 shows the 1,600 mm diameter pipes being butt welded in the field. The timeline of the project, including the diversion of the river flow, was 2 months; because of the fast response, the power supply to Melbourne and the state of Victoria was uninterrupted.

Figure 11. Field butt welding of 1,600 mm diameter pipes for river diversion

Pipe Standards

The plastic pipe industry has successfully developed standards to ensure durability and high reliability and to ensure regulation and adherence to specifications. This was possible due to a strong commitment from the entire value chain including raw material suppliers, product manufacturers, test laboratories, and end users. In turn, standards have created the incentive for suppliers to improve material performance, and for manufacturers to develop more innovative products.

PE materials are only partly defined by melt flow ratio and density; the resin's molecular structure can be more specifically tailored to meet the performance demands of specific applications. Therefore, it is important that standards and specifications for pipe products specify adequate requirements to ensure that only the highest performance materials conform and are specified.

The latest editions of system standards ISO 4427-1/2/3/5:2007 PE water and ISO 4437-1/2/3/4/5:2014 gas reflect the advancements in PE100 material performance and the successful field experience with lower MFR_5 (< 0.2 g/10min) bimodal PE100 resins over the last decade. For example, the 2007 versions of these standards specified an MFR_5 range of 0.20 - 1.40 g/10min. The current versions of the standards allow the use of materials with MFR down to 0.15 g/10min as per the following extract from Table 1 in ISO 4437. With regard to a pipe system made with such PE100, "attention is drawn to the fusion compatibility" as shown in note 9 in the following table.

Table 1. Melt Index Specified Per ISO 4437:2014

Characteristic	Requirement [a]	Test parameters		Test method
		Parameter	Value	
Melt mass-flow rate (MFR) for PE 80 and 100	0,20 to 1,40 g/10 min [fg] Maximum deviation of ± 20 % of the nominated value	Load	5 kg	ISO 1133-1
		Test temperature	190 °C	
		Time	10 min	
		Number of test pieces [b]	According to ISO 1133-1	

[g] Materials of nominated value 0,15 ≤ MFR < 0,20 may be introduced, in such case attention is drawn to the fusion compatibility subclause 4.5. The lowest MFR value resulting from the maximum lower deviation of the nominated value to be not less than 0,15.

Standards also cover advancements in product capability to make large diameter pipes. Even for large dimensions, electrofusion (EF) welding is an indispensable joining method. Today, electrofusion fittings are used for pipe with diameters up to 800 mm in the form of couplers for axial joints and saddle components for branches and connections. Permissible operating pressures of up to 2,500 kPa (water) and 1,000 kPa (gas) have become a norm. Operating practices in some applications like coal seam gas in Australia are moving towards even higher pressures of 1,600 kPa for the transport of gas. Therefore, it is critical to ensure that good integrity EF joints are formed even with the combination of very large pipes and lower MFR_5 (< 0.2 g/10min) PE100.

In the electrofusion process, heat is generated internally by wires embedded in the fitting. Tight tolerances on the roundness of the pipes and the gap between the fitting and pipe are critical. However, as pipes get larger, the specified dimensional tolerances increase.[3] Therefore, improvements in consistency of the dimensions of large pipes achieved through the extrusion process are essential for success in the EF process.

Electrofusion and Joints integrity testing program

A test program to validate the EF compatibility of the PRODIGY PE100 with commercial EF fittings was coordinated between Univation Technologies, Qenos (resin manufacturer), pipe extrusion companies and fittings manufacturers.

The test program involved manufacture of SDR17 pipes, using the PRODIGY PE100 material, in four different diameters: 800 mm, 630 mm, 560 mm and 110 mm. Pipes were extruded as part of commercial production at pipe manufacturers Vinidex (630SDR17 and 560SDR17) and ZEZT (800SDR17 and 110SDR17). Additional details of the test program were presented at the Plastic Pipes XVIII Conference in 2016.[3]

Table 2 outlines some of the EF test conditions. A majority of the investigation was conducted at Plasson (Israel) using electrofusion coupler and saddle type joining and product testing on SDR17 pipes in diameters of 800 mm, 630 mm, 560 mm and 110 mm; with some conducted at Friatec. Both Plasson and Friatec used “off-the-shelf” fittings produced with their standard PE100 materials. Figures 12 and 13 show, respectively, a 630 mm diameter coupler getting hydrostatically tested and a 110 mm diameter saddle assembly getting ready for product testing at Plasson testing premises in Israel.[4] The 800 mm diameter electrofusion coupler testing program was performed by Friatec and SKZ Institute in Germany. [5]

The Plasson program investigated EF weld temperatures from -10° to +45°C, varying the gap between pipe and fitting as well as different weld energy inputs. Electrofusion weld integrity was assessed via pipe hydrostatic pressure testing per EN 12201-3, Decohesion and selected Strip Bend tests according to ISO 21751.

Table 2. Electrofusion Testing Program Parameters

Pipe Diameter, mm	Pipe Wall, mm	Pipe MFR5	Fitting type/ Dimensions	Weld conditions	Hydrostatic test conditions	Decohe-sion
110	7	0.160	Coupler 110SDR11 (PE100)	2xMin + 1xMax [1]	80°C/1000hrs/ 6.3bar[2]	After hydr. test
			Tapping Saddle 110x40SDR11 (PE100)	Min+Max[1]		
560	35	0.150	Coupler 560SDR11 (PE100)	Min+Max[1]	80°C/1000hrs/ 6.3bar[2]	After hydr. test
630	40	0.196	Coupler 630SDR11 (PE100)	Min+Max[1]	80°C/1000hrs/ 6.3bar[2]	After hydr. test [3]
			Tapping Saddle 630x110SDR11 (PE100)			
800	50	0.165	Coupler 800SDR17 (PE100)	“Standard”	80°C/1000hrs/ 5.0bar	After hydr. test

[1]Min and Max outline the most extreme weld conditions, as per below:

Minimum weld conditions:

- Weld temperature: -10°C
- Gap: Maximum gap between fitting and pipe (pipe machined to simulate the situation where the fitting is at its upper internal diameter tolerance, pipe at its minimal external diameter as defined by the standard + removal of 0.6 mm off the pipe diameter to simulate pipe scraping
- Weld energy: minimum possible energy input which is calculated taking into account the controller lower energy tolerance (as defined in the standard) and the product highest resistance allowed based on the product definition

Maximum weld conditions:

- Weld temperature: +45°C
- Gap: Maximum gap between fitting and pipe (pipe machined to simulate the situation where the fitting is at its upper internal diameter tolerance, pipe at its minimal external diameter as defined by the standard + removal of 0.6 mm off the pipe diameter to simulate pipe scraping
- Weld energy: maximum possible energy input which is calculated taking into account the controller upper energy tolerance as defined in the standard, and the product lowest resistance allowed based on the product definition

[2]The test conditions were set in accordance with the pipe SDR/PN rating as in EN 12201

[3]On the 630 mm diameter saddle, a strip bend test was performed according to ISO 21751

Figure 12. 630SDR17 Gas Pipe and Electrofusion Coupler During Hydrostatic Pressure Testing

Figure 13. 110SDR17 Water Pipe and Tapping Saddle Electrofusion Assembly Ready For Product Testing

The EF assemblies were tested in the hydrostatic pressure test under the conditions given in Table 2. Additionally, the EF assemblies were also tested in a decohesion test; the joints should exhibit ductility in the decohesion test. All results from the testing program recorded PASS, demonstrating high quality electrofusion capability for joining of large diameter thick walled pipes made using a PE100 with $MFR_5 < 0.2$ g/10min. Comments from the test laboratories on the test performance results were that electrofusion fittings have all coped well with welding to pipes manufactured using this PE100 material with MFR_5 as low as 0.15 g/10min.

Conclusions

- Using PRODIGY BMC-200 Catalyst Technology, PE100 pipe resin with excellent mechanical properties has been developed and is commercially produced.
- The extrusion characteristics of the PRODGY PE100 resin are unique, facilitating the extrusion of large thick pipes.
- The recent change in standards ISO 4427 and ISO 4437 to include a provision for PE100 resins with $MFR_5 < 0.2$ g/10min reflects the intent of standards to keep pace with the development and innovations associated with pipe materials.
- The electrofusion work demonstrates that high quality joints can be produced with pipes made from PE100 with $MFR_5 < 0.2$ g/10min and commercially available electrofusion fittings
- It is possible to join using electrofusion very large and thick wall pipes as demonstrated by extensive product testing of electrofusion assemblies with pipe of sizes up to 800 mm in diameter and up to 50 mm in wall thickness

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