



Code of Practice

PE Material Selection and Quality

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Important note on use of the *APGA Code of Practice for PE Material Selection and Quality* and its companion papers.

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1.Scope

The scope of this Companion Paper is related to the provision of an overview of the manufacturing process of PE pipes and fittings from polymerisation to extrusion or moulding. A description is given of the major aspects of the processes and the controls that have been adopted to meet the requirements of all stakeholders.

Characteristics of polyethylene, such as time dependence of some of the properties are discussed.

The purpose is to provide a background to the PE 100 material used in the CSG gathering lines (both gas and water), the relevant Australian Standards, pipe extrusion and the material qualification procedures.

A brief reference is also made to crosslinked polyethylene (PE-X) and raised temperature polyethylene (PE-RT), both of which are covered by national and International standards and which may find some application where higher temperature performance is required than that available with PE 100.

Reference is made to the existence of proprietary oriented PE tape reinforced monocomposites (PE-O) which offer higher pressure performance compared to PE 100.

Reference is also made to grades of PE 100 with increased stress crack resistance (known as PE 100RC or PE 100HSCR).

2.Introduction

Within Australia, the pipeline industries transporting water, gas and associated materials are fortunate to be supported in the use of two major materials (PE and PVC) by the Plastics Industry Pipe Association of Australia Limited (PIPA) which has a high international reputation, with excellent cooperation with ISO, among others, TEPPFA in Europe and PPI in USA.

PIPA's Guidelines [POPs] and Technical Notes form the basis on which materials selection is recommended, and the recent advances in risk-based design are fully linked with advances in the components provided by the industry members represented locally by PIPA.

The Australian CSG industry design is essentially based on CBM/CSG experiences in the latter 20th century in USA, adapted and extended for Australian conditions (however with an absence of snow and ice), construction methodology (welding and laying), with principle component suppliers (valves, couplings, etc.) originating from large international companies predominantly based in Europe and Israel, with extensive research and development, advanced QA/QC methodologies; and now with globalisation, most manufacturing is based in Asia and the Middle East.

Similarly, under PIPA's guidance the resin industry in Australia is obliged essentially to supply only pre-compounded resins meeting AS/NZS 4130. The major resin suppliers currently represented in Australia not only have high-quality standards, and are in the forefront of new materials development which is expected to be used within the resource industries (CSG, CBM, mine de-watering, etc.) in coming years to provide innovative solutions to design challenges.

Australia's major PE pipe manufacturers apply best international practice in material selection and extrusion. Accordingly, quality PE pipe should be available to support the resource industries in Eastern Australia for the foreseeable future.

PE 100 is the term to be applied for the current HDPE material used in CSG pipelines. This paper provides the necessary technical background to explain how this material is the preferred product for the CSG industry, handling both water and gas.

3. PE Pipe and Fittings Materials

3.1. PE Polymer Manufacture

In contrast to some types of plastics, PE material for pipes and fittings is sold by the polymer companies as fully pre-compounded granules containing all of the additives, such as anti-oxidants and pigments, needed to process and protect the polymer. This practice is particularly true in Australia, Europe, New Zealand and Scandinavia. Some countries allow additives to be incorporated at the extruder during the extrusion process. These countries include USA and China (only for water or drainage pipes, not gas). The addition of any master batch additive at the extruder is explicitly disallowed in Australia in the pipe Standard AS/NZS 4130. This restriction to only approving fully pre-compounded materials provides the following benefits:

- Greater level of control during the extrusion process.
- Better reproducibility.
- Higher confidence level in the finished product
- Allows the establishment of independent compound verification processes, such as that adopted by PIPA as published in POP004.
- Better butt weld performance, especially for thick-walled pipes

The addition of pigmented master batch at the extruder in other countries is reported to result in clear areas of polymer devoid of additives, especially in thick walled pipes, which may have a deleterious effect on pipe performance.

PE pipe compounds are made in two stages. In the first stage, the monomers are reacted to form a polymer, a very sophisticated process with polymer architecture designed to optimise characteristics such as processing, long-term strength, resistance to slow crack growth and resistance to rapid crack propagation. The polymer comprises long chain hydrocarbon molecules with strategically placed hydrocarbon side chains. The number, length and placement of the side chains play a major role in determining the performance of PE. Recent developments in catalysts and processes have allowed quite specific molecular architecture to be developed, thereby enhancing the PE performance. Furthermore, PE 100 has what is called bimodal molecular weight distribution. The bimodal distribution, in contrast to the older style monomodal distribution, has allowed optimum physical characteristics to be developed in the polymer whilst maintaining processability.

3.2. Definition of PE 100 for CSG

While PE materials are commonly referred to as high density (HDPE), medium density (MDPE), low density (LDPE) or linear low density (LLDPE) these descriptors are not appropriate for the PE pipe compounds used for the CSG industry. There is no universally accepted delineation between MDPE and HDPE and density alone does not adequately describe the material. The term PE 100 should always be applied to the PE pipes used for CSG as it ensures that all of the appropriate performance requirements have to be met rather than simply a density range.

3.3. Compound Extruding Process

After polymerisation and drying, the PE passes through a compounding extruder where the carbon black and other additives are incorporated. The compounding extruders are specially designed to achieve the high level of dispersion and distribution of the additives critical to long-term performance. Pipe extruders are designed to achieve uniform heat distribution in the PE compound and provide high output. They are generally NOT designed to develop the high shear stress rates necessary to disperse additives. This is the primary reason for specifying the use of pre-compounded PE rather than allowing the incorporation of additives such as carbon black into the PE at the pipe extruder.

To ensure the intended compound characteristics have been achieved, compound manufacturers generally test a number of characteristics including density, oxidation induction time (OIT), melt flow rates and dispersion. Density, in this context, is used to show the process is in control. These tests ensure the intended polymer architecture and additive concentration has been achieved, but these aspects are all pre-determined by the close monitoring of the processing conditions.

As the compound exits the compounding extruder it passes through a die and is diced into small, regular granules or beads. The finished compound is packed in 1 tonne bulk bags or container lots for shipment.

3.4. PE-X Material

Crosslinked polyethylene (PE-X) material is similar to PE 100 but the material undergoes a further process after extrusion. This involves forming crosslinks between the polymer chains using either chemical means or radiation. The crosslinks change the thermoplastics material into a thermoset with enhanced performance at elevated temperatures. PE-X pipes are, for example, used in hot water plumbing applications. All PE-X pipes have to meet the same minimum performance characteristics so there is no need to discriminate between pipes made using different methods of crosslinking. Because the crosslinking turns PE-X into a thermoset particular consideration has to be given to the jointing methods. Crosslinking also creates difficulties with respect to the use of rework or recycle.

3.5. PE-RT Material

Raised temperature polyethylene (PE-RT) also has enhanced elevated temperature performance compared to PE 100 but the material remains a thermoplastic and, in all respects, can be processed and joined in the same manner as PE 100. There are currently two Standard grades of PE-RT with different elevated temperature performances. PE-RT Type 2 most closely approximates the strength characteristics of PE100 but does not necessarily achieve a MRS of 10 MPa. The performance characteristics of both PE-X and PE-RT are defined in ISO 15494.

3.6. PE100RC

There have also been improvements in the stress crack resistance of PE100, resulting in grades designated as PE100RC (or proprietary but equivalent designations such as PE 100 HSCR). New methods of test have been developed in order to characterize these high stress crack growth resistant PEs in an acceptable time frame. These materials are true PE 100s in that they have a MRS of 10 MPa, but with even greater resistance to slow crack growth than traditional PE 100. That is, they not only meet the requirements applicable to PE 100 but also the additional stress crack resistance performance requirements listed in POP016.

The enhanced slow crack growth requirements for PE100RC materials have been incorporated into the European PE pipe and fittings Standards and work is in progress to do the same for ISO PE pipes and fittings for both gas (ISO 4437) and water (ISO 4427) applications. It is anticipated the same slow crack requirements for PE100RC will ultimately be incorporated into AS/NZS 4131 and AS/NZS 4130.

PIPA Industry Guideline POP016 *High Stress Crack Resistant and Raised Crack Resistant PE100 Materials* describes the requirements for PE100 HSCR / PE100 RC compounds and POP004 *Polyethylene Pipe and Fittings Compounds* lists those compounds that have demonstrated compliance.

3.7. PE-O Material

There has been an emergence of proprietary pipes made exclusively from polyethylene but which offer significantly higher pressure performance than traditional construction. Commonly referred to as PE-O or monocomposite pipe, these consist of at least three bonded layers of PE materials which perform the following functions: The inner layer is a homogeneously extruded PE tube that conveys the gas/water and provides support for the middle layer. The middle layer provides the additional strength and consists of at least two helical wraps of oriented PE (PE-O) tape applied onto the inner layer. The outer layer is homogeneous coextruded PE applied over the middle layer and serves as a protective wear liner. These 100 per cent PE composites retain similar thermal and chemical resistant

properties to their parent PE pipe materials; however the oriented tape layer significantly improves the MRS across the range of operating temperatures. PE 100, PE-RT or PE-RC materials may be used for internal and/or external bonded liners. Since the oriented tape layer, which provides this increase in MRS, is discontinuous between pipe sections, jointing of PE monocomposite pipes is more complex and relies on proprietary joining couplers and methods to ensure the MRS of the system is retained.

Reference T. Späth, High-pressure pipes made from polyethylene only - A New Generation of Plastics Pipes, Proceedings of the 19th Plastic Pipes Conference PPXV, Vancouver, September 2010.

3.8. Wire reinforced Polyethylene

Proprietary wire reinforced PE pipes with the capability of operating at higher pressures have been developed overseas. These products have their own proprietary jointing systems. There are no existing Australian or ISO Standards for wire reinforced PE pipes although some other national Standards, for example China, have been published.

4. Primary PE Compound Properties

The characteristics of PE paramount to its satisfactory performance in pipe and fittings applications include:

- Resistance to slow crack growth;
- Resistance to rapid crack propagation (RCP);
- strength; and
- Thermal stability.

4.1. Resistance to slow crack growth

Resistance to slow crack growth is one of the characteristics that has been greatly enhanced by the ability to manipulate the molecular architecture during polymerisation. It is an extremely important property as it defines the ability of the material to resist the propagation of a crack through the wall of the pipe and which would ultimately lead to failure. Improvements have been so great that it has been necessary to develop new test methods to assess this characteristic as the tests employed with the older generation PE materials cannot be completed in an acceptable timeframe. That is, modern materials last too long in the traditional tests.

4.2. Resistance to Rapid Crack Propagation

Resistance to Rapid Crack Propagation (RCP) is a characteristic that is not limited to PE but is particularly important for pipelines carrying compressible fluids. AS/NZS 4131 defines the RCP performance requirements for PE 100. Under the temperature and pressure conditions to be experienced in CSG applications in Australia, RCP will not occur in PE 100 pipes.

4.3. Strength

The strength of the PE compound determines the allowable hoop stress, generated by internal pressure that the pipe can be subjected to during service. However, PE along with other plastics materials is viscoelastic and its strength is a time-dependent property. Sometimes this time-dependency is mistaken for degradation of the material with ageing. However, if the strength of a sample of PE is re-measured using the same test conditions, sometime after an initial measurement, the results will be the same. This will be the case unless there has been some chemical degradation due to, for example, UV attack causing a change in the molecular structure. The original short-term strength will be retained even on pipe samples exhumed after many years of service. The time dependency of the strength means particular arrangements need to be made to determine the stress a plastics pipe can withstand for the duration of its intended service life. In the case of PE 100, a large number of pipe samples are subjected to a range of pressures at 3 temperatures, usually 20°C, 60°C and 80°C.

The time to failure and the nature of the failure of each sample is noted and analysed in accordance with ISO 9080. Testing at elevated temperatures accelerates the tests and ISO 9080 provides explicit rules governing the extrapolation of the results to longer periods. The lower 97.5 per cent prediction limit (σ_{LPL}) at 20°C is calculated at 50 years. This number is then used to determine the minimum required strength (MRS) in accordance with ISO 12162. PE 100 materials must have a σ_{LPL} at 20°C of not less than 10 MPa, giving an MRS of 10. The use of σ_{LPL} at 50 years when determining the MRS is a matter of convention and should not be interpreted as a limit on service life. Given the period and temperatures for which PE pipes are usually pressure tested, it is generally accepted that they can be expected to have a service life in excess of 100 years at 20°C.

4.4. Thermal Stability

Antioxidants are incorporated into the polyethylene during manufacture to ensure the polymer has sufficient thermal stability to allow for extrusion and moulding as well as the forming and welding operations which take place above the crystalline melting temperature along with exposure to service conditions without adversely affecting the performance of the material.

5. Secondary PE Compound Properties

Other characteristics of PE pipes of interest to the end user are:

- reversion
- residual stress _
- creep and stress relaxation
- coefficient of thermal expansion
- resistance to degradation.

5.1. Reversion

Reversion is the contraction in length that occurs as a result of the longitudinal stress imposed on a pipe during extrusion. It occurs on standing and is accelerated at higher temperatures. With modern extrusion equipment reversion in PE pipes is invariably well

below the maximum of 3 per cent permitted when tested at 110°C. Reversion is of no real consequence in the context of CSG pipes.

5.2. Residual stress

Residual stress is generated during the cooling process after the pipe exits the extrusion die. Residual stress is responsible for the toe-in of the cut end of the pipe. The magnitude of the toe-in is dependent upon the amount of residual stress and the time dependent modulus of the PE. On a freshly cut pipe end there will be an immediate small toe-in and this will increase slowly with time. The rate of increase in toe-in will progressively slow. On the other hand, as a result of stress relaxation, the residual stress diminishes with time. Toe-in will occur at any freshly cut pipe end, but will be less on old pipes

5.3. Creep and stress relaxation

Creep is a characteristic that is not limited to plastics as it also occurs in metals. The difference is that in plastics its magnitude is significant at normal operating temperatures. Creep is essentially the strain that takes place as a result of the gradual release of localised stresses by short-range molecular rearrangement. Because the strain, at a constant stress, increases with time the modulus of PE is also time dependent, but the rate of change in both strain and modulus diminishes with time.

A related characteristic is stress relaxation in which a specimen subjected to a constant strain exhibits a diminishing stress with time. This characteristic means that stresses created for example, by bending or deflecting a pipe, progressively diminish with time.

The time dependency of some of the properties of plastics is not an impediment to their use in a range of engineering applications as the characteristic is understood and taken into account in product design.

5.4. Coefficient of thermal expansion

Plastics generally have much higher coefficients of thermal expansion than metals and PE is no exception. This may need to be taken into account during installation and design. For example, a hot pipe placed in an open trench during the day might contract significantly during a cold night if not backfilled. Once the trench has been backfilled, soil – pipe friction prevents contraction.

5.5. Resistance to degradation

Non-compounded PE exposed to high temperatures and UV radiation will degrade. For this reason antioxidants and UV stabilisers or UV absorbers are incorporated into the compound. The presence of antioxidant is assessed by the OIT test which is performed both on the compound and the pipe. Residual antioxidant is needed to protect the polymer during extrusion and to protect the pipe during welding and service. In Australia protection against UV radiation is addressed by specifying the amount and type of UV stabiliser or absorber in the compound.

6. Manufacturing

6.1. Pipe Extrusion

The major pipe manufacturers receive PE compound in bulk. Bulk compound is transferred to a silo from which it is passed through a drier immediately prior to extrusion. The drier removes any excess moisture absorbed by the compound and eliminates the possibility of

porosity in the finished product. The function of the extruder is to melt the compound evenly and form it into the pipe shape. The temperature of the extruder components and the polymer melt are monitored continuously to ensure the material is sufficiently hot to be formed into a coherent pipe, but not over heated to cause degradation of the polymer.

Of all the characteristics of a PE pipe, the ones that the pipe manufacturer most controls are the dimensions. He can also influence the residual antioxidant level (as measured by OIT). The strength (i.e. resistance to internal pressure), modulus, slow crack growth and rapid crack growth resistance are determined by the molecular architecture and composition of the compound over which the compound producer has control, but not the pipe manufacturer. Nevertheless, some material properties such as resistance to internal pressure, reversion, pigment dispersion along with a general inspection for freedom from defects may also be tested by the pipe or fitting manufacturer as part of their ongoing QA.

Appendix A of AS/NZS 4130 summarises a set of test requirements for PE pipe along with minimum testing frequencies. Manufacturers often exceed these testing frequencies or employ additional or alternative tests to monitor quality. In some cases, attributes such as dimensions are continuously measured at the extruder.

For QA purposes the most important things for the pipe manufacturer to monitor are the processing conditions, dimensions, OIT and the compound batch quality data issued by the compound producer.

The extrusion process is a continuous one, but usually limited to finite production runs. The length of a run will be determined by demand for the product and availability of the extruder given a single extruder will normally be used for a range of pipe diameters and pressure classes.

6.2. Moulded Fittings

The same comments as made in relation to extrusion apply to moulding except for the difference in the processing operation. Moulding companies produce fittings overseas on an international scale from fully precompounded polyethylene, supplied in bulk. The processing equipment consists of a split metal block mould and an injection moulding press that forces polyethylene melt into the mould. The mould is cooled and the part demoulded when sufficiently cool to retain its shape. The process is intermittent. Fittings are produced in a range of SDRs (i.e. pressure capabilities). Smaller fittings may be produced in multi-cavity moulds and larger fittings in single cavity moulds. Moulds can be interchanged on the machines in order to match the production of specific fittings types with demand. The process is highly automated with sophisticated monitoring of the processing conditions and product.

Fittings supplied in Australia are produced by companies with certified quality systems and the products comply with the relevant ISO product Standards. Fittings are packaged in a manner to protect them from damage during storage and transport. Electrofusion fittings in particular are individually wrapped to ensure welding surfaces are not contaminated.

Injection moulded fittings are permanently marked with traceability including details of the manufacturer, diameter, SDR/pressure class, material and date. Generally, fittings are black and not colour coded.

6.3. Product Certification

The plastics pipes and fittings industry in Australia has had a long experience with third party product certification and it is now the norm for all major products, including PE. Product certification has long been a requirement for plastics pipes supplied to the water and plumbing industries for pressure applications. The certification process has the following elements:

- A formal, auditable quality system and quality plans must exist.
- Compliance with all aspects of the product standard must be demonstrated, including selection of compounds complying with AS/NZS 4131 and satisfactory completion of all type tests.
- Process control methods are in place to ensure the process consistently complies with nominated criteria.
- There are commensurate QA methods to verify the intended outcomes have been achieved.
- Compliance audits are regularly performed to ensure the standards are being maintained.

Despite the value of product certification, asset owners should apply due diligence to the compound analyses, extrusion records and condition of pipe received.

6.4. Quality and Traceability

The traceability of PE pipes is provided by the message printed on the pipe at regular intervals. The message identifies:

- The manufacturer.
- The manufacturing site if the manufacturer has more than one production facility.
- The production line (i.e. the actual extruder)
- The date and time the pipe was made
- The grade of material (for example PE100)
- Dimensional and pressure class information as appropriate
- The number of the product Standard to which it is made
- Product certification details such as licence number.

This information allows people downstream of extrusion to fully identify the pipe and people upstream to link any pipe to the recorded processing conditions, test results and compound as needed. Similar requirements are recorded on PE fittings. Additional information may be printed on PE pipe by arrangement with the manufacturer.

The essence of the PE pipe and fittings quality system comprises:

- Strong, up-to-date raw material and product Standards aligned with best international practice,
- A process for independently assessing compounds to ensure compliance with the compound Standard and listing these compounds in a publicly accessible document (POP 004),

- Making it mandatory for fully pre-compounded materials to be used in the production of pipe and fittings,
- Having independent third-party product certification to ensure products comply with the Standards and that appropriate quality systems and procedures are in place and maintained,
- Comprehensive print messages on products to ensure full traceability.

These methods for the monitoring and control of PE pipe production are considered best practice.

References

AS/NZS 4129 Fittings for polyethylene (PE) pipes for pressure applications.

AS/NZS 4130 Polyethylene (PE) pipes for pressure applications.

AS/NZS 4131 Polyethylene (PE) compounds for pressure pipe and fittings.

ISO 15494 Plastics piping systems for industrial applications — Polybutene (PB), polyethylene (PE), polyethylene of raised temperature resistance (PERT), crosslinked polyethylene (PE-X), polypropylene (PP) — Metric series for specifications for components and the system.

PIPA POP004 Polyethylene pipe and fittings compounds.

PIPA POP016 High stress crack resistant PE100.