INEOS
Olefins & Polymers USA

Practical Applications of PENT Testing

White G. Jee, Staff Chemist,
INEOS Olefins & Polymers USA
La Porte, TX

Abstract

Prior to the recognition of the PENT (ASTM F 1473) test as a means to measure the resistance to slow crack growth in ASTM D 3350 (Standard Specification for Polyethylene Plastics Pipe and Fittings Materials) and ASTM D 4976 (Standard Specification for Polyethylene Plastics Molding and Extrusion Materials), INEOS Olefins & Polymers, formerly known as Solvay Polymers utilized the PENT test to evaluate its materials in various end-use applications. This paper reports some practical applications in which our company has come to use the PENT test as a tool in evaluating slow crack growth performance of materials. These applications include development of materials in pipe applications, evaluation of various gas pipe resins, and the effect of production process changes on polymer performance as evidenced by the PENT test results.

Introduction

The resistance to slow crack growth of a material is one of the most important factors that determine the lifetime of any piping system. There are various test methods in measuring resistance to slow crack growth. The most common test method utilized by the pressure pipe industry is the hydrostatic pressure test as defined in ASTM D 1598 and ASTM D 2837 (1)(2). ASTM F 1473 (the PENT test) is the standard test method for Notch Tensile Test to Measure Resistance to Slow Crack Growth of Polyethylene Pipes and Resins (3). INEOS Olefins & Polymers is one of the major polyethylene resin suppliers participating in the pipe market.

Currently we have two PE 2406 resins and five PE 3408 resins listed in PPI TR-4 (4). Prior to the establishment of the PENT test as a tool to evaluate slow crack growth performance, INEOS Olefins & Polymers utilized the accepted industry standard of sigma 5 testing (80°C hydrostatic testing @ 5 MPa hoop stress) and ASTM D 1693 ESCR testing as means to evaluate long-term performance of resins. These methods of evaluating resins provided meaningful data but were extremely time-consuming (over two months without failure). Therefore, a need existed in the industry to identify a test method that could provide useful information within a relatively short time period.

A published report had previously indicated that a correlation exists between PENT results and the expected lifetime of a gas pipe system (5) (6). Therefore, since the PENT test is faster, requires fewer specimens than the hydrostatic test, and has been established as an ASTM test method, the opportunity to utilize the PENT test as a tool for practical industrial applications appeared promising and thus was explored. This paper reports on the various projects in which PENT data has been utilized in an industrial environment to make constructive decisions. Also the paper reports on interesting PENT observations relative to materials that are being utilized in the pipe market.

Background

The PENT test was originally published as an ASTM test method in1994. After participating on round robin testing of compression molded plaque sand pipe samples and identifying sample preparation techniques to reduce variability, INEOS Olefins & Polymers began to utilize the PENT test more extensively in evaluating and monitoring its products, identifying areas to utilize the PENT test in conjunction or in lieu of hydrostatic testing, and establishing internal guidelines as a go/no go test. Additionally, the PENT testing capability has been used to monitor competitive resins and to service our customers in evaluating pipe samples that require further in-depth analyses. INEOS Olefins & Polymers along with other resin suppliers was actively involved in the development of the precision and bias statement for the PENT test method. Originally, the PENT testing equipment utilized was a two test station apparatus built by and purchased from Dr. Norman Brown of the University of Pennsylvania. Subsequently, the PENT testing capability was expanded in 1996 with the purchase of a 24 station apparatus from Tech Team, Inc. All PENT testing data was obtained under the standard testing conditions of 80°C (176°F) and 2.4 MPa (348 psi) stress. Recently, the PENT test gained greater emphasis with revisions to ASTM D 3350 and ASTM D 2513 (7)(8). In ASTM D 3350, the PENT test was added as an alternative method in determining the test property "slow crack growth resistance". The other method in ASTM D 3350 to measure slow crack growth resistance is ASTM D 1693 which is known as the bent-strip ESCR test (9). In ASTM D 2513, a PENT requirement of 100 hours minimum was established on materials used in this standard specification.

Table 1. ASTM D 3350 Slow Crack Growth Resistance Test Property

Property	Method	0	1	2	3	4	5	6	7
ESCR	D1693	#	A	B	C	C	N/A	N/A	*
a. Test Condition			48	24	192	600			
b. Test duration, hours			50	50	20	20			
c. Failure, max %									
PENT	F1473	#	0.1	1	3	10	30	100	*
Molded Plaque, 80 C, 2.4 MPa									

Establishing Minimum PENT Values for Existing Products

INEOS Olefins & Polymers' PE2406-1 is a medium density PE 2406 resin that is used in the natural gas industry. Extensive PENT data has been generated on this formulation along with the high density PE 3408 resins (INEOS Olefins & Polymers-1 and INEOS Olefins & Polymers' PE3408-2) used in pressure pipe. Using the cell class information identified in ASTM D 3350, the product data sheets for each formulation have been conservatively updated to include the PENT information. Actual PENT value ranges for certain formulations may be significantly higher than indicated on the product data sheet. In general, the industry has established sigma 5 testing (6 pipes @ 5 MPa hoop stress, 80°C) as a quick means to predict long-term performance. The sigma 5 test may also be known as the five sigma test. Although this testing is useful, the lengthy time-to-failure using this method is sometimes impractical in an industrial environment. Therefore, extensive PENT data has been generated on our resins and the PENT test has been useful as a tool in conjunction or in lieu of sigma 5 testing. Results have indicated a strong relationship between PENT data and sigma 5 data but more data is needed to determine if a direct

correlation exists between the test data for each formulation.

Table 2. PENT Information on INEOS O&P Pipe Products

Product Formulation	Material Designation	PENT Minimum (hours)	D335 Cell Classification	Typical PENT Range (hours)
INEOS O&P 2406-1	PE 2406	>100	234363E	>2500
INEOS O&P 3408-1	PE 3408	>100	345464C	150 -350
INEOS O&P 3408-2	PE 3408	>30	345454C	30 – 150
INEOS O&P 3408-3	PE 3408	>10	335444C	10 – 40
INEOS O&P 3408-4	PE 3408	>100	346564C	.1500

Use of PENT Data for Internal Optimization Projects In order to provide the quality resins required into today's demanding pipe markets, INEOS Olefins & Polymers has made various improvements in its chemical process, extrusion process, antioxidant and pigment supply over the last few years. The PENT test has been used as a tool in determining whether the changes resulted in affecting or improving the long-term performance properties of the material. The polyethylene manufacturing process is a continuous process whereby product formulations are scheduled in the most practical efficient manner to reduce the cost of transition from one product formulation to another. In order to minimize the transition period, the PENT test was used to evaluate the long term performance properties of the transition product. As a result of the PENT data generated, the transition period from one product formulation to another was reduced. INEOS Olefins & Polymers offers its customers in the gas pipe industry a pre-compounded yellow (INEOS Olefins & Polymers' PE2406-1) or precompounded black (INEOS Olefins & Polymers' PE3408-1 or INEOS Olefins & Polymers PE3408-2) material. As a means of assuring long-term performance properties are not affected by changes in suppliers of yellow concentrate, carbon black, or antioxidants, the PENT test is used as a tool in qualifying the supplier. The supplier qualification process typically involves production of the supplier's product into pipe and an evaluation of the resulting product for both PENT testing and sigma 5 testing. As a result of the PENT data generated, INEOS Olefins & Polymers has approved new alternative supplier(s) for its concentrate, carbon black, and antioxidants. Since the inception of its first listed product formulation in the early 1980's, INEOS Olefins & Polymers has devoted efforts into development of improved pipe product formulations. Today's product formulations used in the gas industry are significantly improved over the initial product formulations. However, using proprietary catalyst and extrusion technology, new products have been formulated for the next generation of resins. The PENT test has been used as a tool in assessing the long-term performance properties of these developing new product formulations. Often low, unsatisfactory PENT results can help eliminate potential new generation resin formulations prior to more extensive testing; thus reducing time and expenditure spent on development efforts.

Table 3. Summary of Internal Projects Utilizing PENT as an Evaluation Tool

1. 0ptimization of a Product Formulation's Transition Time
2. Qualification of Yellow Concentrate Suppliers
3. Qualification of Carbon Black Suppliers
4. Qualification of Alternative Antioxidant Suppliers
5. Evaluation of 'Next Generation' Resins for Lon -Term Performance
6. Developing a Correlation between PENT and Sigma 5 for Each Product Formulation

PENT Testing of Pipe Materials Used in Natural Gas Distribution

In the latest revision of ASTM D 2513 (Standard Specification for Thermoplastic Gas Pressure Pipe, Tubing, and Fittings), a materials requirement of 100 hours minimum PENT was added. Based upon the implementation of this new requirement, a joint study was conducted with some of our customers in evaluating the PENT of various materials that are being used in the natural gas industry. A total of 13 samples were collected from the field. Each sample was compression molded into a sample plaque and tested in duplicate for PENT. Results indicated that all the PE 2406 materials tested met the minimum 100 hours requirement; however, there is a large degree of variation in the different PENT values exhibited by the various PE 2406 materials. Results indicated that some of the PE 3408 materials tested were on the borderline in meeting the minimum 100 hours requirement.

Table 4. PENT Results of Materials Used in Natural Gas Distribution

PE2406	PENT Results	PE3408	PENT Results
Sample # 1	On Test >1440 hrs	Sample # 8	Failed at 81 hrs
Sample # 2	On Test >1440 hrs	Sample # 9	Failed at 52 hrs
Sample # 3	On Test >912hrs	Sample # 10	On Test >816 hrs
Sample # 4	On Test >912 hrs	Sample # 11	Failed at 182 hrs
Sample # 5	Failed at 880 hrs	Sample # 12	On Test >576 hrs
Sample # 6	Failed at 290 hrs	Sample # 13	Failed at 208 hrs
Sample # 7	Failed at 519 hrs		

Although the resistance to slow crack growth is important in determining the lifetime potential of a piping system, only the gas distribution industry has set a PENT requirement for materials used in this industry. Many different resins are used in other piping applications. As a means to valuate the types of products being utilized for the less regulated piping applications, PENT testing was performed on the various formulations.

Results indicated materials used in the corrugated pipe and conduit applications have lower resistance to slow crack growth than the materials used in pressure pipe applications.

Table 5. PENT Testing of Other Piping Applications

Piping Application	PENT Test Range (hours)
Corrugated Piping	0 – 5
Conduit	0 – 20
Water Service	30 - 50

Conclusion

This paper has focused on the practical applications of using the PENT test in an industrial environment. Based upon the numerous applications in which PENT data has been utilized as a means to measure resistance to slow crack growth, BP Solvay Polyethylene has identified the usefulness of ASTM F 1473 in defining the value for its products, optimizing internal projects, and evaluating competitive products. Results from the various applications indicate that the PENT test is a useful tool for the industry.

References

(1) ASTM F 1598, "Standard Test Method for Time-to-Failure of Plastic Pipe under Constant Internal Pressure ", American Society for Testing and Materials, vol. 8.04.

(2) ASTM D 2837, "Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials ", American Society for Testing and Materials, vol. 8.04.

(3) ASTM F 1473, "Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins ", American Society for Testing and Materials, vol. 8.04.

(4) PPI TR-4/99, "PPI Listing of Hydrostatic Design Bases and Maximum Recommended Hydrostatic Design Stresses for Thermoplastic Materials ", Plastics Pipe Institute, Inc., 1999.

(5) Brown, N. and Lu, X., 13th Plastic Pipe Symposium, A.G.A., San Antonio, TX.

(6) Brown, N., 14th Plastic Pipe Symposium, A.G.A., San Diego, CA.

(7) ASTM D 3350, "Standard Specification for Polyethylene Plastics Pipe and Fittings Materials ", American Society for Testing and Materials, vol. 8.03.

(8) ASTM D 2513, "Standard Specification for Thermoplastic Gas Pressure Pipe, Tubing and Fittings ", American Society for Testing and Materials, vol. 8.04.

(9) ASTM D 1693 "Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics ", American Society for Testing and Materials, vol. 8.02.