TECHNICAL REFERENCE ON THE PHYSICAL, MECHANICAL, and CHEMICAL PROPERTIES OF POLYAMIDE 12 (PA12) FOR HIGH PRESSURE GAS DISTRIBUTION APPLICATIONS

Technical Reference Report
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EXECUTIVE SUMMARY

Since their introduction during the 1960's, the use of PE plastic piping materials has grown at an exponential rate. Their benefits have been clearly established: coupled with its relative ease of use, plastic piping materials eliminate the need for costly long-term corrosion control measures and the associated monitoring costs.

The design and construction of plastic piping systems are governed by Title 49, Part 192 of the Code of Federal Regulations, which establish the minimum requirements for the safe use of plastic piping systems. In particular, sections 192.121 and 192.123 prescribe procedures for determining the design pressure of thermoplastic pipe and its design limitations. Section 192.121, Design of Plastic Pipes, defines the formula used for computing the design pressure. Section 192.123, Design Limitations of Plastic Pipe, limits the maximum pressure of plastic pipe to 125 psig – per latest rule change announced June 2004. As a result, there exists a desire on the part of utilities to leverage the benefits of thermoplastic piping materials and extend them to increased pressure ranges and larger diameters without sacrificing flow capacity.

One promising family of thermoplastic materials is Polyamide materials. Since 1997, GTI has sponsored research to evaluate the technical feasibility for the use of Polyamide 11 (PA11) material at increased pressures. The cumulative results of both laboratory experiments and field evaluations have amply demonstrated PA11's ability to operate at pressures up to 200 psig for 2-inch IPS SDR 11 pipe sizes, as evidenced by the recent successful installations at various location throughout the United States. The installations took place under approved waivers for pressures above 125 psig and with the use of a 0.40 design factor.

While PA11 appears to be a promising candidate material, there are several limitations including the fact that the PA11 piping material cannot be supplied cost effectively in larger diameter sizes. Hence, there is significant interest on the part of the gas utility companies to identify alternate candidate materials for high pressure applications and larger diameters which will not adversely affect capacity considerations.

Through the support of the GTI Operations Technology Development program and resin suppliers, a comprehensive program has been established to perform testing and evaluation of Polyamide 12 (PA12) material. Specifically, to validate the technical feasibility for the use of Polyamide 12 (PA12) pipe at higher operating pressures and larger diameters through a series of laboratory and field experiments focused on the development of comprehensive physical properties and critical construction, maintenance, and operating considerations data.

This report presents a comprehensive summary of the testing and evaluation (short term and long term properties) to date for the UBE, Degussa, and EMS Grivory PA12 materials. The results of the testing demonstrate that PA12 from the various resin suppliers appears to a be a very promising candidate material for high pressure gas distribution applications.

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1.1 Polyamides

Polyamide 12 is a thermoplastic belonging to the general class of polymers called polyamides. Polyamides are characterized by methylene groups of various lengths joined by amide linkages. The general formula for polyamides like Polyamide 12 is:

$$[NH\ (CH_2)_x\ CO]_n$$

Polyamides are named by the number of carbon atoms in the monomer unit.

In general, polyamides are produced by polycondensation using one of three monomer types. Polyamides can be produced from mixtures of diamines and diacids, from lactams or from amino acids. Polyamide 6.6, 6.10, 6.12 and 12.12 are examples of polyamides produced from diacids and diamines. Polyamide 6 and Polyamide 12 are produced from caprolactam and lauryl lactam respectively. In each case, the polymer is named for the number of carbon atoms in the monomer. For example, the monomer for Polyamide 11, undecanoic amino acid is:

$$NH_2\ (CH_2)_{10}\ COOH$$

Polyamides produced from diacids and diamines are named for the number of carbon atoms in each of the monomers. The diamine is listed first. For example, Polyamide 612 is produced from hexamethylenediamine, a 6 carbon diamine, and dodecandioic acid, a 12 carbon diacid. Each of these types of polyamides are homopolymers.

Copolyamides are also available. Convention denotes copolyamides by separating the monomers with a slash. For example, the copolymer of caprolactam, a 6 carbon monomer and lauryl lactam, a 12 carbon monomer is designated Polyamide 6/12.

1.2 Polyamide 12

The development of Polyamide 12 was started in the 1960's. The first commercial production of Polyamide 12 began in the 1970's at what is now Degussa in Marl, Germany. At the present there are four commercial suppliers of Polyamide 12 worldwide:

- Degussa AG – Marl, Germany
- UBE Industries, Ltd. – Tokyo, Japan
- EMS-Grivory – Domat, Switzerland
- Arkema – Paris, France

The monomer for Polyamide 12 is laurolactam. Laurolactam is produced from the trimerization of butadiene and several subsequent steps. Butadiene is a by product of the petroleum refining process.

Laurolactam is polymerized in a two step process. First, the lactam ring is hydrolyzed at high temperatures and pressures. In the second step, the molecular weight of the oligomer produced in the first stage is increased to the desired value. The second step is similar to the production of polyamides from an amino acid. Typical number average molecular masses for commercial grades of Polyamide 12 are in the range 15,000 to 40,000. Commercial grades of Polyamide 12 are typically stabilized against thermal oxidative and UV degradation by incorporating a suitable stabilizer package in a post-polymerization compounding step. The chemical formula for Polyamide 12 is:

$$[\mathrm{HN(CH_2)_{11}CO}]_n$$

1.3 Polyamide 12 Properties

The presence of amide groups in the polymer backbone are the characteristic that gives polyamides their unique property profile. The amide group is characterized by the following formula:

$$-\!\!\!-(\mathrm{NHCO})-\!\!\!-$$

The frequency of occurrence of the amide groups (amide density) differentiate between specific polyamides.

Due to the presence of the amide group and amide density, polyamides exhibit varying degrees of polarity. As a consequence, polyamides exhibit interchain and intrachain hydrogen bonding. The presence of hydrogen bonds contributes to the overall strength, flexibility and toughness of polyamides. Additionally, the presence of polar sites within the polyamide molecule affects the moisture absorption characteristics.

The rate of moisture absorption and the amount of moisture absorbed at equilibrium is determined by the amide density. Moisture absorption in polyamides has the effect of increasing the overall toughness and increasing flexibility. The effect of moisture in the solid state is reversible.

Table 1 presents a physical property comparison between rigid grades of Polyamide 12 and Polyamide 11.

Property	PA12	PA11
Specific gravity	1.01	1.03
Melting point, F	356	374
Tensile stress @ yield, psi	6670	5220
Elongation @ yield, %	6	22
Tensile strength, psi	9280	9860
Elongation, %	250-300	360
Flexural modulus, psi	210,000	184,000
HDT @ 264 psi, F	122	117
Coefficient of thermal expansion, 10^{-5} in/in-F	11	8.5
Surface Resistivity, ohm	10^{14}	10^{14}
Moisture content, equilibrium, %	1.5	1.9

Table 1: Comparison of typical physical properties of the Polyamide materials

In the late 1970's, The Australia Gas Light Company (AGL) identified the need to rehabilitate corroded cast iron service lines in New South Wales, Australia. At the time, polyamide 11 was identified as a candidate material for this application due to a combination of high strength, excellent toughness and resistance to chemical degradation.

It was found that the use of polyamide 11 allowed AGL to conveniently line the corroded cast iron pipe with a thin walled PA 11 pipe without compromising the operating conditions of the system. A development program was initiated by AGL to develop a Polyamide 11 system suitable for rehabilitation.

During the early 1980's, a project was initiated to rehabilitate cast iron mains in Sydney with a Polyamide 11 solvent bonded system operating at low pressures. Concurrently, a program was initiated to introduce polyamide systems, up to pipe sizes of 110 mm, for new and replacement gas distribution systems operating at pressures up to 30 psig (210 kPa). As a result of the success of Polyamide 11 systems in the 1980's' a project was initiated to rehabilitate the entire low pressure cast iron pipe system in Sydney in 1988. The new polyamide system was designed to operate at 30 psig (210 kPa) with a future supply capacity of three times the existing load.

In the mid eighties, AGL identified polyamide 12 as an alternative to polyamide 11 due to economic benefits and flexibility of supply.

In 1987, the Australian standards AS 2943, *"Plastics Pipes and Fittings for Gas Reticulation – Polyamide Compounds for Manufacture"* and AS 2944 , *"Plastics Pipes and Fittings for Gas Reticulation – Polyamide, Part 1 –Pipes, Part 2 –Fittings"* were developed. The standards outline the requirements for polyamide materials and pipe and fittings produced from polyamide materials operating at pressures up to 58 psig (400 kPa).

In the 1990's, polyamide distribution systems operating up to 58 psi (400 kPa) were installed in Poland and Chile.

In 1995, an evaluation was completed on a Polyamide 12 grade from UBE Industries, Ltd. The evaluation demonstrated that UBE PA12 was in compliance with the relevant Australian standards and was suited for the intended applications at lower costs.

Since 1991, the total consumption of polyamides for gas reticulation has been approximately 120 Mt/year. Approximately 50% of the total volume of pipe installed is Polyamide 12 . Most typically, 32 mm SDR 25 Polyamide 12 pipe is installed. Based on an annual volume of approximately 60 Mt/year, this translates to annual installed lengths of approximately 500 km/yr (approximately 300 miles/year).

Installation of polyamide pipe for gas distribution continues at AGL today. Approximately 80% of the distribution mains currently in service operate with a polyamide pipe installed by insertion.

Through extensive research performed at Agility Management Pty. Ltd. (Technical and Development Section) in Australia and through approximately 10 years of positive field service performance, Polyamide 12 has proven to be a viable candidate material for gas distribution systems.

1.4 Referenced Standards for Polyamide 12 Materials

The following standards are either approved or under development to allow the use of Polyamide 12 in natural gas distribution systems.

ASTM D 2513-04a Annex 5, *"Supplemental Requirements for Gas Pressure Pipe and Fittings Produced from Polyamide Material"*

AS 2943, *"Plastics Pipes and Fittings for Gas Reticulation – Polyamide Compounds for Manufacture"*

AS 2944 , *"Plastics Pipes and Fittings for Gas Reticulation – Polyamide, Part 1 –Pipes, Part 2 –Fittings"*

ISO 15439 Parts 1-6, *"Plastics piping systems for the supply of gaseous fuels under pressure up to 0.4 MPa (4 bar)*

ISO 22621 Parts 1-6, *"Plastics piping systems for the supply of gaseous fuels under pressure up to 2 MPa (20 bar)*

Section 2
Characterization of Mechanical, Physical, and Chemical Properties

Title 49, Part 192 of the Code of Federal Regulations governs the minimum requirements for the safe use of plastic piping systems. In particular, sections 192.121 and 192.123 prescribe procedures for determining the design pressure of thermoplastic pipe and its design limitations. Section 192.121, Design of Plastic Pipes, defines the formula used for computing the design pressure. Section 192.123, Design Limitations of Plastic Pipe, limits the maximum pressure of plastic pipe to 125 psig – as per the latest rule change announced in June 2004. In addition, through reference, Part 192 requires that all thermoplastic piping materials suitable for use in gas distribution applications must conform to the requirements contained within ASTM D2513-98[1] specification entitled *"Standard Specification for Thermoplastic Gas Pressure Pipe, Tubing, and Fittings"* [1]. Within the main body of the ASTM D2513, there are requirements that are applicable to all thermoplastic materials. Additional requirements are contained within Annexes specific to each respective thermoplastic material, e.g. PE materials are in Annex A1, PA11 and PA12 materials are in Annex A5, etc.

In order to demonstrate conformity to ASTM D2513-98 requirements and its applicable Annexes, GTI performed comprehensive testing and evaluation of the PA12 pipe materials supplied by the various PA12 resin suppliers including UBE (Japan), Degussa (Germany) and EMS (Switzerland). Arkema (France) is the fourth supplier of PA12; however, they did not participate in the program due to commercial considerations. The results are summarized in the sections to follow. It is important to note that throughout the body of this text, there are several comparisons made to PE piping materials in order to provide additional insight into the discussions. However, given its increased pressure carrying capabilities, as compared to PE, PA12 is intended to provide a cost-effective alternative to the use of steel piping.

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[1] Per the rule change issued during May 2004, and effective July 2004, the previous specified ASTM D2513-96a has been changed to ASTM D2513-98

2.1 Minimum Hydrostatic Burst Pressure (Quick Burst)

The minimum hydrostatic burst pressure, commonly referred to as quick burst, is obtained through testing in accordance with ASTM D1599 entitled *"Standard Test Method for Short-Time Hydraulic Failure Pressure of Plastic Pipe, Tubing, and Fittings"* [2]. This particular test method includes guidelines for determining the hydraulic pressure necessary to produce a failure within 60 to 70 seconds. While the results of the test are a useful measure of the ultimate strength of the material, they are not indicative of the long term strength or durability of the resin or pipe.

Five specimens approximately 18 inches in length, were measured and conditioned in a liquid bath at 74°F for over 1 hour and then filled with water and submerged in a water bath at 73°F. The pressure was then increased uniformly until each of the specimens failed. Based on these pressures, the hoop stress at failure for each specimen is calculated as follows:

$$S = \frac{p(D-t)}{2t} \tag{1}$$

where:

S = hoop stress, psi
p = internal pressure, psi
D = average outside diameter, in.
t = minimum wall thickness, in.

The results of the testing are summarized in Table 2 below.

PA12 Suppliers	2 inch PA12 SDR 11 Pipe		
	Avg. Burst Pressure (psig)	Avg. Hoop Stress (psi)	Failure Mode
UBE	1432	6867	Ductile
Degussa	1429	6899	Ductile
EMS	1318	6589	Ductile

Table 2: Summary of the quick burst data for PA12 pipe from each resin supplier

Based on the results of the testing, the PA12 pipe supplied from each of the respective PA12 resin suppliers exceed the hoop stress requirements stated in ASTM D2513-98 Annex A5 of 3900 psi.

2.2 Tensile Strength Determination

Tensile properties for the PA12 material were obtained utilizing ASTM D638 entitled *“Tensile Properties of Plastics”* [3]. This particular test method includes determining the tensile properties of plastics by performing tests on standard specimens under controlled conditions of specimen preparation, temperature, humidity, and testing machine speed.

During this particular study, six samples from each respective PA12 resin supplier were die-cut in the form of “Type I” specimens, as shown in Figure 1 under the specifications provided in Table 3.

Dimensions	Type I, mm (in.)	Tolerances, mm (in)
W – width of narrow sections	13 (0.50)	± 0.5 (0.02)
L – length of narrow sections	57 (2.25)	± 0.5 (0.02)
WO – width overall	19 (0.75)	± 6.4 (0.25)
LO – length overall	165 (6.5)	No max
G – gage length	50 (2.00)	±0.25 (0.010)
R – radius of fillet	76 (3.00)	± 1 (0.04)
D - Distance between grips	115 (4.5)	± 5 (0.2)

Table 3: Dimensional requirements for Type I specimens prescribed under ASTM D638 Test Method for Tensile Properties of PA12

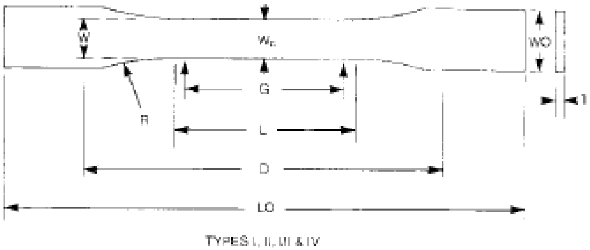


Figure 1: Schematic for Type I specimens prescribed under ASTM D638 test method for tensile properties

Six specimens from each of the PA12 suppliers were conditioned at 74 ºF and 50% relative humidity for 48 hours prior to testing. Measurements were taken for the width and the thickness for each of the specimens and placed in the grips of the testing machine. The testing machine speed was 2 inch/min, and the tensile strength at yield and break and the elongation at yield and break were obtained. The results of the testing are summarized in Table 4 below:

PA12 Suppliers	2-inch PA12 SDR 11 pipe – Die Cut Type I Specimens per ASTM D638 Test Method			
	Avg. Tensile Strength at Yield (psi)	Avg. Elongation at Yield (%)	Avg. Tensile Strength at Break (psi)	Avg. Elongation at Break (%)
UBE	6607	10	7776	254
Degussa	5370	12	6457	219
EMS	5790	5	6928	190

Table 4: Summary of the tensile strength properties for PA12 pipe

The results of the testing conform to expectations and are within the requirements of ASTM D2513 Annex A5.

2.3 Flexural Modulus

A second means of quantifying the tensile properties includes the determination of the flexural modulus of PA12 pipe; specifically, the stiffness. Five specimens from each of the three lots of pipe were tested in accordance with ASTM D790 entitled *"Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials"* [8].

Standard flexural specimens were die cut from both the UBE and Degussa pipe samples. Since the wall thickness of the pipe is closest to 1/4 inch, the dimension for 1/4 inch thick specimens were used. The specimen width was ºƒ½ inch and the specimen length was 5 inches. The specimen thickness was equal to the pipe wall thickness for 2 inch SDR11 pipe.

ASTM D790 Method I was used for all testing, which is a three point bend of the sample. The span between fixed supports was 4 inches. The strain rate for testing was 0.1 inches per minute. Samples were conditioned for a minimum of 48 hours at 74°F and 50% relative humidity prior to testing. All testing was preformed at 74°F and 50% relative humidity.

For the tests, each specimen was measured prior to the test. The specimen width and depth were recorded. The sample was then placed in the test jig and centered between the fixed supports. The moving support travels down into the specimen at a fixed rate of 0.1 inches per minute. The tangent modulus was recorded and reported. The tangent modulus is defined as the slope of the steepest linear portion of the load deflection curve. These flexural modulus data are summarized in Table 5 for each of the PA12 suppliers product. This data is consistent with the requirements of ASTM D2513.

PA12 Supplier	Flexural Modulus
UBE	231.6 ksi
Degussa	213.6 ksi
EMS	173 ksi

Table 5: Summary of the flexural modulus data from the various PA12 suppliers

2.4 Apparent Tensile Strength Determination

Additional tensile property measurements for the PA12 materials were obtained utilizing ASTM D2290 entitled *"Apparent Tensile Strength of Ring or Tubular Plastics and Reinforced Plastics by Split Disk Method"*. This particular test method includes determining the comparative tensile strength of plastics by performing tests on split disks under controlled conditions of specimen preparation, temperature, humidity, and testing machine speed [9].

During this particular study, six samples from each of the three lots of pipe material were prepared per ASTM D2290 specifications, as shown in Figure 2 under the testing specifications provided in Table 6.

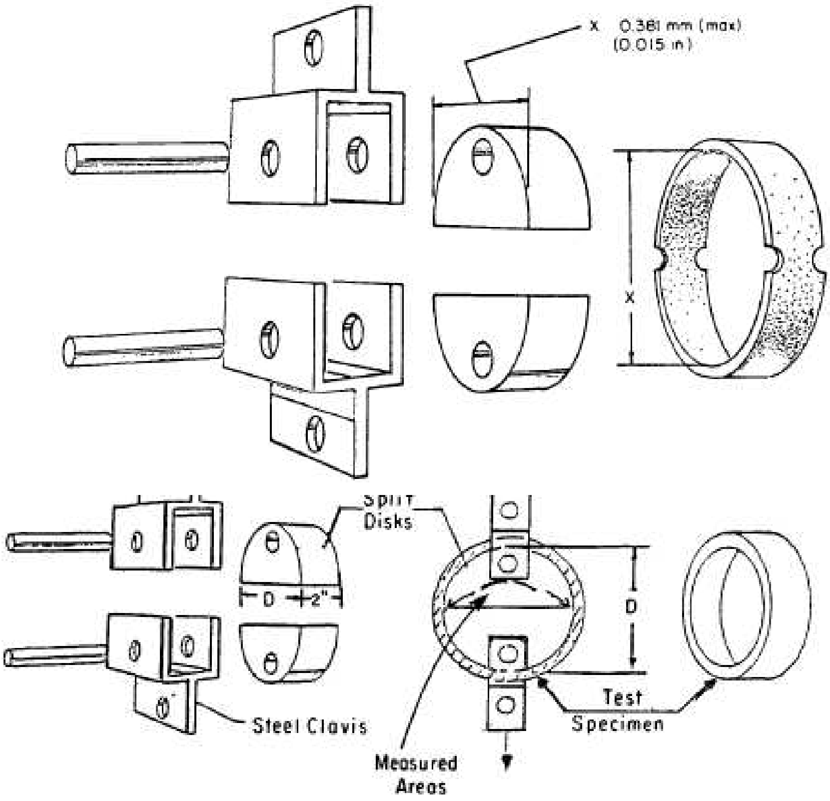


Figure 2: Schematic illustration of the split ring tensile specimen and the test fixture (Taken from ASTM D2290 Specification)

Parameter	Value
Conditioning Temperature	74F
Relative Humidity	50%
Specimen Thickness	0.50 inches
Reduced Wall Thickness	0.250 inches
Test Speed	0.5 in./min

Table 6: Dimensional requirements for Split Ring specimens per ASTM D2290 Test Method for Tensile Strength Properties

Each of the six specimens from both the UBE and Degussa PA12 pipes were conditioned at 74 ºF and 50% relative humidity for 48 hours prior to testing. Measurements were taken for the width and the reduced sections for each of the specimens. The specimens were then placed in the test fixture of the testing machine, as shown in Figure 3. The testing machine speed was set equal to 0.5 in./min. The tensile strength at yield and break and the elongation at yield and break were obtained. The results of the testing are summarized in Table 7 below.

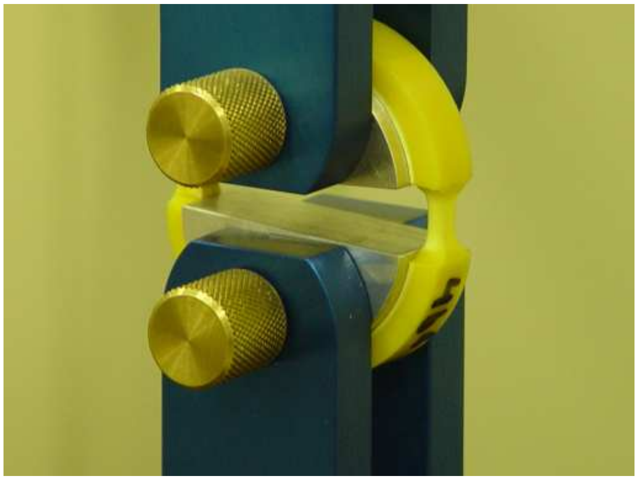


Figure 3. Apparent Tensile strength determination testing for PA12 pipe specimens

As per ASTM D2513-98 Annex A5, the minimum apparent tensile strength at yield shall be greater than 3900 psi. As with the hydrostatic quick burst results, the tensile strength at yield for each of the PA12 supplier's product was two times the requirement.

PA12 Supplier	Avg. Apparent Tensile Strength at Yield (psi)
UBE	6972
Degussa	7086

Table 7: Apparent tensile strength at yield for various PA12 resin suppliers

These data not only provide corroboratory guidance of a material's resistance to circumferential stress, but more importantly, they provide for a control in comparing the effects of exposure to various chemical reagents as discussed in the next section.

2.5 Chemical Resistance Testing

In order to determine the effectiveness of plastic piping material to withstand certain types of chemical attack, laboratory testing was performed in accordance to ASTM D2513, which lists five chemicals agreed upon by industry consensus and testing according to ASTM D543 *“Standard Test Method for Resistance of Plastics to Chemical Reagents”* [10].

This particular test method includes determining the comparative apparent tensile strength of specimens by performing tests on split disks under controlled conditions of specimen preparation, temperature, humidity, and testing machine speed, and exposure to prescribed chemical reagents. This method includes provisions for measurement of changes in weight, dimension, appearance, and strength properties. It is important to note there are certain limitations to this particular type of testing and the correlation of the results to actual field exposure. In particular, the choice and types of reagents and its respective concentration, duration of immersion, and the temperature at test are critical parameters that can have a significant effect. Furthermore, the effect of stresses on various types of polymers in contact with environmental agents can also have a significant effect and should be taken into account. These issues are not addressed in this study.

ASTM D2513 specifies five industrial chemical reagents shown below in Table 8 with the specified concentration levels.

Chemical Reagent	Concentration (% by Volume)
Mineral Oil	100
Tertiary Butyl Mercaptan (TBM)	5 in Mineral Oil
Methanol	100
Ethylene Gylcol	100
Toluene	15 in Methanol

Table 8: Description of the various chemical reagents for determining the chemical resistance properties of PA11 per ASTM D2513

Testing was performed on five (5) split ring specimens obtained from extruded pipe with the same specifications used to determine the apparent tensile properties, see Figure 2. Each specimen was initially weighed and completely immersed in the respective solutions for 72 hours prior to the start of the testing. Upon removal, the specimens were carefully wiped clean of excess chemical and allowed to air dry for approximately 2.5 hours and then reweighed. Both initial and final weights were recorded. The specimens were tested within one-half (1/2) hour of weighing in accordance to the testing methodology. The speed of testing was equal to 0.5 in./min., equal to that of the apparent ring tensile strength measurements discussed earlier.

ASTM D2513 and Annex A5 specifies the maximum percent change in both weight and tensile strength properties for PA11, as shown in Table 9. Given that the PA12 is analogous to the PA11 material and of the same family of Polyamide materials, the results of the testing were compared to the PA11 under Annex A5 for comparative purposes.

	Polyamide 11 (PA11)	
Chemical	Change in Weight (%)	Change in Tensile Yield Strength (%)
Mineral Oil	< 0.5	- 12
Teritary Butyl Mercaptan	< 0.5	- 12
Methanol	< 5	- 35
Ethylene Gylcol	< 0.5	- 12
Toluene	< 7	- 40

Table 9: Allowable change in both percent weight and apparent tensile strength at yield per ASTM D2513 for PA11

It is important to note that the allowable percent change in weight and apparent yield strength for PA11 appears to be relatively large as compared to polyethylene. Per ASTM D2513, pipe, tubing, and fittings made from polyethylene shall not increase in weight more than 0.5% (1.0% for toluene in methanol) and the percent change in the apparent yield strength shall not decrease more than 12%. In contrast, PA12 pipe has relatively larger tolerances due to its inherent material and chemical characteristics, as discussed in Section 1.

Overall, the results of the testing indicate that the PA12 material from both UBE and Degussa compared well with the established PA11 specifications – consistent with expectations. The data is summarized in Tables 10 and 11 for each of the respective PA12 suppliers.

UBE PA12 Split Ring Specimens for Chemical Resistance Testing			
Reagent	Change in Weight (%)	Tensile Strength at Yield (psi)	Change in Tensile Strength at Yield (%)
Control	----	6972	----
Mineral Oil	0	6954	0
Toluene in Methanol	2.3	5070	-27
Methanol	2.3	4795	-31
Ethylene Glycol	0	7041	-1
Tertiary Butyl Mercaptan	0	7017	-1

Table 10: Summary of the chemical resistance testing data for UBE PA12 pipe

Degussa PA12 Split Ring Specimens for Chemical Resistance Testing			
Reagent	Change in Weight (%)	Tensile Strength at Yield (psi)	Change in Tensile Strength at Yield (%)
Control	----	7086	----
Mineral Oil	0	7148	+1
Toluene in Methanol	2.8	6219	-12
Methanol	2.5	5641	-20
Ethylene Glycol	0	6704	-5
Tertiary Butyl Mercaptan	0	6198	-12

Table 11: Summary of the chemical resistance testing data for Degussa PA12 pipe

From Tables 10 and 11, it can be seen that the most significant reduction in tensile strength occurred under exposure to methanol and toluene in methanol. This is as expected given that methanol is a polar solvent. From fundamental chemistry, polar solvents tend to have a chemical affinity to polar materials. For this reason, while there is a strength reduction under exposure to methanol (polar solvent), there is minimal strength reduction under the influence of heavy hydrocarbons (non polar). For this reason, Polyamides (11 and 12) offer an attractive alternative to the use of PE piping materials in areas contaminated by heavy hydrocarbons including gasoline.

2.6 Melt Characteristics

Differential scanning calorimetry (DSC) is a useful tool to measure several fundamental properties of organic, inorganic, and metallic materials. DSC measures the thermal transitions of these materials between –50° and 700°C. In particular, properties such as heat of fusion, melting point, glass transition temperature, heat capacity, purity, and the degradation or decomposition temperatures can be obtained. Because structural features in the various materials can be readily identified by any of these properties, the results may be correlated to potential service life.

The key property of interest for this study is the melting point of polyamide 12. All three lots were tested to determine their melting points. Measurement of the melting point of the pipe was performed in accordance with ASTM D 3418 [14]. A 12.0 mg sample size was tested using 350°C at 10°C/min. The results of the testing are summarized in Figures 4-6 for both UBE and Degussa pipe specimens, respectively.

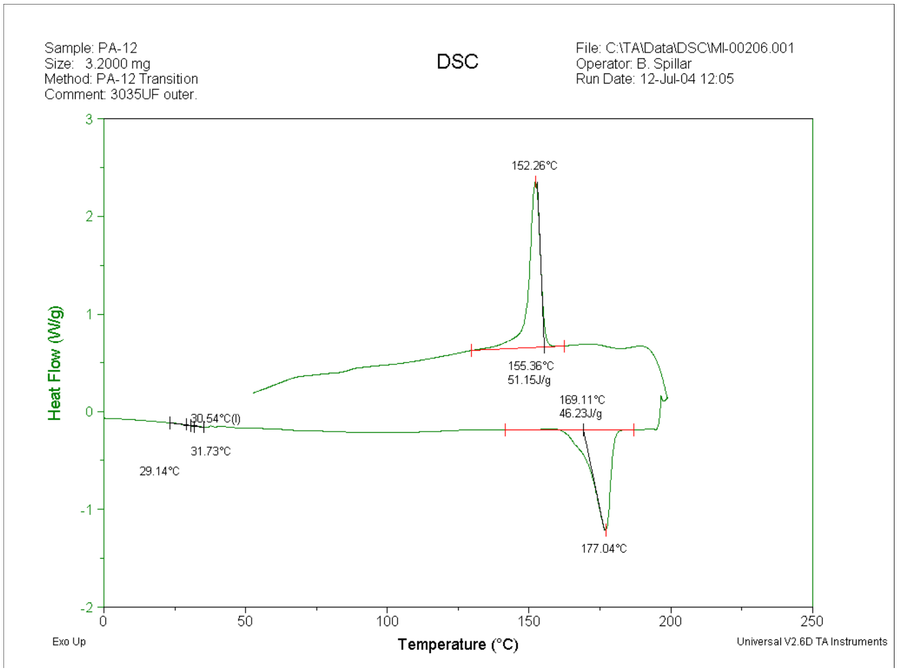


Figure 4: Melt point index for the UBE PA12 pipe taken from the outer surface

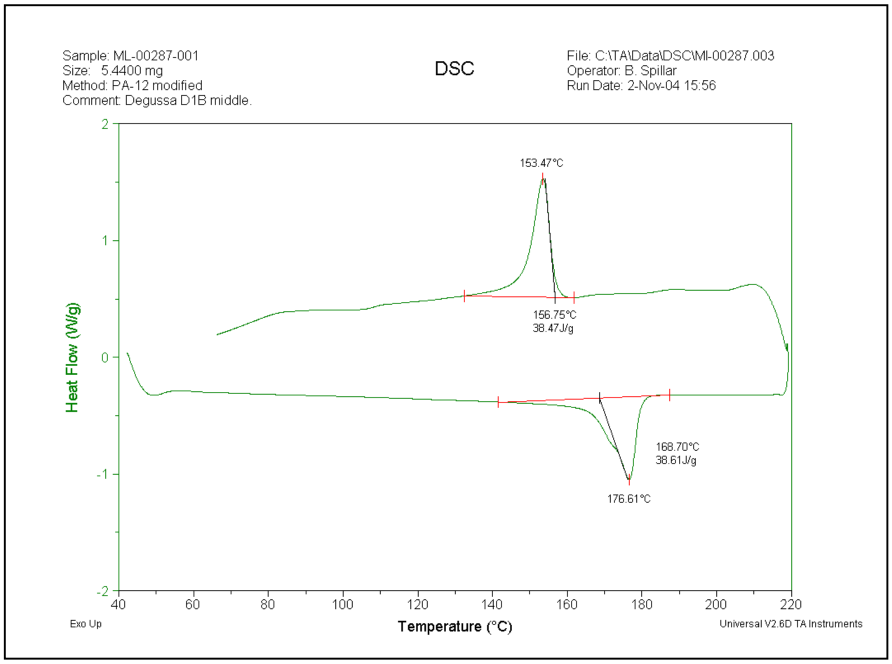


Figure 5: Melt point index for the Degussa PA12 pipe taken from the middle of the pipe wall

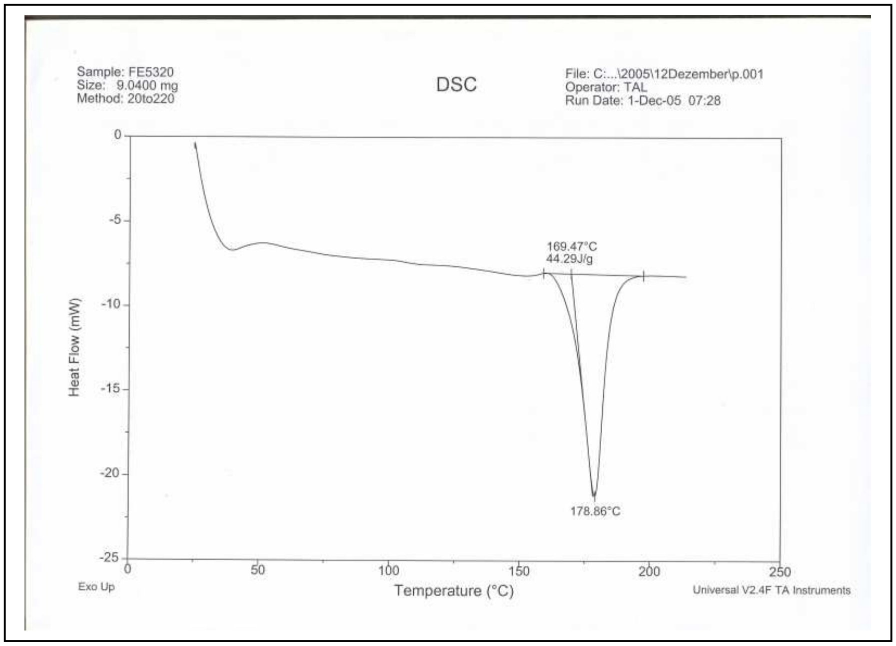


Figure 6: Melt point index for the EMS PA12 pipe (Courtesy EMS GRIVORY)

2.7 Summary

The cumulative results of the various short term testing used to characterize the mechanical, chemical, and physical properties of PA12 indicate that the material conforms to the requirements of ASTM D2513 and its respective Annexes. Specifically, the material meets and/or exceed the requirements and compares well with the PA11 requirements.

On the basis of this test data, it can be readily inferred that both the PA11 and PA12 should be within the same Annex within ASTM D2513 given the similarities in the performance criterion.

Section 3

Characterization of Long Term Performance Testing

The preceding discussion has been focused on performing short-term quality control type testing as specified in ASTM D2513-98 to characterize the mechanical and physical properties for PA12 and failures that occur in the “ductile” mode. However, with all plastics, the strength and durability can vary significantly with the time of loading, temperature, and environment. Plastics are very complex combinations of elastic and fluid like elements and they exhibit properties shared between those of a crystalline metal and a viscous fluid – viscoelasticity.

Because of this viscoelastic behavior, conventional hydrostatic quick burst and short-term tensile tests, as discussed in Section 2.1 and 2.2, respectively, of this report, cannot be used to predict long-term performance of plastics under loading. When a plastic is subjected to a suddenly applied load that is then held constant, it deforms immediately to a strain predicted by the stress-strain modulus. It then continues to deform at a slower rate for an indefinite period. If the stress is large enough, then the rupture of the specimen will eventually occur. This particular time dependent viscous flow component of deformation is known as creep, and the failure that terminates it is known as creep rupture.

As the stress levels decrease, the time to failure increases and material deformation becomes smaller. At very long times to failure, deformation is usually less than 5% for most thermoplastics. The fracture is then a result of crack initiation and slow crack growth (SCG). A large body of previous GTI sponsored research and empirical observations in the field indicates that this type of “brittle” failure, not the excessive deformation, is the ultimate limit of the long-term performance of plastic pipe in service. Failures in the ductile mode also may potentially occur, but only in operating conditions where the pressure in service is accidentally increased.

As a result, there is an overwhelming need to conduct long-term testing to identify the longevity of the material when it fails in the brittle mode. This section outlines the test procedures used and the data which was developed to validate the PA12 materials' long term hydrostatic strength and data from other widely accepted tests to characterize the material's resistance to slow crack growth.

3.1 Determination of the Long Term Hydrostatic Strength

During the early 1960's, the Plastics Pipe Institute (PPI) proposed a new method for forecasting the long term strength of thermoplastic pipe materials. Soon after the industry adopted this method to stress rate their materials. In 1967, after the addition of some refinements, ASTM adopted the PPI proposal as ASTM D2837, *"Standard Method for Obtaining Hydrostatic Design Basis (HDB) for Thermoplastic Pipe Materials"*.

ASTM D2837 establishes a pipe material 's hydrostatic design basis (HDB) through empirical testing as outlined below: (Note: Interested readers are also referred to PPI TR3 documentation for a detailed description of submitting and performing the required testing to establish a materials' HDB. This is only intended to serve as a background of the approach used in D2837).

1. Hoop stress versus time-to-fail data covering a time span from about 10 to at least 10,000 hours are developed by conducting sustained pressure tests on pipe specimens made from the material under evaluation. The required test procedure is ASTM method D 1598, "Time-to-Failure of Plastic Pipe Under Constant Internal Pressure". The test is conducted under specified conditions of external and internal environment (usually water, air, or natural gas inside and outside the pipe) and temperature (generally 73^{o}F (23^{o}C) for ambient temperature design);

2. The resultant data are plotted on log hoop stress versus log time-to-fail coordinates, and the *'best-fit straight line'* running through these points is determined by the method of least squares;

3. Provided the data meet certain tests for quality of correlation, the least squares line is extrapolated mathematically to the 100,000 hour intercept. The primary assumption is that the empirical data for the first 10,000 hours will be linear through the 100,000 hour intercept. The hoop stress value at this intercept is called the long-term hydrostatic strength (LTHS);

4. Depending on its LTHS, a material is categorized into one of a finite number of HDB categories. For example, if a material has an LTHS between 1,200 and 1,520 psi (8.27 and 10.48 MPa), it is assigned to the 1,250 (8l62 MPa) psi HDB category. If its LTHS is between 1,530 and 1,910 (10.55 and 13.17 MPa) psi, it is placed in the next higher HDB category, 1600 psi (11.03 MPa). By the D 2837 system, the value of each higher HDB category is 25 percent above the preceding one. This preferred number categorization was selected to reduce the number of material strength categories and, thereby, simplify pressure rating standardization.

This is illustrated graphically in Figure 7 below:

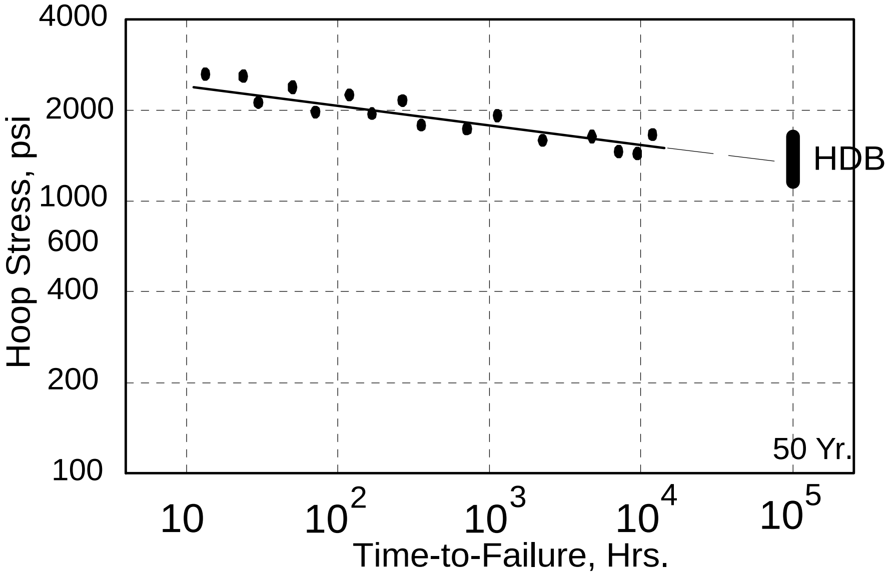


Figure 7: Determination of the HDB rating per ASTM D2837 method

Once the HDB for a particular pipe material has been determined, the MAOP of the system can be calculated as follows – note Equation (2) is a restatement of the equation prescribed in CRF Title 49, Part 192.121 [6]:

$$MAOP = \frac{2 \bullet HDB \bullet F}{SDR - 1} \tag{2}$$

where:

HDB = Hydrostatic Design Basis, psi
F = Design Factor, 0.32 for gas piping
SDR = Standard Dimension Ratio defined as the ratio of the mean outside diameter to the minimum wall thickness

At present, there are concurrent on-going efforts on the part of the various PA12 suppliers to establish the long term hydrostatic strength and the corresponding HDB ratings. Based on data to date, the UBE PA12 material has an established Experimental E-6 rating (after 6,000 hours of testing) of 3150 psi listed within the PPI TR-4. The testing is on-going and will continue to the 10,000 hours.

The most significant implication of this particular HDB rating is that the PA12 material can operate at pressures 25% greater than the PA11 piping material. Using a design factor of 0.32 in Equation (2), the PA12 piping system can operate at 200 psig as compared to 160 psig for the PA11 piping system. Using a design factor of 0.40, the PA12 piping system can operate at pressures up to 250 psig for SDR 11 pipe sizes.

3.2 Validation of the Hydrostatic Design Basis

Based on the preceding discussions, it is important to note that in applying the ASTM D2837 methodology, the fundamental assumption was that the stress versus time-to-fail line depicted by the first 10,000 hours is ***linear*** and will continue through at least 100,000 hours. If this is not the case and if there is a departure from linearity, the ASTM D2837 will yield an overestimate of a material’s actual long term hydrostatic strength, as shown in Figure 8.

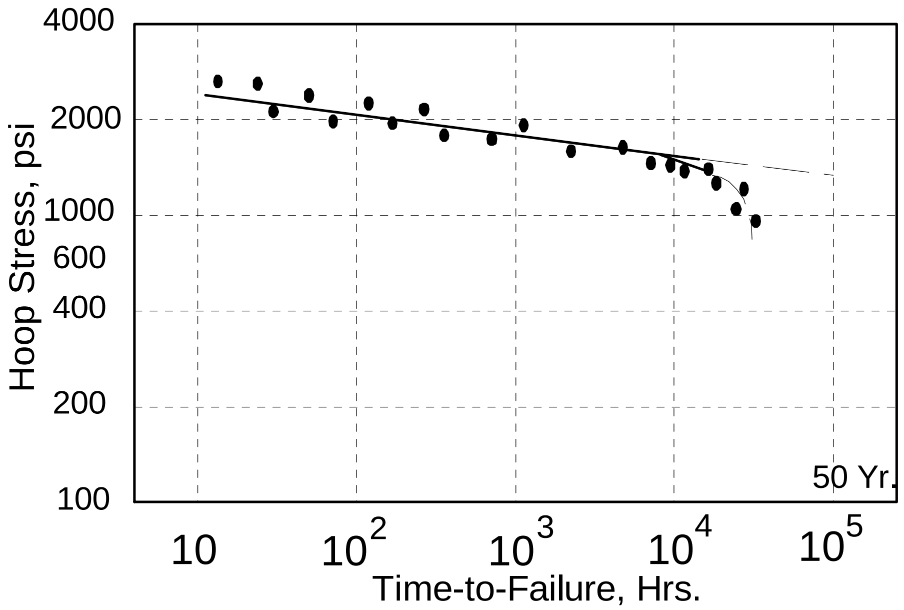


Figure 8: Departure from linearity used to establish the long term hydrostatic strength

By the late 1970's it was generally recognized that this assumption of linearity did not accurately reflect the actual long term performance of all plastic piping materials. Sustained pressure testing at time to failures greater than 10,000 hours indicated that for some plastic materials, there was a faster rate of regression beyond the 10,000 hours as compared to the initial stages of loadings. Furthermore, in the region of the faster rate of regression of strength the failures were brittle-like, the result of the transition from a ductile to the brittle-like SCG failure mechanism, as shown in Figure 9.

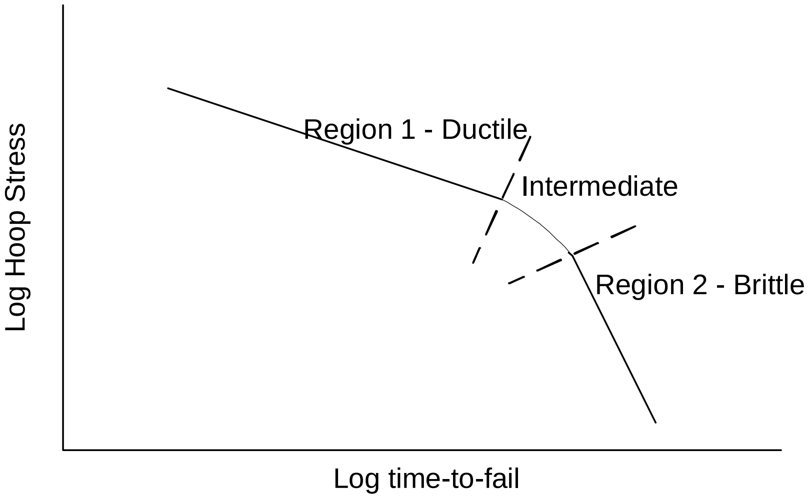


Figure 9: Illustration of transition from ductile to brittle failure mode

The real consequence of an overestimated LTHS was that it resulted from the unanticipated transition from a ductile to a SCG failure mechanism. And it was the SCG mechanism, and not unsatisfactory pressure strength, that accounted for the observed field failures. Thus, it was determined that the overwhelming design criterion was the nature of the failure mechanism and not merely the circumferential stress at which failure occurred.

By the mid-1980's changes began to be made to ASTM D2513, Standard specification for Thermoplastic Gas Pressure Pipe, Tubing and Fittings, that were intended to exclude materials that have inadequate resistance to SCG. The fundamental change required elevated temperature testing to validate the assumption that the straight-line behavior exhibited by the first 10,000 hours of testing under method D2837 shall continue through at least 100,000 hours. To enhance the efficacy of this proposed validation requirement, the rate process based requirement was added to ASTM D2837 for validating the 73°F HDB ratings for all PE pipe materials. Through the adoption of the validation requirement, the *window* in ASTM D2837, which allowed the selection of PE materials with less than adequate resistance to SCG, was closed. The net effect of this requirement ensured that only materials with sufficient ductile behavior were to be utilized in gas distribution applications – the central aspect in the safe and effective long term design of plastic piping systems. Table 12 presents the time, temperature, stress combinations which are utilized to validate the HDB ratings for PE materials.

From Table 12, for a given high density PE material with a HDB rating of 1600 psi, the 100,000 hour HDB can be validated using a stress value of 735 psi at 90°C for 70 hours. Alternatively, the 100,000 hour HDB can be validated using a stress value of 825 psi at 80°C for 200 hours.

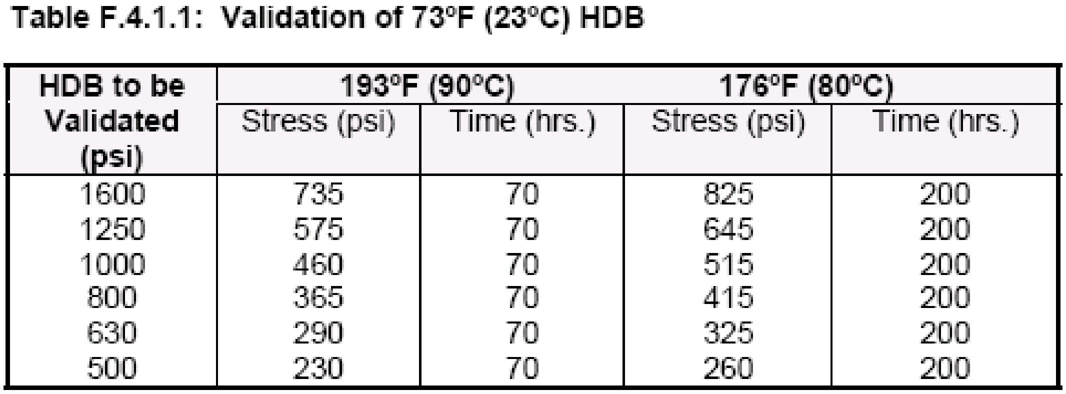


Table F.4.1.1: Validation of 73°F (23°C) HDB

HDB to be Validated (psi)	**193°F (90°C)**		**176°F (80°C)**	
	Stress (psi)	Time (hrs.)	Stress (psi)	Time (hrs.)
1600	735	70	825	200
1250	575	70	645	200
1000	460	70	515	200
800	365	70	415	200
630	290	70	325	200
500	230	70	260	200

Table 12: HDB validation requirement under PPI-TR3 policies

However, for the case of Polyamide materials, there are no such requirements in place. That is, the highest HDB value in Table 5 is for 1600 psi, which is considerably less than the projected HDB rating of 3150 psi. As a result, GTI performed analytical calculations using the bidirectional shift theory to develop acceptable time, temperature, and stress criterion, which would validate the linearity of the HDB data up to the 100,000 hour intercept.

In general, the bidirectional shift functions are a widely accepted technique to transfer data from a given time, temperature, stress state to another time, temperature, stress state through the use of the following formulas:

$$\begin{aligned} a_T &= \exp[0.109(T_t - T_s)] \\ t_s &= t_t a_T \end{aligned} \tag{1}$$

$$\begin{aligned} b_T &= \exp[-0.0116(T_t - T_s)] \\ p_s &= \frac{p_t}{b_T} \end{aligned} \tag{2}$$

Therefore, for example, to determine the appropriate values for the test time and stress at 80°C that correspond to a HDB rating of 1600 psi for 100,000 hours at 23°C, one can readily substitute the corresponding values into both Equations (1) and (2), as shown below.

$$\begin{aligned} a_T &= \exp[0.109(T_t - T_s)] \\ a_T &= \exp[0.109[80 - 23]] \\ q_T &= 499.2 \\ \\ \frac{t_s}{a_T} &= t_t \\ \frac{100{,}000}{499.2} &= t_t \\ t_t &= 200.3 \end{aligned} \tag{3}$$

and,

$$\begin{aligned} b_T &= \exp[-0.0116(T_t - T_s)] \\ b_T &= 0.516 \\ p_s &= \frac{p_t}{b_T} \\ p_t &= 825.9 \end{aligned} \tag{4}$$

The same methodology was then applied for the PA12 pipe specimens. Because there was insufficient data with respect to the HDB rating of the PA12 material, an estimated HDB rating of a of 2500 psi (minimum as a direct comparison to PA11) at 23°C was used as a first approximation in order to determine the appropriate test time and stress conditions at 80°C. From Equation (3) and (4), the calculated stress and time is 1290 psi for 200 hours to validate linearity to the 100,000 hour intercept for a HDB rating of 2500 psi. Similarly, using an estimated HDB rating of 3150 psi at 23°C to validate linearity up to the 100,000 hour intercept, the calculated stress and time are 1626 psi for 200 hours.

While the conditions stated above provide for assurances of linearity of up to 100,000 hours, ASTM D2513 requires additional substantiation of the linearity up to the 50 year intercept (438,000 hours). As a result, the calculated test time from Equation (3) is 877 hours for the particular HDB rating to be validated at 80°C.

Table 13 presents a summary of the test conditions for the particular validation and/or substantiation of interest. It is important to note, a similar analysis can be performed to obtain the appropriate time/stress combinations at a test temperature of 90°C.

HDB to be Validated (psi)	**Test Temperature = 80°C**			
	a_T = 499.2 b_T = 0.516			
	100,000 hours Validation		**50 year Substantiation**	
	Stress	**Time**	**Stress**	**Time**
1600	825	200	825	877
2500	1290	200	1290	877
3150	1626	200	1626	877

Table 13: Test critera for HDB validation/substantiation using bi-directional shift functions

It is important to note, the above conditions are based on analytical modeling using the same methodology applied to develop validation conditions for PE materials. In addition, there is a degree of uncertainty in that the constants contained within the bi-directional shift functions are empirically derived values for PE materials. These constants may be different for Polyamide materials; however, they have been applied here as a first approximation.

Initially, six specimens from both UBE and Degussa were tested at 1290 psi (258 psig test pressure) for a period of 875 hours at 80°C. There were no failures at these conditions for times greater than 1000 hours – see Table 14 below. The results of the testing demonstrated that the PA12 material could easily substantiate a 2500 psi rating at 23°C for a period of well over 50 years.

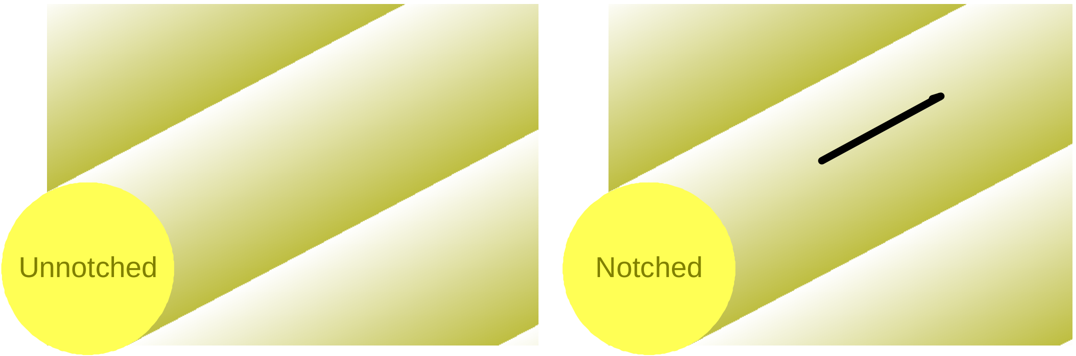
However, in order to conform to pending changes at the ISO level for Polyamide materials (PA11 and PA12) and noting the degree of uncertainty in the constants used in the bi-directional shift functions, and additional set of six specimens were tested at higher stress levels – 1450 psi (290 psig internal test pressure or 20 bar for SDR 11 pipe specimens). The results of the testing showed no failures at these conditions for times greater than 2000 hours providing additional assurances of 50 year substantiation for a projected minimum HDB rating of 2500 psi at 23°C. Testing at this level was performed on pipes supplied from all three pipe manufacturers (UBE, Degussa, and EMS).

3.3 Notched Pipe Testing

Notwithstanding the inclusion of the validation protocols with the ASTM D2837 test method, additional tests have been developed to characterize the effect of externally induced flaws on pipe and its resistance to failures by the SCG mechanism. One promising test includes ISO 13479 entitled *"Determination of resistance to crack propagation – Test Method for slow crack growth of notched pipe (notch test)"*. The importance of this test to characterize the SCG performance is under scored by the fact that the test specimens within ASTM D2387 do not contain any external flaws other than those introduced within the pipe manufacturing process.

The notched pipe test is analogous to the validation testing required under ASTM 2837 whereby actual pipe specimens are subjected to sustained pressure testing at elevated temperatures. However, the notched pipe test provides for intentionally introducing a controlled notch along the axial direction of the pipe specimens located 90° apart circumferentially. The notched pipe specimens are then subjected to constant internal pressure and the time to failure is recorded.

In order to gain a better understanding of the test protocol and its meaningfulness, consider the case of high density PE piping: the validation protocols within ASTM D2837 require that the pipe specimens must not fail prior to 200 hour test time at an applied stress of 825 psi (165 psig). In the case of the notched pipe test per ISO 13479, suitable SCG resistance is provided for when the notched pipe specimens do not fail prior to 165 hours at an internal pressure of 135 psig. Assuming that the pipe is not notched (100% of the wall thickness), the resulting applied stress is 676 psi. However, with the inclusion of a controlled notch that is 20% of the wall thickness, the calculated value of the applied stress at the location of the notch (remaining ligament) is 860 psi. This is significant in that the applied stress (860 psi) on the remaining ligament (20% wall loss) is greater than the stress used to validate the HDB rating (825 psi). This is illustrated in Figure 10 below.



Temp = 80℃
Internal Test Pressure = 165 psig
Test Stress = 825 psi
Time to Failure = 200 hours

Temp = 80℃
Internal Test Pressure = 135 psig
Test Stress (100% wall) = 676 psi
Time to Failure = 165 hours

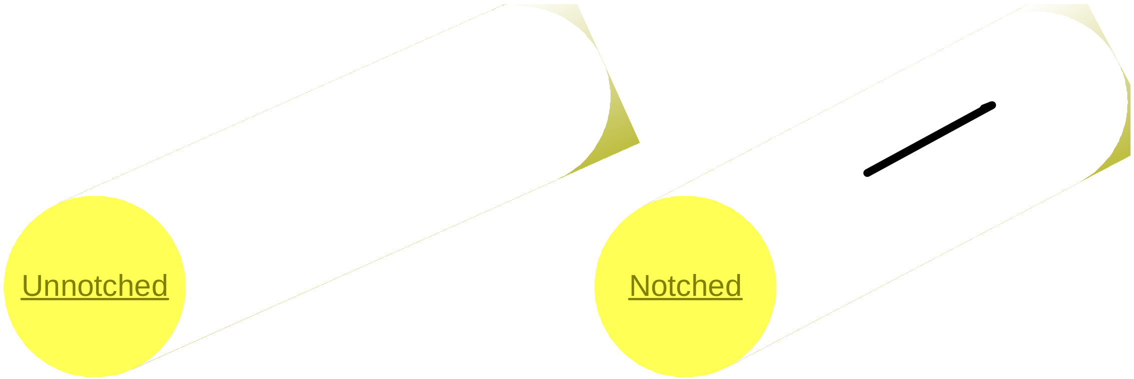
$$s = \frac{p(D-t)}{2t}$$

Temp = 80℃
Internal Test Pressure = 135 psig
Test Stress (80% wall) = **860 psi**
Time to Failure = 165 hours

Figure 10: Illustration of the ISO 13479 Notched Pipe Test Requirements to Characterize the SCG Resistance of HDPE materials

As with the HDB validation protocols, there are no test provisions for materials with increased HDB ratings greater than 1600 psi. As a result, suitable test conditions were established using practical considerations.

Under typical operating conditions, piping materials that contain damaged and scratched sections along the length of buried pipe are subjected to same internal pressure as pipe lengths, which do not have any damage. It stands to reason then, that the same internal test pressure should be used to evaluate pipe sections, which contain damage as compared to those sections that are pristine. This is illustrated in Figure 11 below.



Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress = 1450 psi
Time to Failure = 877 hours

Note: Time is for 50 yrs
Substantiation

Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress (100% wall) = 1450 psi
Time to Failure = 165 hours

Note: Current Time Requirements
Per ISO 13479

$$s = \frac{p(D-t)}{2t}$$

Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress (80% wall) = **1848 psi**
Time to Failure = 877 hours

Note:
Increased Stress/Time Conditions

Figure 11: Notched pipe test conditions for PA12 piping materials with 20% notch

Consequently, GTI performed comprehensive long term sustained pressure testing at the same conditions as the HDB validations protocols. Specifically, six pipe specimens from each of the three pipe manufacturers were tested at an internal pressure of 290 psig for a period of 877 hours at 80°C with a 20% axial notch located 90° apart in the circumferential direction. These conditions are not only representative of actual field conditions but also represent test conditions which are substantially greater that the 50 year substantiation requirements (Note: increased stress value of 1848 psi on the remaining ligament as compared to the stress value of 1450 psi assuming 100% wall). The results of the testing demonstrated that there were no failures in any of the pipe specimens tested after 2000 hours. The data is summarized in Table 14 below:

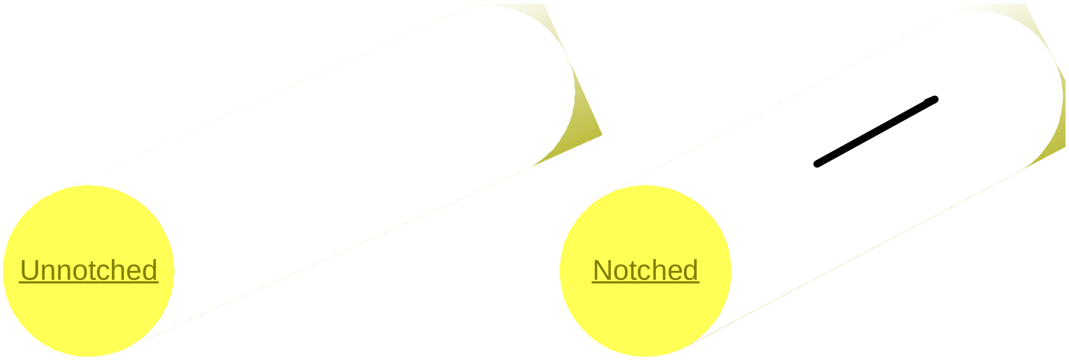
PA 12 Supplier	Test Criterion	Time to Failure (hrs)
UBE	Test Pressure: 290 psig (20 bars) Notch Depth: 20% Stress at remaining ligament: 1848 psi Test Temperature: 80°C 50-year substantiation time: 877 hours	> 2000
Degussa		> 2000
EMS		> 2000

Table 14: Notch pipe testing per ISO 13479 for PA12 pipe specimens

While the results of the testing were extremely positive given the significant degree of conservatism in the test stress conditions, additional tests were performed to examine the notch sensitivity of the PA12 material. Specifically, tests were performed using a 30% notch depth and 50% notch depth, which result in excessive circumferential stress states at the location of the remaining notch ligament. This is shown graphically in Figure 12 below.

Six specimens from the UBE PA12 pipes were subjected to long term sustained pressure testing with both a 30% notch and 50% notch and placed under an internal pressure of 290 psig at 80°C. The results of the testing showed no failures after 2000 hours with a 30% notch. With the pipe specimens containing a 50% notch, three of the six specimens failed in times less than 500 hours. It is important to emphasize that the 50% notch depth is a very unrealistic test condition. Regardless, even with the 50% notch, the PA12 had greater than expected time to failures. The results of the testing are summarized in Table 15 below.

The cumulative results of the notched pipe testing unequivocally demonstrate the excellent SCG resistance of the PA12 material given the strong degree of conservatism inherent in the test criterion.



Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress = 1450 psi
Time to Failure = 877 hours

Note: Time is for 50 yrs
Substantiation

Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress (100% wall) = 1450 psi
Time to Failure = 165 hours

Note: Current Time Requirements
Per ISO 13479

Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress (70% wall) = **2132 psi**
Time to Failure = 877 hours

Temp = 80℃
Internal Test Pressure = 290 psig
Test Stress (50% wall) = **3043 psi**
Time to Failure = 877 hours

Figure 12: Notch pipe testing criterion with 30% notch and 50% notch for SCG

Conditions	Test Conditions	Time to Failure (hrs)
Condition 1 (UBE PA12)	Test Pressure: 290 psig (20 bars) **Notch Depth: 30%** **Stress at remaining ligament: 1848 psi** Test Temperature: 80°C 50-year substantiation time: 877 hours	> 2000 hours with no failures
Condition 2 (UBE PA12)	Test Pressure: 290 psig (20 bars) **Notch Depth: 50%** **Stress at remaining ligament: 1848 psi** Test Temperature: 80°C 50-year substantiation time: 877 hours	> 500 hours with no failures of 3/6 specimens

Table 15: Notch pipe testing of UBE PA12 pipe specimens with at 30% and 50% notch depth

3.4 PENT Testing

In addition to the validation protocols and the notched pipe testing described previously, another relative index of a materials' resistance to SCG is the PENT time to failure data. It is important to emphasize that the PENT test is a useful quantitative index of a plastic piping materials' resistance to SCG for comparative purposes. The data does not provide for an accurate value for the predicted life, i.e., the data does not correlate to any performance considerations such as long term performance under constant stress.

A small controlled notch is introduced into a compression-molded plaque and is subjected to a uni-axial stress. The specimens are then tested to failure at 80°C and a stress of 2.4MPa (350 psi), with the time to failure being determined and recorded. A representative geometry for the specimens is shown in Figure 13:

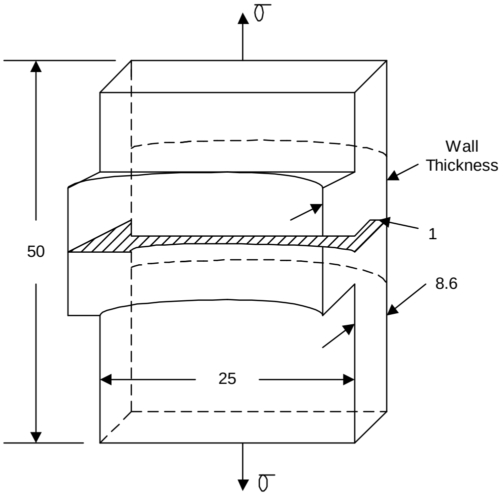


Figure 13: Schematic Illustration of PENT test specimens. Arrows designate the direction of the tensile stress (σ). All dimensions are in mm.

While the standard for the PENT test does not specify an acceptable failure test time, it is generally agreed that acceptable gas pipe resins are those that can resist failure for at least 50 to 100 hrs in a PENT test. Presently, the requirements within ASTM D2513 for PE materials require PENT time to failure of 100 hours. However, no such requirements are in place for Polyamide materials.

Two replicates of the PA12 materials from each of the PA12 suppliers (UBE, Degussa, and EMS) were tested in accordance to ASTM F1743 requirements. The results of the testing indicated that there were no failures with any of the specimens after 1000 hours, as shown in Table 16. The testing was discontinued after 1000 hours.

PA12 Supplier	Test Conditions	Results
UBE	Test Temp: 80°C Stress: 2.4 Mpa	> 1000 hours
Degussa		> 1000 hours
EMS		> 1000 hours

Table 16: Summary of the PENT time to failure data for the various PA12 pipe

3.5 Rapid Crack Propagation

It general, RCP considerations become more critical with increasing pressures, increasing diameters, increasing wall thickness, and decreasing temperatures. In order to effectively characterize the RCP resistance of plastic piping materials, promising test methodologies have been developed including the small-scale steady-state (S4 test) and full scale RCP test (FST). Given the cost effectiveness of the S4 test, it is the preferred test method.

The S4 test is performed in accordance to ISO 13477 guidelines *“Thermoplastic pipes for conveyance of fluids – Determination of rapid crack propagation (RCP) – Small-scale Steady-state (S4 Test).* Per the test requirements, a specified length of the plastic piping material is pressurized and maintained at a specified test temperature of 32°F in a test rig. The specimen is then impacted to initiate a fast growing longitudinal crack along the pipe length.

In order to establish the appropriate test conditions, a series of initiation tests are performed with un-pressurized pipe specimens at 32°F. Using a blade speed of 15m/s ± 5m/s, the pipe specimen is impacted and the crack growth is measured. For a given set of temperature and blade speed conditions, if the crack growth is greater than one (1) pipe diameter, the initiation conditions are considered to be satisfied and the same conditions are then used to determine the S4 critical pressure values.

Following the initiation testing, a series of iterative tests are performed using the initiation blade speed and temperature conditions at varying internal pressures. Crack propagation is then defined at pressure values where the measured crack exceeds 4.7 times the pipe diameter. The transition pressure from crack arrest to crack propagation then determines the S4 critical pressure value. It is important to note, the temperature is the most critical parameter. If the temperature of the pipe specimen is not closely monitored, then the S4 values obtained through this test can be overstated.

A series of S4 tests were performed using 6-inch SDR 11 pipe specimens supplied from both Degussa and EMS at varying internal pressures and 32°F until the S4 critical pressure values were obtained. Additional S4 tests were performed on 4-inch SDR 11 pipe supplied from UBE. The results of the testing are presented in Table 17 below.

PA12 Supplier	S4 Critical Pressure at 32°F
Degussa (6-inch SDR11)	55 psig
EMS (6-inch SDR 11)	40 psig
UBE (4-inch SDR 11)	40 psig

Table 17: Summary of the S4 critical pressure for the various PA12 suppliers

At present, no definitive statements can be made with respect to the significance of this particular test and its correlations to service performance. There is tremendous degree of uncertainty associated with the test procedure and the correlations to full-scale critical pressure values and maximum allowable operating pressure. Additional work has been proposed at the ISO level to perform full scale RCP testing of the PA12 materials by the various PA12 suppliers.

Section 4

Characterization of Critical Operating Considerations

4.1 Polyamide 12 Joining Procedures

A critical construction and maintenance concern involves the safety and integrity of various types of joints on plastic piping systems. By definition, thermoplastic materials are those materials that soften upon heating and re-harden upon cooling. This characteristic allows for joining thermoplastic materials by heat fusion. Heat fusion joining uses a combination of heat and force that results in two melted surfaces flowing together to make a joint.

Typically, heat fusion joining consists of the following:

1. Clean each pipe end
2. Insert facing tool and face pipe ends until the facer reaches the stops
3. Check alignment
4. Check heater (iron) plate temperature and insert between pipe ends
5. Bring ends of pipe in contact with the heater plate
6. Heat for prescribed times for the given size of pipe
7. Remove heater plate and promptly bring the melted ends together
8. Allow fusion joint to cool for prescribed times

To promote the safe joining of plastic piping materials, Title 49CFR 192.283 and 192.285 prescribes certain guidelines for developing and qualifying approved joining procedures that must be in place at each utility for their thermoplastic piping materials. Specifically, per Part 192 requirements, joining procedures are qualified when heat fusion joints are made in accordance to those procedures and are then subjected to a combination of tensile strength tests and either the quick burst or long term sustained pressure tests.

There are several factors that govern the integrity of the joint including pipe preparation, heater (iron) temperature, applied force, and cooling times. In order to develop suitable ranges for these parameters, GTI performed comprehensive parametric testing using the UBE PA12 material for 2-inch pipe sizes.

In previous GTI sponsored research, it has been demonstrated that the two parameters which affect the long term integrity of the heat fusion joints include the applied force (interfacial pressure) and the heat soak times (time the heater iron is in contact with the pipe material). A general practice of utilities is not to change the temperature of the heater iron when butt fusing in varying weather conditions. Instead, most utilities will consider modifying the "soak" time to allow more or less heat to absorb into the pipe ends for proper melting. To determine the impact of each of these parameters, several joints were prepared by varying each parameter while maintaining all others fixed. This is summarized in Table 18 below.

Condition	Test Parameter	Joining Conditions
1	Applied Torque Range of (10-20 ft-lbs) Using Heat Soak = 60 sec	Heater Iron Temp: 500F Heat Soak: 60 sec **Applied Torque: 10 ft-lbs** Torque Hold: 60 sec Clamp Time: 10 min
2		Heater Iron Temp: 500F Heat Soak: 60 sec **Applied Torque: 20 ft-lbs** Torque Hold: 60 sec Clamp Time: 10 min
3	Applied Torque Range of (10-20 ft-lbs) Using Heat Soak = 90 sec	Heater Iron Temp: 500F Heat Soak: 60 sec **Applied Torque: 10 ft-lbs** Torque Hold: 90 sec Clamp Time: 10 min
4		Heater Iron Temp: 500F Heat Soak: 60 sec **Applied Torque: 20 ft-lbs** Torque Hold: 90 sec Clamp Time: 10 min
5	Heat Soak Time 60 – 90 sec at Applied Torque of 10 ft-lbs	Heater Iron Temp: 500F **Heat Soak: 60 sec** Applied Torque: 10 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min
6		Heater Iron Temp: 500F **Heat Soak: 90 sec** Applied Torque: 10 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min
5	Heat Soak Time 60 – 90 sec at Applied Torque of 20 ft-lbs	Heater Iron Temp: 500F **Heat Soak: 60 sec** Applied Torque: 20 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min
6		Heater Iron Temp: 500F **Heat Soak: 90 sec** Applied Torque: 20 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min

Table 18: Fusion conditions utilized for parametric study to qualify PA12 joining procedures

Several fusion joints were made for each of the condition specified in Table 19 and tested in accordance to Part 192.283 requirements including the tensile strength determination, quick burst, and long term sustained pressure testing.

The results of the testing are summarized in Table 19 below for each of the tests.

Evaluation of Fusion Parameters – UBE PA12 Pipe				
	Condition 1	**Condition 2**	**Condition 3**	**Condition 4**
Quick Burst (Hoop Stress / Failure Mode)	7129 psi (Ductile)	7142 psi (Ductile)	7276 psi (Ductile)	7324 psi (Ductile)
Tensile Strength at Yield	6072 psi	5914 psi	6017 psi	5957 psi
Elongation at Yield	11%	11%	11%	11%
Tensile Strength at Break	---	---	---	---
Elongation at Break	120%	123%	119%	121%
LTHS Testing at 80°C and 290 psig (20 bars)	>1000 hours	>1000 hours	>1000 hours	>1000 hours
Evaluation of Fusion Parameters – Degussa PA12 Pipe				
	Condition 1	**Condition 2**	**Condition 3**	**Condition 4**
Quick Burst (Hoop Stress / Failure Mode)	7235 psi (Ductile)	7359 psi (Ductile)	7243 psi (Ductile)	7126 psi (Ductile)
Tensile Strength at Yield	6072 psi	5914 psi	6017 psi	5957 psi
Elongation at Yield	12%	11%	11%	12%
Tensile Strength at Break	---	---	---	---
Elongation at Break	123%	116%	121%	107%
LTHS Testing at 80°C and 290 psig (20 bars)	>1000 hours	>1000 hours	>1000 hours	>1000 hours

Table 19: Results of the testing per CFR Part 192 requirements to develop qualified PA12 heat fusion procedures

Based on the results of the testing, it is evident that the PA12 material, like the PE material, can be joined effectively using a wide range of heat fusion conditions. The results of the testing for each of the heat fusion joints are consistent with the values of pristine pipe previously presented in the respective sections above.

4.2 Effects of Squeeze-off

In addition to being able to effectively join piping segments to construct the gas distribution systems, an equally important maintenance consideration is effective flow control. A commonly used practice to shutoff the flow of gas is squeeze-off. The practice involves placing the piping materials between two plates and compressing the pipe until the internal pipe walls meet (“squeezed” together). In previous GRI sponsored research, it has been amply demonstrated that improper squeeze techniques can potentially adversely impact the long term performance of the piping material.

In order to ensure that long term performance is not compromised, ASTM D2513 Annex A1 specifies that pipe subjected to squeeze-off shall exhibit no leakage or visual evidence of splitting, cracking, breaking, or reduction in 1000-hour sustained pressure values.

To test the effect of squeeze-off on the PA12 materials, six specimens from each of the pipe producers were squeezed (un-pressurized) and then subjected to long term sustained pressure testing. Because the primary motivation is to ascertain information with respect to the long term performance after squeeze-off, the time, temperature, and stress condition were the same as the conditions utilized to validate the HDB ratings discussed in Section 3.2 above. Specifically, long term sustained pressure testing was performed at 80°C with an internal test pressure of 290 psig (20 bars) for a period of 1000 hours.

The results of the testing are summarized in Table 20 below. Based on a review of the data, there were no failures of any of the PA12 piping materials after 1000 hours of testing. It is important to emphasize that these conditions are significantly more aggressive than the validation protocols (80°C, 20 bar, for 200 hours) utilized on pristine pipe that has not been squeezed. This confirms the excellent SCG resistance of the PA12 material as evidenced by other SCG tests discussed in the previous sections above.

PA12 Supplier	Test Conditions	Results
UBE	Test Temp: 80°C Test Pressure: 290 psig (20 bars)	> 1000 hours
Degussa		> 1000 hours
EMS		> 1000 hours

Table 20: Summary of the long term sustained pressure testing to characterize effects of squeeze-off

4.3 Weathering

As part of the project to develop installation, operation and maintenance procedures for the use of Polyamide 12 in high-pressure natural gas distribution systems, an evaluation of the materials ability to withstand outdoor exposure conditions is essential. From a practical viewpoint, a gas utility using any thermoplastic material in its distribution system will be in a situation where thermoplastic pipe, fittings, etc. may be stored at its facility for an extended period of time. Therefore, a material's ability to withstand the effects of outdoor storage and its effect on the long-term performance of the material is a consideration.

All thermoplastics are subject to degradation due to outdoor exposure conditions. Degradation can occur through a combination of thermal/oxidative mechanisms, the absorption of UV irradiation and various environmental conditions such as moisture absorption and hydrolysis and/or chemical degradation due to pollutants. In general, the effect of degradation due to environmental exposure is material embrittlement and a reduction in physical and mechanical properties resulting in a potential for reduced service life.

In general, resin suppliers protect material against degradation due to environmental exposure through the use of suitable stabilizer packages incorporated into the polymer during the polymerization process or in subsequent compounding. Typical stabilizer packages protect the base material from degradation by acting as short and long-term thermal energy and UV absorbers and free radical scavengers. The degree of protection is a function of the efficiency and the quantity of the stabilizers chosen for use.

The natural gas industry recognizes the need for a material to withstand outdoor exposure conditions. ASTM D 2513 Annex5, Section A5.4.5, *"Outdoor Exposure Stability"* states that "PA pipe stored outdoors and unprotected for at least two years from the date of manufacture shall meet all of the requirements of the specification". Additionally, draft ISO specification 22621-1, *"Plastics Piping Systems for the Supply of Gaseous Fuels for Maximum Operating Pressure up to 20 bar – Polyamide (PA) – Part 1: General"*, requires that material meeting the specification exhibit outdoor weathering resistance with exposure levels greater than or equal to 3,5 GJ/m^2 such that exposed test specimens have minimum elongation at break values greater than or equal to 160%.

Due to the wide variation in environmental conditions from region to region, it is extremely difficult to make broad recommendations about a material's environmental resistance from an outdoor weathering study. Additionally, the correlation between results obtained from an outdoor weathering study and accelerated testing performed under laboratory conditions is generally poor. However, laboratory degradation studies offer the following advantages:

- Conditions are well controlled
- Variables can be eliminated or accurately controlled
- Small samples can be used
- Simultaneous experiments can be conducted yielding results in a shorter period of time.

It is generally accepted that of all the laboratory accelerated weathering procedures available, Xenon Arc weathering provides exposure conditions most closely simulating outdoor exposure conditions. Exposure response under the Xenon Arc are outlined in ASTM D2565-99 entitled, *"Standard Practice for Xenon-Arc Exposure of Plastics Intended for Outdoor Applications"*. In order to obtain useful information, GTI performed testing with polyamide 12 samples and PE samples which have known empirically observed weathering resistance.

To determine the effect of environmental exposure on the physical properties of PA 12, MDPE, and HDPE, ASTM D 638 tensile Type I specimens were fabricated from each of the plastic piping materials and exposed in a Q-Sun Xenon Test Chamber shown in Figure 14 below. The Xenon Arc testers produce UV, visible light, and infrared, and also simulate the effects of moisture through water spray and/or humidity control systems.

Figure 14: Q-Sun Xenon Test Chamber

Since ASTM standards do not quantify exposure limits and is inherently generalized, the ISO specification was used as to develop suitable test parameters. ISO specification 22621-1 requires that material meeting the specification exhibit outdoor weathering resistance with exposure levels greater than or equal to 3,5 GJ/m^2. Therefore, the selected irradiance output of the Xenon Test Chamber was set to 0.35 W/m^2 at 340 nm, with a typical irradiation value of 41.5 W/m^2 between 300 – 400 nm, to satisfy this requirement. The proposed exposure cycle is Cycle 1 from Table 1 in ASTM D2565, which calls for 102 minutes of light only exposure followed by 18 minutes light with water spray[2], i.e., 120 minutes (2 hours) of exposure per one cycle.

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[2] In Florida, the UV solar radiation per year at a 45-degree tilt angle is about 286 MJ/m2 or about 4.76% of the total solar irradiation (6000MJ/m^2). Therefore, the minimum total UV irradiation is about 166 MJ/m^2. If the cycle is 2 hours long and puts out an irradiance of 41.5 W/m^2, the number of cycles to reach 166 MJ/m^2 is 555. Assuming 12 cycles/day, the total irradiation of 166 MJ/m^2 will be met in 46 days.

Based on input from the project team, it was reasoned that all of the samples would be placed in the Xenon Arc chamber and conditioned at the appropriate irradiation levels. At periodic intervals corresponding to a certain number of cycles, samples would be removed and tensile strength determinations would be measured as the key response criterion. Specifically, the tensile strength at yield and elongation at yield would be measured. In doing so, if there was any appreciable change or a transition from a ductile to brittle region, then the corresponding total irradiation level would be known. Table 21 presents a summary of the exposure cycles and the total absorbed irradiation.

Number of cycles	Time (days)	Total Irradiation (MJ/m^2)
36	3	10.8
360	30	108
1080	90	323
1800	150	538
2160	180	645
2520	210	753

Table 21: Total Irradiation Values as a function of exposure intervals in Xenon Arc

The results of the testing are summarized in Table 22 below.

Xenon Weathering Exposure, Tensile Test Results, ASTM D 638, UBE PA12

Property/S	Control	3 Days	30 Days	90 Days	180 Days
Yield Strength(psi)	6607/120	6091/45	6024/279	5873/177	6071/127
Elongation at Yield (%)	10.0/0.7	12.9/0.2	13.0/0.7	10.8/0.8	12.7/0.5
Modulus (ksi)	NR	235/27	239/12	224/9	203/4
Break Stress (psi)	7776/185	7343/281	7132/140	6804/323	7126/155
Elongation @ Break (%)	258/8	258/38	258/22	0.94	252/8

Xenon Weathering Exposure, Tensile Test Results, ASTM D 638, Degussa PA12

Property/S	Control	3 Days	30 Days	90 Days	180 Days
Yield Strength(psi)	5370/98	5439/162	5162/219	5130/101	5256/127
Elongation at Yield (%)	12.0/1.1	13.9/0.7	14.8/0.2	14.5/0.5	15.4/0.9
Modulus (ksi)	194/15	211/26	194/2	177/5	210/19
Break Stress (psi)	6457/177	6207/176	5991/253	6050/227	6141/185
Elongation @ Break (%)	219/9	213/4	216/4	14.5/0.5	224/13

Xenon Weathering Exposure, Tensile Test Results, ASTM D 638, MDPE

Property/S	Control	3 Days	30 Days	90 Days	180 Days
Yield Strength(psi)	3064/47	3069/71	---	---	3089/55
Elongation at Yield (%)	11.5/0.9	12.0/0.6	---	---	11.7/0.4
Modulus (ksi)	152/21	116/8	---	---	115/8
Break Stress (psi)	NR	2268/104	---	---	2186/47
Elongation @ Break (%)	662/94	733/40	---	---	661/60

Xenon Weathering Exposure, Tensile Test Results, ASTM D 638, HDPE

Property/S	Control	3 Days	30 Days	90 Days	180 Days
Yield Strength(psi)	3141/82	3414/97	---	---	3427/33
Elongation at Yield (%)	10.5/0.9	11.0/0.8	---	---	11.7/0.2
Modulus (ksi)	123/5	133/1	---	---	127/9
Break Stress (psi)	0.13	2330/78	---	---	2245/31
Elongation @ Break (%)	578/111	334/79	---	---	317/56

Table 22: Measured tensile response for various thermoplastic piping (PA12, MDPE, and HDPE) after exposure to Xenon Arc accelerated weathering at various time intervals

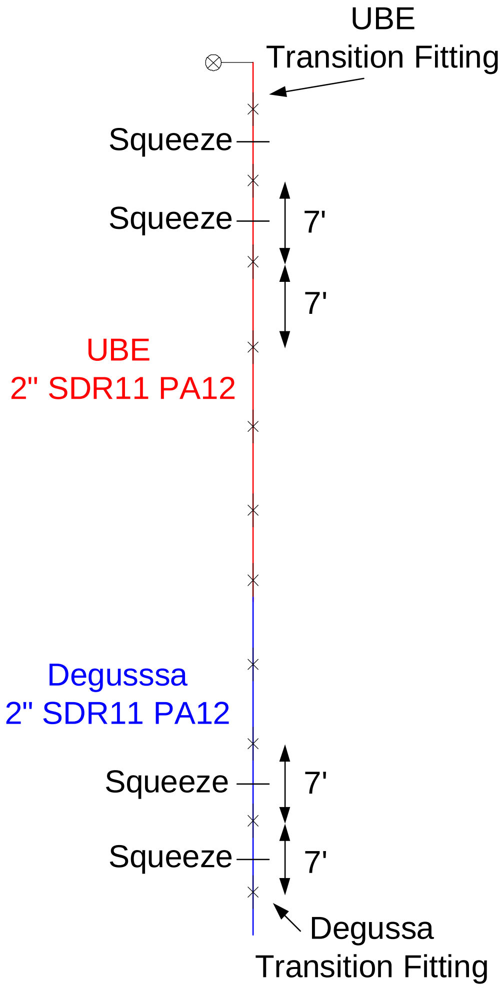
Section 5

Small-Scale Field Demonstration

In addition to the comprehensive laboratory evaluation, a small scale field installation was performed on GTI private property to gain better insight into the construction and maintenance of the PA12 piping systems and to characterize the effects of in-service conditions during February 2005.

Specifically, the primary objectives of this field demonstration were to evaluate the handling capabilities of PA12 pipe and the impact of squeeze-offs on PA12 piping material.

Two inch IPS SDR 11 PA12 pipe was used for the installation. The pipe was provided by two suppliers, Degussa and UBE. Approximately 70’ of PA12 pipe was installed, of which 42’ was UBE and 28’ was Degussa. The schematic below is a layout of the PA12 piping material used for the field installation.



The PA12 pipes were supplied in 7 foot stick lengths, which were fused together using PA12 joining procedures developed as part of this program, See Section 4.1:

- Butt Fusion Interface Pressure Range 60 - 90 psi
- (Corresponding Torque) (7 - 12 ft-lb)
- Heater Surface Temperature Range 495 – 505°F
- Time of contact with Heater Face 60 – 75 sec

Pipe ends were cleaned about 1-2” back with an alcohol wipe. The pipes were then clamped into a McElroy No. 14 Pitbull fusion machine. Alignment of the pipe ends were checked and adjusted as necessary. The pipe ends were faced to obtain clean, smooth mating surfaces. A heating tool was used to simultaneously heat both pipe ends. The temperature range of the heating tool was 495 – 505°F. The pipe ends were in contact with the heating tool for 60 seconds. Upon removing the heat source, the pipe ends were fused together using an applied torque of 10 ft-lb. This force was applied for 45 seconds. The fused pipe ends remained in the machine for a period of 10 minutes to ensure the integrity of the joint, as shown in Figure 15. Two transition fittings, one of each of the respective suppliers, were also installed. The transition fittings were heat fused to the end pipe lengths of each of the respective suppliers.

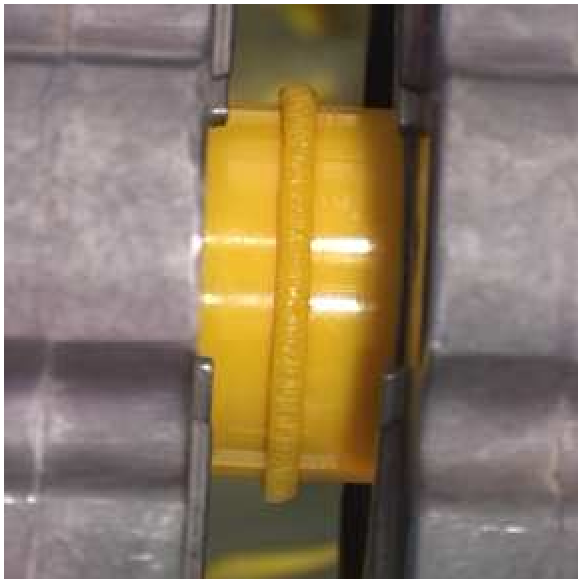


Figure 15: PA12 butt fusion joint

Since a squeeze-off technique is commonly used to control the flow of gas in the natural gas industry, the effects of this technique needed to be tested. Therefore, squeeze-offs were performed on sections of both Degussa and UBE pipe. As seen in the above diagram, two squeezes were performed on pipe segments of each of the respective suppliers. Each squeeze was performed in the middle of a 7’ pipe stick length, so that it was not in close proximity to any fusion joints. The PA12 pipe was inserted into a squeeze tool and centered between the squeeze-off bars. The squeeze time for a 2” diameter pipe is approximately 4 minutes. The screw clamp was turned 360° every 15 seconds to compress the tubing. The squeeze was continued until the tubing was completely compressed. The squeeze was then held in the squeeze tool for 4 hours, as shown in Figure 16.

Figure 16: Squeeze-off procedure on PA12

After the 4 hours elapsed, the tool was released in the same manner as it was applied. The screw clamp was turned 360° every 15 seconds until the tubing was completely released, as shown in Figure 17.

Figure 17: Completed squeeze-off on PA12

After completing the fusions and squeeze-offs, a trench was excavated for the field installation. The trench was approximately 100’ in length, 1’ in width, and 3’ in depth. Once installed, the PA12 flow loop was pressure tested for one hour to observe any leaks. The loop was pressure tested at 1.5 x MAOP, or 375 psig. After the pressure testing, the pressure of the flow loop was decreased and maintained at 250 psig, as shown in Figure 18.

Figure 18: PA12 Flow Loop Installation

After the flow loop was installed, the trench was backfilled. Approximately 6” of sand were placed above and below the pipe to mark the location of the flow loop for future excavations. It is proposed that the PA12 flow loop will be removed from the ground one year from the date of installation to perform comprehensive testing and evaluation on the transition fittings, joints, and squeeze-offs to characterize the effects of in-service conditions after exposure to one complete seasonal cycle.

Section 5

Summary and Conclusions

Through the support of the Operations Technology Development program and resin suppliers, a comprehensive program has been established to perform testing and evaluation of Polyamide 12 (PA12) material. Specifically, to validate the technical feasibility for the use of Polyamide 12 (PA12) pipe at higher operating pressures and larger diameters through a series of laboratory and field experiments focused on the development of comprehensive physical properties and critical construction, maintenance, and operating considerations data.

Based on the cumulative results of the comprehensive testing, several conclusions can be made:

- The results of the comprehensive testing with respect to the physical, mechanical, and chemical properties demonstrate the PA12 piping material conforms to all of the requirements contained with ASTM D2513 and its respective Annexs
- The results of each of the SCG tests demonstrate that the PA12 piping material has excellent resistance to the SCG mechanism. This is substantiated by the lack of failures in all of the testing including: HDB validation, notched pipe testing (20%, 30%, and 50%), and PENT testing using very aggressive test conditions
- Critical construction and maintenance procedures can be readily applied to the PA12 piping material without the need for additional equipment and or major modifications to existing procedures used for PE piping systems
- The results of the RCP testing are inconsistent with expectations. The calculated maximum operating pressure is lower than the target range of 200 psi; however, the meaningfulness of the test procedure, the efficacy of the correlation function, and the implicit safety factor are at best questionable. These doubts do not apply exclusively to the PA12 piping material but also to PE materials. As a result, at present, there are no requirements in place for either the PE materials and/or the Polyamide 11 and 12.

Based on the cumulative results of the testing, it can be reasonably inferred that the PA12 material is a suitable for material for high pressure gas distribution piping applications.