

**Summary of August 26, 2008 meeting to discuss technical reports and updated information related to Polyamide-12 (PA-12) piping systems intended for higher operating pressures - Docket ID: PHMSA-2007-29042**

On August 26, 2008, the following individuals were present at a meeting at U.S.DOT Headquarters to discuss technical reports and updated information related to PA-12 piping systems intended for higher operating pressures:

Dr. Jörg Lohmar – Evonik Industries (Germany)  
Hitesh Patadia – Tej Group, Inc. (U.S.)  
Richard Wolf – Polymer Processing Solutions, Inc. (U.S.)

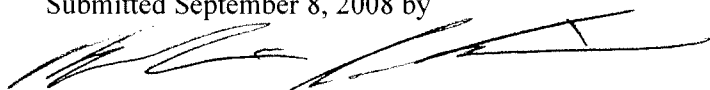
Mike Israni (U.S. DOT PHMSA)  
Max Kieba (U.S. DOT PHMSA)  
Alan Mayberry (U.S. DOT PHMSA)

On April 27, 2007, the group sent a letter (also on docket) for PHMSA to consider a petition to amend relevant safety regulations in 49 CFR Part 192. The petition was returned due to lack of technical information in a number of areas. The group's presentation on August 26 was a follow-up to another letter to PHMSA in May 2008 with new reports and information to describe how they addressed, or are addressing, the issues.

Included in this document are the following attachments:

- May 31, 2008 letter to PHMSA providing additional information and reports, and requesting follow-up meeting
- Generic butt heat fusion guidelines for PA12 piping systems (referenced in May 31 letter)
- Characterization of the Rapid Crack Resistance characteristics of the PA 12 piping systems (referenced in May 31 letter)
- Field Demonstrations and Aging Characterization of the PA 12 piping systems (referenced in May 31 letter)
- Two technical reports developed by the Gas Technology Institute (GTI) as part of the work sponsored by the Operations Technology Development (OTD) NFP (reference in May 31 letter)
  - o TECHNICAL REFERENCE ON THE PHYSICAL, MECHANICAL, and CHEMICAL PROPERTIES OF POLYAMIDE 12 (PA12) FOR HIGH PRESSURE GAS DISTRIBUTION APPLICATIONS
  - o EVALUATION OF POLYAMIDE 12 (PA12) FOR HIGH PRESSURE GAS DISTRIBUTION APPLICATIONS
- August 26 presentation at PHMSA
- POLYAMIDE 12 (PA12) FIELD EVALUATIONS UTILITY FIELD DEMONSTRATION PROGRESS REPORT submitted September 8, 2008

Submitted September 8, 2008 by



Maximillian (Max) J. Kieba  
General Engineer  
U.S. DOT PHMSA Office of Pipeline Safety

9/8/2008

May 31, 2008

Mr. Jeff Wiese  
Associate Administrator for Pipeline Safety  
PHMSA / Office of Pipeline Safety  
U.S. Department of Transportation  
400 7th Street, SW  
Room 2103  
Washington, DC 20590-0001

**Subject: Technical Reports and Updated Information Related to Polyamide 12  
(PA 12) Piping System Intended for Higher Operating Pressures  
Reference Docket ID: PHMSA – 2007 - 29042**

Dear Mr. Wiese

In response to the request for additional technical information by DOT-PHMSA, Evonik-Degussa AG and UBE Industries are pleased to submit the attached technical summary reports which demonstrate the ability to safely construct, maintain, and operate PA12 piping system at higher operating pressures up to 250 psig.

Specifically, three reports are being presented for review and considerations. These include:

- Generic butt heat fusion guidelines for PA12 piping systems
- Characterization of the Rapid Crack Resistance characteristics of the PA12 piping systems
- Field Demonstrations and Ageing Characterization of the PA12 piping systems

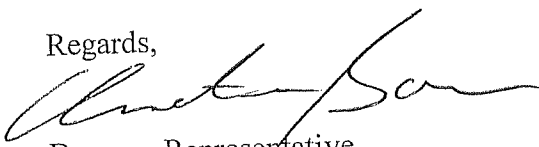
In addition to the aforementioned reports, two technical reports developed by the Gas Technology Institute (GTI) as part of the work sponsored by the Operations Development Group (OTD) NFP are also being submitted for review and consideration.

The cumulative body of the technical information that is being presented in these respective reports strongly supports the case for the inclusion of the PA 12 piping systems within Title 49 CFR Part 192 requirements as an effective alternative to metallic piping and their associated issues.

We would kindly like to request some time with you and your staff to go over the comprehensive body of the technical work related to the PA 12 program and provide an update prior to the resubmitting the revised PA12 NPRM.

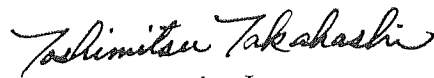
We would like to thank you for your time and consideration of this matter.

Regards,

i.A. 

Degussa Representative

Dr. Christian Baron



UBE America Inc.

President

Toshimitsu Takahashi

## **Generic Butt Fusion Joining Procedures for Field Joining of Polyamide 12 (PA 12) Pipe**

Prepared By:  
PA12 Suppliers  
Evonik-Degussa and UBE Industries

May 2, 2008

## OVERVIEW

To promote the safe joining of plastic piping materials, Title 49CFR Part 192 prescribes certain requirements for developing and qualifying approved joining procedures that must be in place at each utility for use with their plastic piping materials. Specifically,

- Each joint must be made in accordance with written procedures that have been proven by test or experience to produce strong leak tight joints – CFR Part 192, §192.273
- Written procedures for various types of joints must be qualified by subjecting them to various required tests – CFR Part 192, §192.283
- All persons making joints must be qualified under the operators written procedures - CFR Part 192, §192.285
- Gas system operators must ensure that all persons who make or inspect joints are qualified - CFR Part 192, §192.285 and §192.287

In order to ensure compliance to CFR Part 192 requirements, the Polyamide 12 (PA 12) suppliers (Evonik Degussa and UBE Industries) have performed comprehensive testing and evaluation to establish the technical basis for generic heat fusion procedures specific to PA 12 piping systems. The specific intent of the testing was to address the following key considerations:

- Establishing the optimum set of butt heat fusion joining parameters which lead to strong joints that can effectively perform over the intended service life
- Verifying the ability to make strong joints over a range of pipe sizes
- Verifying the compatibility of cross-fusion using PA12 pipe from the various manufacturers

In order to address each of these key considerations, extensive sample fabrication and testing was performed at leading independent research institutes and McElroy manufacturing. The comprehensive results of the testing confirm the ability to make strong heat fusion joints using a range of heat fusion parameters.

The following report presents a summary of the sample fabrications details, testing protocols, and results. Based on these results, a generic PA 12 heat fusion procedure has been established for the industry. It is important to note that these procedures are being presented as guidelines to help assist gas utility companies in developing and/or qualifying their own procedures for use with PA12 piping systems. Moreover, these procedures have also been integrated within ISO 22621 Part 2 requirements for PA 12 piping systems. Additional work is on-going to integrate these procedures within ASTM and other industry accepted standards and specifications for use in the United States.

## QUALIFICATION OF PA 12 HEAT FUSION PROCEDURES

### *Gas Technology Institute (GTI) Program*

In order to comply with the relevant requirements contained within CFR Part 192 requirements, the PA 12 suppliers with the support of the Gas Technology Institute and McElroy Manufacturing performed comprehensive testing to establish and validate a suitable range of generic heat fusion parameters. From the onset, it was noted that previous experience with other thermoplastic materials, specifically polyethylene and polyamide 11 materials, there is a wide range for key heat fusion parameters which can influence the strength and integrity of the butt heat fusion joint.

In general, there are numerous variables which can impact the overall strength and integrity of a butt heat fusion joint including surface preparation, heater iron temperature, interfacial pressures, and overall heating and cooling times. In order to aid the industry in developing a uniform and standardized heat fusion procedure for PE materials, the Plastics Pipe Institute, during the mid-1990's, performed extensive testing to develop a range of suitable heat fusion parameters and procedure. This procedure was issued as PPI TR-33 guidelines.

As a first step in the development of the PA 12 heat fusion procedure, several PA 12 heat fusion joints were made in accordance to PPI TR-33 guidelines for PE materials with the exception of using a higher heater iron temperature of 500F. The higher heater iron temperature was specified given the higher melt point for the PA 12 materials as compared to PE materials. However, it is important to emphasize that for some high density PE materials, this higher heater iron temperature is also specified by a majority of the gas utility companies. The remaining parameters were kept the same as compared to PPI TR-33 guidelines.

Subsequently, for the PA 12 materials, the following parameters were evaluated using both 2-inch IPS SDR 11 and 6-inch IPS SDR 11 PA 12 pipe sizes from three of the four (3/4) PA 12 suppliers including Evonik-Degussa, UBE Industries, and EMS Grivory.

*Heater Iron Temperature:*     $500^{\circ}\text{F} \pm 10^{\circ}\text{F}$

*Interfacial Pressure Range:*     $60 - 90 \text{ psi}$

All of the fusions were performed using the visual melt bead width guidance provided under PPI TR-33 guidelines.

Six specimens (like to like, e.g. Evonik pipe to Evonik pipe) were made using the upper and lower bound limits for interfacial pressure ranges and heating times with the appropriate heat fusion equipment settings as follows:

- Condition 1: Minimum heating time / Minimum interfacial Pressure (60 sec / 60 psi)
- Condition 2: Minimum heating time / Maximum interfacial pressure (60 sec / 90 psi)

- Condition 3: Maximum heating time / Minimum interfacial pressure (90 sec / 60 psi)
- Condition 4: Maximum heating time / Maximum interfacial pressure (90 sec / 90 psi)

The specimens were then tested in accordance to the requirements contained within CFR Part 192.283. Specifically, CFR Part 192.283 requires either the burst pressure testing or long term sustained pressure testing and tensile strength determinations. For the PA 12 butt heat fusion joints, all three tests were performed. The results were consistent with expectations – strong joints can be effectively made using these conditions. The results of the burst pressure testing and tensile strength testing demonstrated that the resulting PA 12 heat fusion joint has similar properties as compared to PA 12 un-fused pipe. Moreover, results of long term sustained pressure testing at 80°C at test pressures corresponding to 290 psi demonstrated that there were no failures for test times greater than 1000 hours. Representative test results are summarized in Table 1 below:

<b>Evaluation of Fusion Parameters – PA12 Pipe (Typical)</b>					
	<b>Control</b>	<b>Condition 1</b>	<b>Condition 2</b>	<b>Condition 3</b>	<b>Condition 4</b>
Quick Burst (Hoop Stress / Failure Mode)	6899 psi (Ductile)	7235 psi (Ductile)	7359 psi (Ductile)	7243 psi (Ductile)	7126 psi (Ductile)
Tensile at Yield	5370 psi	6072 psi	5914 psi	6017 psi	5957 psi
Elongation at Break	219%	123%	116%	121%	107%
Failure times for long term sustained pressure testing at 80°C and 290 psig (20 bars)	No Failures at times greater than 1000 hours	No Failures at times greater than 1000 hours	No Failures at times greater than 1000 hours	No Failures at times greater than 1000 hours	No Failures at times greater than 1000 hours

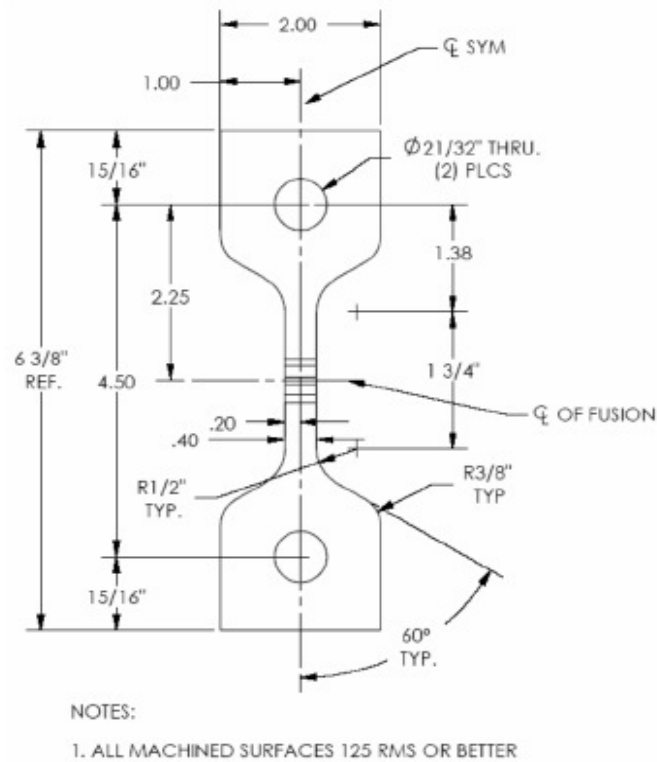
**Table 1: Results of the testing per CFR Part 192 requirements to develop qualified PA12 heat fusion procedures**

Given that the primary intended application for PA12 piping system is for higher pressures and larger diameters, additional tests were performed to qualify these procedures for 6-inch IPS pipe specimens. Comprehensive tests were performed on parametrically controlled fusion joints made in accordance to the previously developed PA12 joining procedures with exception of varying the interfacial pressures and heater iron temperatures. Moreover, the compatibility of cross-fusions between each of the PA12 resin suppliers' product was also investigated.

With the assistance of McElroy Manufacturing, several 6-inch PA12 butt heat fusion joints were prepared using the specific PA12 joining procedures. Specifically,

- 12 fusion joints were made from using the Evonik VESTAMID and UBE UBESTA PA12 materials
- 1 base materials from each of the PA 12 resin suppliers – pipe used as control specimens
- 3 cross fusions from different supplier materials

Four (4) coupons were machined from each fusion joint and subjected to McSnapper™ testing, as shown in Figures 1. The McSnapper™ is a high speed tensile-with-impact testing machine which combines the Tensile Impact Test ASTM D1822 and High Speed Tensile Test ASTM D2289. Figure 2 illustrates the progression of the McSnapper™ testing apparatus.



**Figure 1: Schematic Illustration of test specimen used for McSnapper™ Testing**



**Figure 2: Typical McSnapper™ test set-up and testing progression of a typical PA12 test specimens at an average speed of 6 in/s.**

The results of the McSnapper™ testing on the 6-inch PA12 heat fusion joints were consistent with expectations. There was an excellent correlation between the tensile strength values of the PA12 joints as compared to PA12 pipe specimens. Figures 3-4 presents a summary of the test data for various joints and control specimens. Detailed summary of the McSnapper™ test results are presented in Appendix A of this report.

In addition to validating the performance of PA12 joints made from “like to like” PA12 pipe, additional joints were fabricated using the PA12 pipe from other suppliers’ resin – “unlike” joints or cross-fusions. The test results confirmed the ability to make strong and effective cross-fusion joints, i.e. joints made using PA12 pipes from different suppliers. It was reported that in all of the test specimens, the failures originated outside the fusion interface. The overall results demonstrated that strong effective 6-inch PA12 joints can be made using the generic PA12 joining procedures.

In addition to the McSnapper™ testing, additional long term sustained pressure testing at elevated temperatures were performed on 6-inch PA12 pipe specimens using 290 psig at 80C. As expected, the results of the testing were consistent with expectations. There were no failures in any of the PA12 heat fusion joints at test times greater than 1000 hours.

Cumulatively, the results of the testing (tensile impact testing and long term sustained pressure testing at elevated temperatures) amply demonstrated the ability to make strong joints using the qualified PA12 joining procedures consistent with CFR Part 192 requirements.

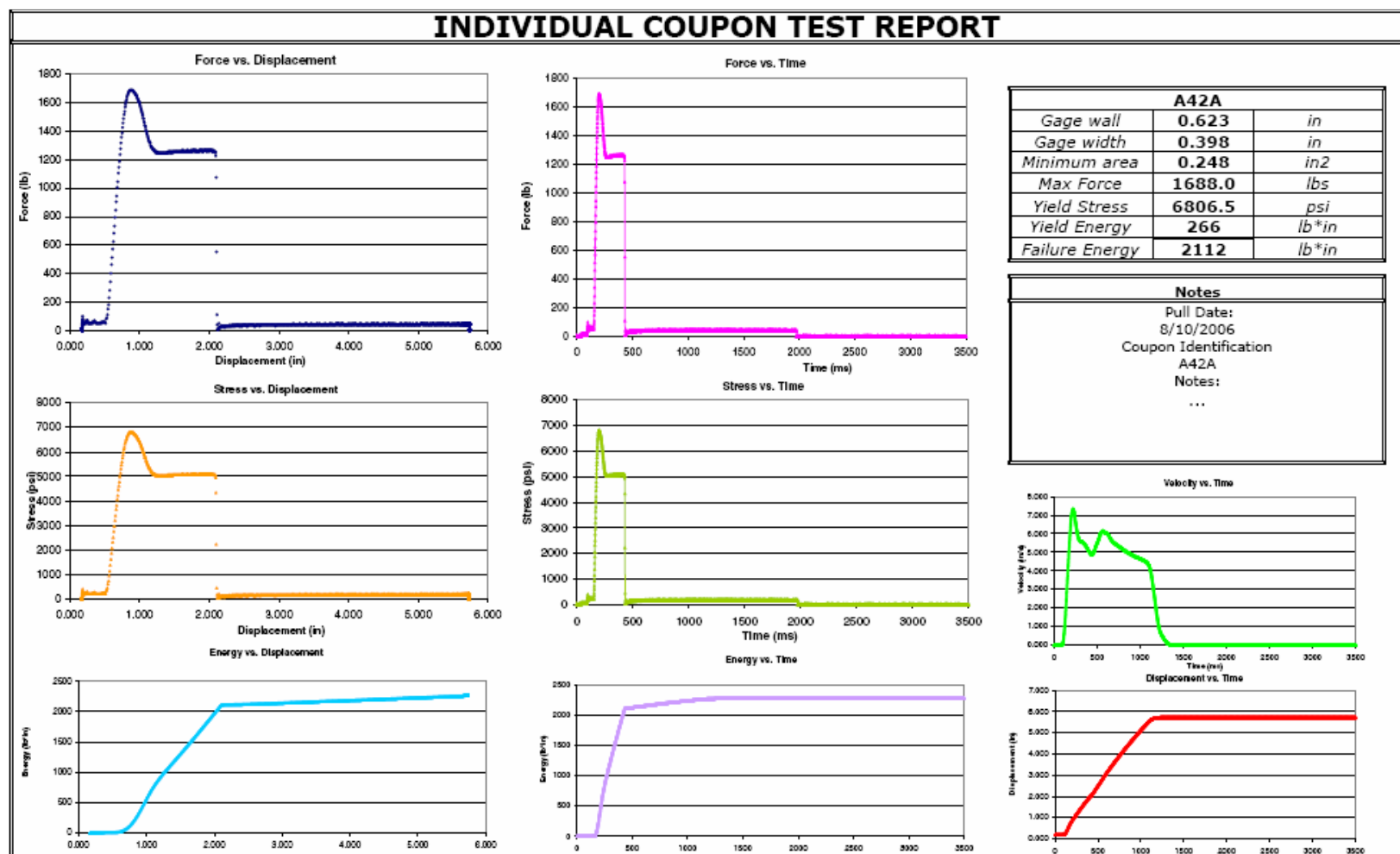


Figure 3: Illustration of test results from McSnapper testing for a typical PA12 heat fusion joint

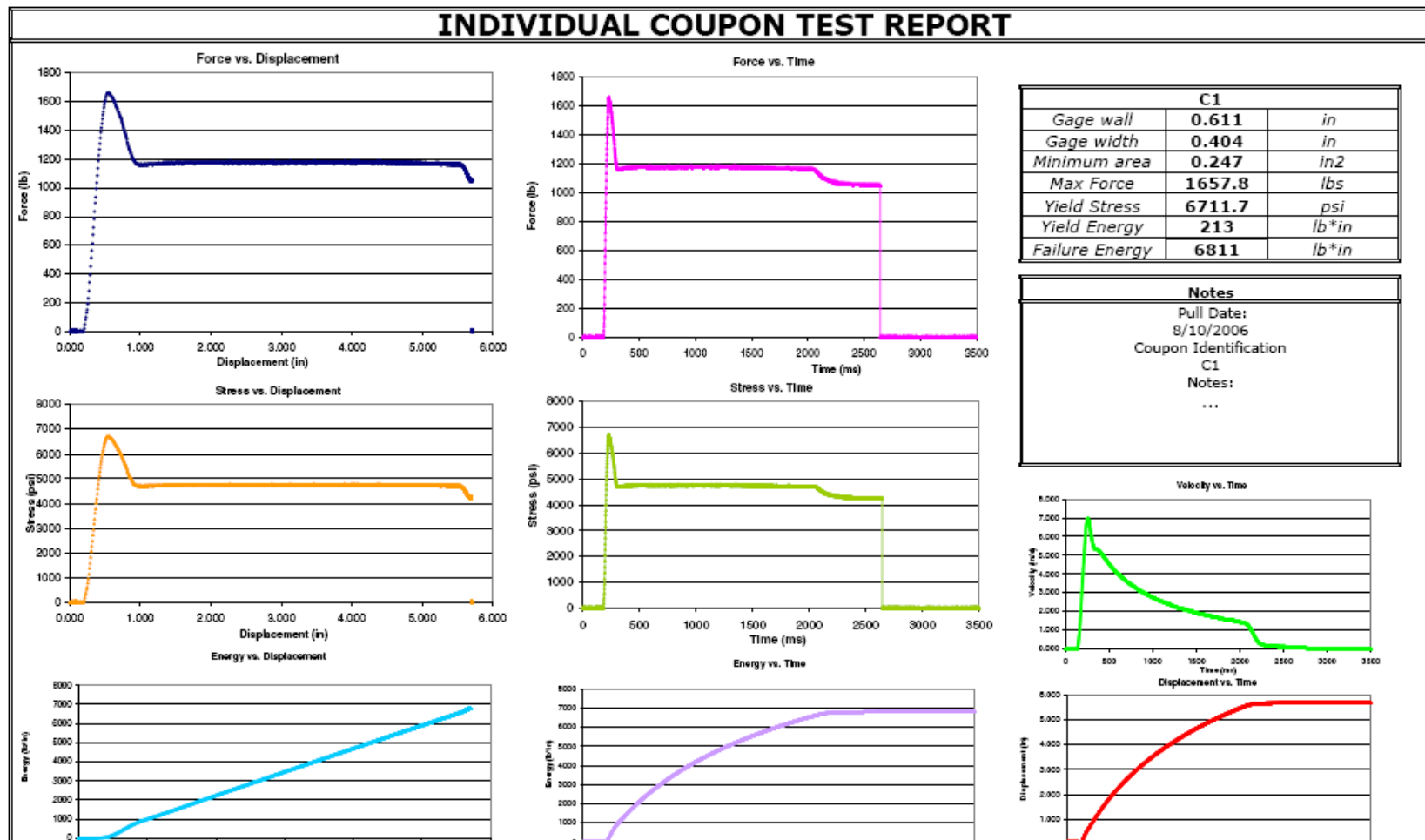


Figure 4: Illustration of test results from McSnapper testing for a typical PA12 pipe specimen

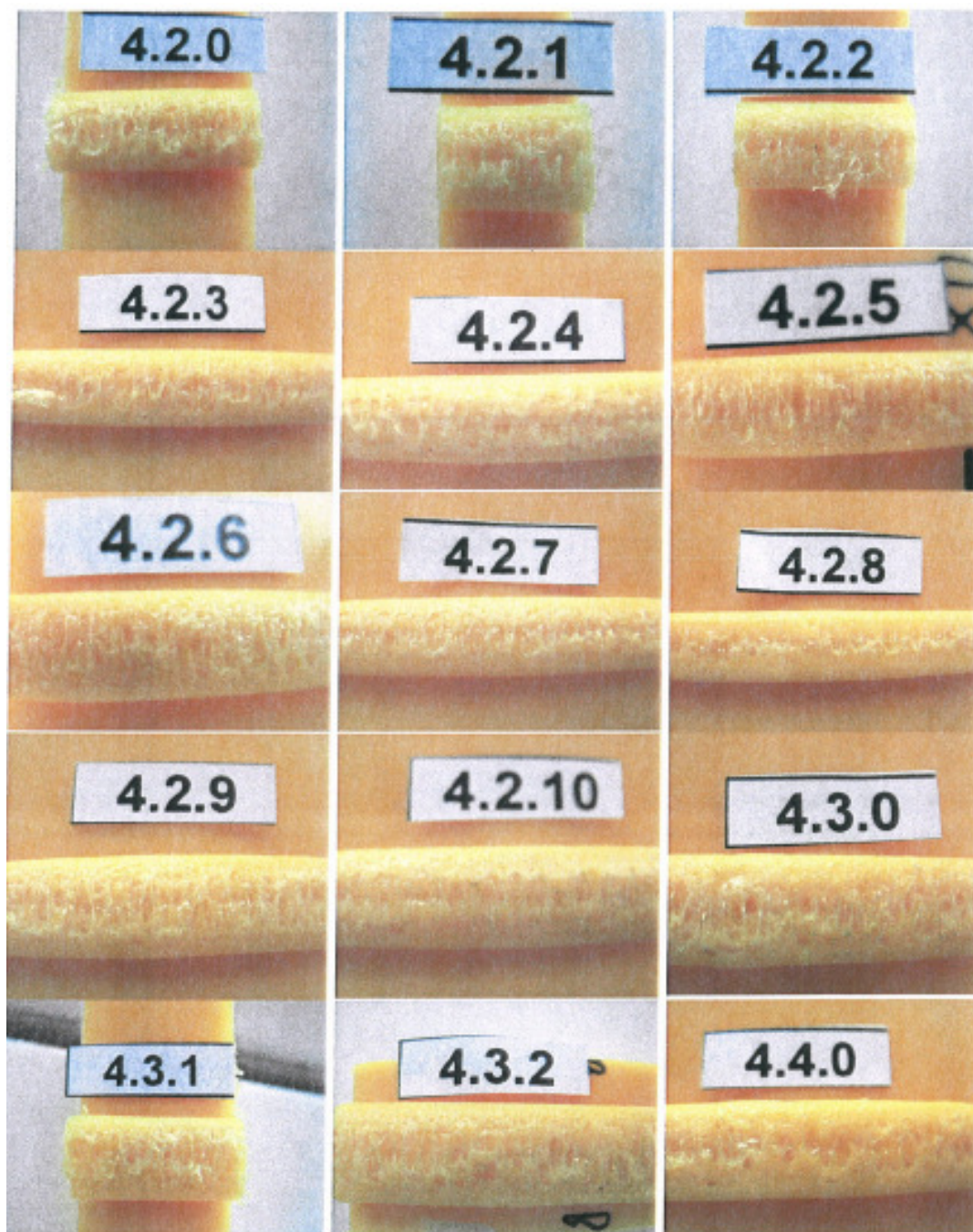
### ***Sueddeutsches Kunststoff Zentrum (SKZ) Program***

In addition to the GTI program, Evonik Degussa also commissioned an extensive research program to optimize the PA 12 butt heat fusion parameters at a leading German research institute – SKZ Welding Institute. While the results of the GTI program, as previously discussed, verified the ability to make strong butt heat fusion joints using the range of interfacial pressures as specified under PPI TR-33 guidelines with a heater iron temperature of 500F, it was noted that there are potentially significant differences in heat fusion practices throughout the world. Specifically, in Europe and elsewhere, a general industry practice is to utilize lower interfacial pressures as compared to the higher interfacial pressures specified in the United States. As a result, additional parametrically controlled tests were performed.

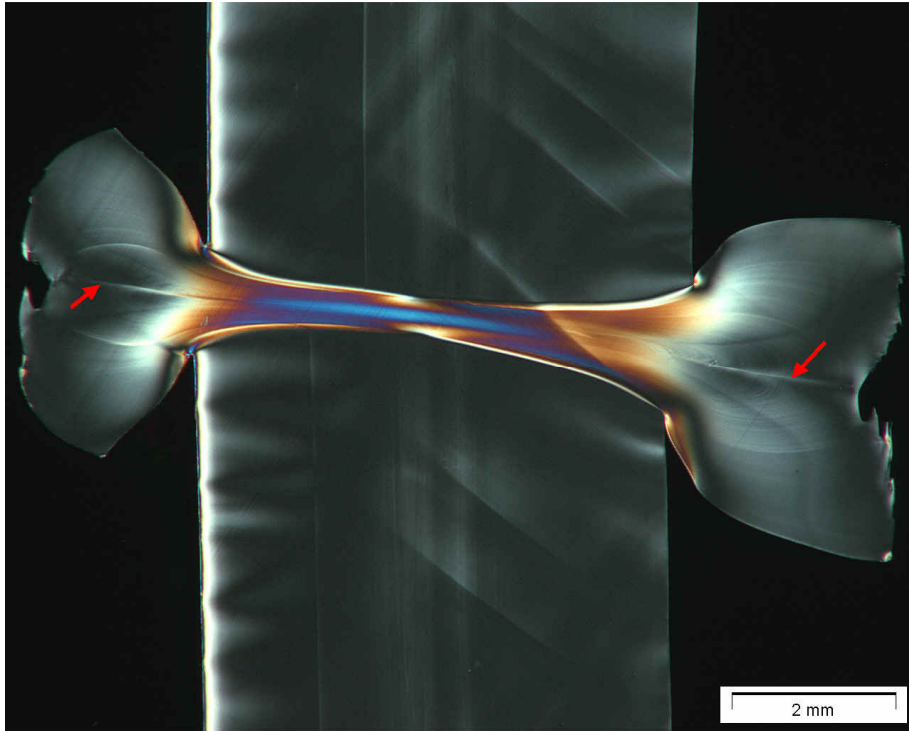
In the SKZ study, a range of heater iron temperatures were evaluated from 200C – 260C (392F – 500F) using different interfacial pressure ranging from 0.3 N/mm<sup>2</sup> to 0.6 N/mm<sup>2</sup> and varying heating times. The results of the testing also confirmed the ability to make strong joints over a wide range of butt heat fusion parameters as specified in the generic PA12 heat fusion procedure. Moreover, the results further confirmed that there a smaller and more well defined range of parameters within the generic heat fusion parameters can be used which will not only produce strong joints but will also lead to more visually acceptable joints.

In general, Polyamides tend to absorb moisture to varying degrees – PA12, based on its inherent chemical characteristics, tends to absorb the lowest amount of moisture of the various commercially available polyamides. Regardless, it is noted that during the application of heat in the joining process, the water tends to evaporate. Subsequently, the final bead appearance tends to be slightly bit different as compared to polyethylene. This is illustrated in Figure 5 which shows the pipe interface following the completion of the specified heating time to produce the required melt bead. As one increases the heater iron temperature, it is observed that there is a corresponding impact on the bead appearance. However, as the testing results demonstrate, at lower heater iron temperatures around 240C (465F) and the appropriate interfacial pressures, strong joints can be made with a relatively better visual appearance. It is important to emphasize that anecdotal experience throughout the world indicates that the final visual bead appearance is merely aesthetic and has no correlation to joint strength.

To further illustrate that the overall bead appearance of the PA12 does not correlate with the strength of the overall joint across the joint interface, additional x-ray analysis was performed which shows that there is continuity of the polymer across the interface and that there are no voids and/or other discontinuities. This is illustrated in Figure 6 below.



**Figure 5: Melt appearance of the PA 12 pipe ends as a function of temperature**



**Figure 6: Representative X-Ray illustration of typical PA12 heat fusion joint (Note: there are no discontinuities or voids across the joint interface)**

## **CONCLUSION AND RECOMMENDATIONS**

The cumulative results of both the GTI and SKZ study indicate that there is a suitable heat fusion procedure for PA 12 materials has been developed on the basis of the required testing per CFR Part 192 requirements – see Appendix A – which can produce strong PA 12 butt heat fusion joints. The PA 12 pipes supplied from the different pipe suppliers for the purposes of this program meet ASTM D2513 requirements and are suitable for gas distribution applications. Based on the available information and the results of the testing, there is a strong likelihood that that the generic heat fusion procedure for PA 12 can be qualified by gas distribution companies under DOT’s regulations in Part 192 for use with PA 12 gas piping products.

## APPENDIX A

### **Generic Butt Fusion Procedure for Polyamide 12 (PA 12) Pipe**

The following butt fusion procedures are intended for use with polyamide 12 pipe. Critical parameters in the butt fusion process are heater iron surface temperature, heat soak time, interfacial pressure during initial contact of the molten pipe ends and during cooling and cooling time. The heat soak time and cooling time parameters vary as a function of pipe size and wall thickness. As a general guideline, the heat soak time should be sufficiently long to produce a melt bead of approximately 1/16 – 1/8". The pipe should be held under pressure until it is sufficiently cool enough to touch with bare hands.

#### **Butt Fusion Procedure Parameters:**

Interface Pressure Range<sup>1</sup>: 60 – 90 psi

Heater Surface Temperature Range: 460 - 500 ± 10°F

#### **Butt Fusion Procedures:**

The principle of heat fusion is to heat two surfaces to a designated temperature, then fuse them together by application of a sufficient force. This force causes the melted materials to flow and mix, thereby resulting in fusion. When fused according to the proper procedures, the joint area becomes as strong as or stronger than the pipe itself in both tensile and pressure properties.

Field-site butt fusions may be made readily by trained operators using butt fusion machines that secure and precisely align the pipe ends for the fusion process.

The seven steps involved in making a butt fusion joint are:

1. Clean the pipe ends
2. Securely fasten the components to be joined
3. Face the pipe ends
4. Align the pipe profile
5. Melt the pipe interfaces without pressure.
6. Join the two profiles together.
7. Hold under pressure until cool.

#### **Clean**

Clean the inside and outside of the pipe to be joined by wiping with a clean lintfree cloth. Remove all foreign matter.

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<sup>1</sup> Interfacial pressure is NOT the same as the gauge pressure. The interfacial pressure is used to determine the joining pressure setting on hydraulic machines when joining specific pipe diameters and SDR values. These values will vary based on the heat fusion equipment supplied by various manufacturers.

## **Secure**

Clamp the components in the machine. Check alignment of the ends and adjust as needed.

## **Face**

The pipe ends must be faced to establish clean, parallel mating surfaces. Most, if not all, equipment manufacturers have incorporated the rotating planer block design in their facers to accomplish this goal. Facing is continued until a minimal distance exists between the fixed and movable jaws of the machine and the facer is locked firmly and squarely between the jaw bushings. This operation provides for a perfectly square face, perpendicular to the pipe centerline on each pipe end and with no detectable gap.

## **Align**

Remove any pipe chips from the facing operation and any foreign matter with a clean, untreated, lint-free cotton cloth. The pipe profiles must be rounded and aligned with each other to minimize mismatch (high-low) of the pipe walls. This can be accomplished by adjusting clamping jaws until the outside diameters of the pipe ends match. The jaws must not be loosened or the pipe may slip during fusion.

## **Melt**

Heating tools that simultaneously heat both pipe ends are used to accomplish this operation. These heating tools are normally furnished with thermometers to measure internal heater temperature so the operator can monitor the temperature before each joint is made. However, the thermometer can be used only as a general indicator because there is some heat loss from internal to external surfaces, depending on factors such as ambient temperatures and wind conditions. A pyrometer or other surface temperature-measuring device should be used periodically to insure proper temperature of the heating tool face. Additionally, heating tools are usually equipped with suspension and alignment guides that center them on the pipe ends. The heater faces that come into contact with the pipe should be clean, oil-free and coated with a nonstick coating as recommended by the manufacturer to prevent molten plastic from sticking to the heater surfaces. Remaining molten plastic can interfere with fusion quality and must be removed according to the tool manufacturer's instructions.

Plug in the heater and bring the surface temperatures up to the temperature range. Install the heater in the butt fusion machine and bring the pipe ends into full contact with the heater. To ensure that full and proper contact is made between the pipe ends and the heater, the initial contact should be under moderate pressure. After holding the pressure very briefly, it should be released without breaking contact. Continue to hold the components in place, without force, while a bead of molten polyamide develops between the heater and the pipe ends. When the proper bead size is formed against the heater

surfaces, the heater should be removed. The bead size is dependent on the pipe size. See table below for approximate melt bead sizes.

Pipe Size	Approximate Melt Bead Size
1 1/4" and smaller (40mm and smaller)	1/32" – 1/16" (1-2 mm)
Above 1 1/4" through 3" (above 40 mm-90mm)	About 1/16" (2 mm)
Above 3" through 8" (above 90mm – 225mm)	1/8"-3/16" (3-5mm)

## Joining

After the pipe ends have been heated for the proper time, the heater tool is removed and the molten pipe ends are brought together with sufficient force to form a bead against the pipe wall. The fusion force is determined by multiplying the interfacial pressure by the pipe area.

For manually operated fusion machines, a torque wrench may be used to accurately apply the proper force. For manual machines without force reading capability of a torque wrench, the correct fusion joining force is the force required to form a homogeneous bead during joining. For hydraulically operated fusion machines, the fusion force can be divided by the total effective piston area of the carriage cylinders to give a hydraulic gauge reading in bar. The gauge reading is theoretical; the internal and external drag need to be added to this figure to obtain the actual fusion pressure required by the machine.

## Hold

The molten joint must be held immobile under pressure until cooled adequately to develop strength. Allowing proper times under pressure for cooling prior to removal from the clamps of the machine is important in achieving joint integrity. The fusion force should be held between the pipe ends until the surface of the bead is cool to the touch. The pulling, installation or rough handling of the pipe should be avoided until the joint cools to ambient temperature (roughly an additional 30 minutes).

## Visual Inspection

Visually mitered (angled, off-sets) joints should be cut out and re-fused (straight or coiled pipe).

Coiled pipe may leave a bend in some pipe size that must be addressed in the preparation of the butt heat fusion process. There are several ways to address this situation.

1. Straighten and re-round coiled pipe before the butt fusion process (ASTM D2513 requires fielder-rounding coiled pipe before joining pipe sizes larger than 3" IPS)
2. If there is still curvature present, install the pipe ends in the machine in an "S" configuration with the print lines approximately 180° apart in order to help gain proper alignment and help produce a straight joint.

3. If there is still a curvature present, another option would be to install a straight pipe of pipe between the two coiled pipes.

Every effort should be made to make the joint perpendicular to the axis of the pipe.

# **RCP Correlations for the Polyamide 12 (PA 12) Piping Systems**

Prepared By:  
PA12 Suppliers  
Evonik-Degussa and UBE Industries

May 2, 2008

## BACKGROUND

A critical design consideration for piping systems is to ensure ample safeguard between the maximum operating pressure and the propensity for a brittle, rapidly propagating crack that can lead to large volumes of gas being lost through a failure of the gas pipeline. Rapid Crack Propagation (RCP) in plastic pipe is characterized by a brittle failure in which crack growth can occur at speeds ranging from 300 to 1400 ft/sec. This mode of failure is distinctly different than the slow crack growth failures which propagate at minute rates and over very small lengths. There are several factors that contribute to RCP including temperature, pipe diameter, wall thickness, polymer type, processing, and etc.

From the onset it is important to emphasize that, in general, RCP has not been a documented problem in current operations of plastic gas distribution piping systems for several possible reasons. The first is that a necessary pre-requisite for RCP - the existence of a large, through-wall, axial crack - seldom occurs. Secondly, current systems operate with relatively low pressures and use small diameter pipe, both of which tend to strongly diminish the risk of an RCP failure. However, given the current unmistakable trend towards installing larger diameter pipes and using higher operating pressures, the risk of RCP potentially rises appreciably. Particularly when either of these conditions is coupled with low operating temperatures, even moderately large cracks that come into existence by mechanical impact, improper squeeze-off, and installation damage could trigger RCP. Therefore, it is imperative to quantify the susceptibility to RCP for any new pipe material being used for gas distribution applications.

Over the past decade, two standardized test methods have been developed which effectively characterize the RCP resistance of plastic piping materials. These include: the small-scale steady state (S4 test under ISO13477) and full scale RCP test (FST under ISO 13478). Given the cost effectiveness and widespread availability of the S4 test as compared to the full scale RCP test, it is generally the preferred test method.

The S4 test method involves conditioning pipe specimens at a specified test temperature (32°F). The pipe specimen(s) is then subjected to an impact designed to initiate a fast-running longitudinal crack. Prior to performing a series of S4 tests, initiation tests are performed on unpressurized pipe specimens, typically five (5) nominal diameter ( $d_n$ ) in length, which are impacted at a striker velocity of  $15\text{m/s} \pm 5\text{m/s}$  to generate a crack length of at least one (1) pipe diameter in length.

Once the initiation conditions have been satisfied, a series of iterative S4 test are performed at varying internal test pressures but at a constant test temperature (32°F). The crack length,  $a$ , is measured for each test condition (test pressure, test temperature, striker velocity, etc). If the crack length is less than 4.7 times the nominal diameter ( $a < 4.7(d_n)$ ), then the crack is defined as an “arrest”. Conversely, if the crack length equals or exceeds 4.7 times the nominal diameter ( $a \geq 4.7(d_n)$ ), then the crack is defined as “propagation”. The maximum test pressure at which the criteria (ratio of crack length as compared to the outside diameter) have been satisfied is noted as the S4 critical pressure.

The full scale test (FST) is considerably more arduous and involves taking a long length of PE pipe specimen and placing it in a trench with cooling water circulating around it to maintain the

appropriate test temperature. The pipe is then buried to simulate recommended installation conditions. The pipe is pressurized by air and a crack is initiated by a steel blade in the initiation zone which has been conditioned to -60°C. If the crack extends over 90% of the specimen length, the crack is considered to have propagated. If the crack stops at a distance less than 90% of the test specimen length, the crack is considered as an “arrest”.

Historically, the consensus technical opinion within the technical community was that the correlation between the S4 test and the FST was a factor ranging from 3 to 10. However, in the late 1990’s, theoretical calculations based on gas dynamics theory, correlations between the S4 critical pressure and the full-scale RCP critical pressure and MAOP were developed as shown below.

$$p_c = ((3.6 \times p_{c,S4}) + 2.6) \times 14.5 = \text{psig} \quad (\text{Eqn. 1})$$

Where:  $P_{c,S4}$  = critical pressure at 32°F using the S4 test (bars)

Attempts were made to confirm the S4-full scale correlation factor with limited success. The conclusion of confirmation testing was that the correlation factor yielded a conservative approximation of full scale critical pressure from S4 test results and the additional refinement was unnecessary and impractical.

Additionally, the European community imposed a convention whereby the estimate full scale critical pressure is related to the maximum allowable operating pressure according to the following relationship:

$$p_c = 1.5 \times MAOP \quad (\text{Eqn. 2})$$

The technical rationale in establishing this relationship is unknown.

The veracity of these relationships has been the subject of significant technical debate within the technical community here in the US and the ASTM F17.60 subcommittee which governs ASTM D2513 requirements.

Since their inception, the technical community has maintained that the above correlations are based on gas dynamic principles and that these correlations are material independent. In order to validate whether or not the above correlations hold true for all materials, both Evonik-Degussa and UBE Industries have performed comprehensive series of S4 test and full-scale tests using their respective PA12 pipe materials. The results demonstrate that the above correlations are specific to PE materials and that the PA12 has an entirely different set of constants. This is also confirmed by work performed by Arkema for their Polyamide 11 (PA 11) pipe material as well.

The remaining sections of this report outline the RCP test data, the correlation functions for the PA12 pipe material, and the resulting maximum pressure limitations to ensure sufficient safeguards against RCP.

## RESULTS OF RCP TESTING (S4 and FULL-SCALE)

As previously noted, a series of S4 tests were performed at various laboratories and full-scale tests were performed at Advantica to test the veracity of the current ISO 4437 correlations and their applicability for PA12 piping systems developed by both Evonik-Degussa and UBE Industries.

A comprehensive battery of S4 tests were performed using two different pipe sizes at 4 different laboratories. While there was some level of data scatter, overall the results were consistent with expectations. The results of the testing are summarized in Table 1 below for each supplier.

Pipe Size	S4 Critical Pressure
110 mm SDR-11 (4-inch)	2.7 – 6.0 bar (39 – 87 psig)
170 mm SDR-11 (6-inch)	3.2 – 3.8 bar (46 – 55 psig)

**Table 1: Summary of the S4 test data developed at 4 different laboratories**

In addition, a comprehensive battery of full-scale tests was performed for the 110mm and 170mm pipe sizes to test and reconcile the correlation functions for the PA 12 piping materials. The results of the testing are summarized in Table 2 below.

Pipe Size	Full-Scale Critical Pressure
110 mm SDR-11 (4-inch)	$\geq 30$ bar ( $\geq 435$ psig)
170 mm SDR-11 (6-inch)	$\geq 25$ bar ( $\geq 363$ psig)

**Table 2: Summary of the Full-Scale test data developed at 4 different laboratories**

Based on the cumulative results of the S4 testing and Full-Scale testing, the same methodology used to develop the ISO 4437 correlations for PE was employed for the PA12 pipe material. The results of the analysis demonstrated that the resulting correlation function for the PA12 pipe material is not the same as for PE materials.

The resulting correlation for the PA 12 pipe material was determined to be:

$$p_{c,PA12} = ((7.8 \times p_{c,S4}) + 6.8) \times 14.5 = \text{psig} \quad (\text{Eqn. 3})$$

Where:  $P_{c,S4}$  = critical pressure at 32°F using the S4 test (bars)

$$p_{c,PA12} = 1.5 \times MAOP \quad (\text{Eqn. 4})$$

It is important to note, the resulting correlation for the PA 12 pipe material is consistent with the correlation developed for the Polyamide 11 (PA 11) pipe material.

## CONCLUSIONS

At the present time, no RCP requirements exist for any thermoplastic materials in use in natural gas distribution systems in the US. However, comprehensive understanding with respect to the RCP failure mechanism is of significant interest and is the subject of considerable discussion within the U.S. natural gas distribution community.

To date, the majority of research into understanding the rapid crack failure mechanism has been performed in Europe and is reflected in the established requirements in the relevant ISO documents. Within the ISO community, a considerable effort has been expended attempting to understand the relationship between full scale rapid crack propagation test results as determined by ISO 13477 (S4 testing) and the test results obtained from testing according to ISO 13478 (full scale testing). This is evidenced by the work being performed under the auspices of the ISO TC 138 SC5 WG2 which specializes in the evaluation and establishment of rapid crack propagation test methods.

According to the recommendation of this task group, rapid crack propagation test results can be obtained by either the S4 test or the full scale test. By applying the appropriate correlation factors or functions to the S4 test results, the full scale test values can be estimated. However, in the event of dispute, the full scale test is the referee test method based on the test values obtained by performing testing in accordance to ISO 13477.

Applying this logic to the PA12 data, the conclusion can be drawn that the full scale critical pressure for PA12 obtained by testing according to a modified ISO 13477 test method are well above the value necessary to safely operate a natural gas system at 200 psig.

**COMPREHENSIVE TESTING TO VALIDATE THE  
IN-SERVICE PERFORMANCE AND AGEING  
CHARACTERISTICS OF POLYAMIDE 12  
(PA 12) PIPING SYSTEMS FOR HIGH PRESSURE GAS  
DISTRIBUTION APPLICATIONS**

Prepared by:  
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May 15, 2008

## **BACKGROUND**

The use of plastic piping systems for gas distribution applications is governed by Title 49 CFR Part 192 which prescribes a series of minimum requirements to ensure safe long term performance. Through reference, CFR Part 192 Appendix B incorporates the requirements contained in ASTM D2513 with respect to both the short term and long term performance considerations.

Since 2004, the Polyamide 12 (PA 12) suppliers (Evonik-Degussa and UBE Industries), have been engaged in a comprehensive program to perform the necessary testing in order to validate the safe long term performance of Polyamide 12 (PA 12) piping systems for high pressure application.

In addition to the guidance within 49 CFR Part 192 requirements, sound engineering practices and previous experiences were employed to develop an effective hybrid approach consisting of laboratory evaluations and field demonstrations to validate the performance characteristics of the PA 12 piping systems. The results of comprehensive testing to characterize the mechanical, chemical, and physical properties of the PA 12 material amply demonstrate that the PA 12 material conforms to all relevant requirements contained within ASTM D 2513. As a result, the ASTM Committee F17 on Plastic Piping Systems approved the inclusion of PA 12 within Annex A5 of ASTM D2513.

While the results of laboratory testing are necessary and help to characterize the material specifications, an important technical consideration for any material relates to its in-service performance for the intended application. That is, while the material specifications help to ensure that a product has good overall stability with respect to short term and long term properties, the real issue is how well it performs under actual field conditions, and if there are any special construction and maintenance requirements which need to be established for the PA12 piping system.

Subsequently, the PA 12 suppliers supported a comprehensive hybrid approach to better understand the actual in-service performance of PA 12 piping systems operating at higher pressures. A series of laboratory tests were performed with actual pipe specimens subjected to a combined stress states resulting from both the internal pressure and effects of add-on stresses including rock impingement, excessive bending strain, and earthloading. In addition and more importantly, a series of field trials were performed to corroborate actual in-service performance under various types of add-on stress states with the data developed as part of the laboratory testing. Following the successful trial installations, additional comprehensive testing was performed on actual PA12 pipe specimens removed from service to evaluate any potential impact to the mechanical properties resulting from the various in-service conditions. The remaining sections of this summary report outline the results of the testing and evaluation.

## EVALUATION OF IN-SERVICE STRESS STATES ON PA12 PIPING SYSTEMS

An important consideration in the overall acceptance of any new thermoplastic piping system relates to its performance under exposure to various types of in-service conditions for the intended application.

Over 40 years of safe operating experience with the use of plastic piping materials has demonstrated that the long term performance is not dependent solely on the plastic piping materials ability to withstand failures due to internal pressure. A complete analysis and evaluation of the stresses arising from various conditions must be taken into account and evaluated. Therefore, in any effort to effectively validate the performance of plastic piping systems, it is imperative to characterize the materials ability to mitigate localized stress intensifications resulting from various types of secondary effects which can potentially lead to failures in the “brittle” manner due to slow crack growth (SCG) mechanism.

While there are several industry accepted tests which help to characterize a materials resistance to the SCG mechanism, e.g. PENT test or three-point bend sector test, these tests are merely useful relative indices and do not correlate with actual field performance considerations with respect to piping system considerations.

In general, a typical gas distribution piping system can be potentially subjected to various types of additional stresses (add-on stresses or secondary stresses) which act in combination with the effects of internal pressure. Subsequently, additional battery of laboratory tests was performed to evaluate the effects of add-on stresses on the PA12 piping systems including:

- Effects of surface scratches
- Effects of rock impingement
- Effects of excessive bending strain
- Effects of earthloading (compressive stresses on the pipe)

The results of the testing are summarized in Table 1 below and were consistent with expectations – the PA12 piping system can safely operate at the increased stress levels.

Secondary Stress	Test Criterion	Results
Surface Scratches and Notches	<i>Varying notch depths = 20%, 30%, 50%</i> Test Pressure = 290 psig Test Temperature = 80C	Test time > 1000 hours with No Failures at 20% scratch depth
Rock Impingement	<i>½” Indentation</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Earth Loading	<i>5% Deflection of Outside Diameter</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Bending Strain	<i>20 times OD</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures

**Table 1: Summary of test conditions to simulate effects of secondary stresses**

## **EVALUATION OF FIELD PERFORMANCE CONSIDERATIONS**

In addition to the aforementioned laboratory testing, a series of actual simulated field trials were performed in various parts of the United States to validate the safe operations of the PA12 piping systems at the increased stress levels taking into account the combined effects of internal pressures and various add-on stresses. A summary of the various field trials is presented in Table 2 below and detailed discussions for each of the trials (1-4) can be found in Final Report issued by the Operations Technology Development, NFP. A summary report for the recently completed installations at the City of Mesa, WE Energies, and DTE (Michcon) is being finalized and will be submitted separately.

The cumulative results of the various installations amply validate the ability the field performance of the PA12 piping systems. Specifically, the results show:

- PA12 piping systems can be safely installed and operated at higher operating pressures up to 250 psig using various pipe sizes ranging from 2-inch through 6-inch IPS
- Conventional construction and maintenance practices specific to PE piping systems are readily transferrable to PA12 piping systems
- An array of procedures (butt heat fusion and electrofusion) and appurtenances (transition fittings, mechanical fittings, etc) were successfully utilized

Location	Key Criterion	Description	Status and Comments
February 2005 – Chicago, IL (GTI)  Pressure = 250psig	Indigenous Backfill	2-inch SDR11 Sections of squeezed pipe and heat fusion joints 2-inch transition fittings installed.	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections removed at 30 months for ageing studies</li> <li>• Planned removal after 36 months of exposure</li> </ul>
October 2006 - Chicago, IL (GTI)  Pressure = 250psig	Rocky Soil (80/20 mix of rocks and clay soil which is compacted over the pipe)	6-inch SDR11 Sections contain electrofusion couplings and heat fusion joints 6-inch transition fittings installed	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections scheduled for removal following 24 months of ageing</li> </ul>
October 2006 - Chicago, IL (GTI)  Pressure = 250psig	Flowable Fill (highly compressive strength backfill)	6-inch SDR11 Sections contain electrofusion couplings and heat fusion joints 6-inch transition fittings installed	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections scheduled for removal following 24 months of ageing</li> </ul>
October 2006 – Buffalo, NY (National Fuel)  Pressure = 250psig	Bending Strain (90 times pipe OD at joint and 20 times pipe OD on straight pipe)	6-inch SDR11 Sections contain electrofusion couplings and heat fusion joints 6-inch transition fittings installed 6-inch mechanical saddle installed and tapped at 250 psig.	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections removed at 14 months for ageing studies</li> <li>• Planned removal after 36 months of exposure</li> </ul>
March 2008 – Phoenix, AZ (City of Mesa)  Pressure = 160 psig (pressure based on HDB rating at 140F)	Static and dynamic and vehicular loading	4-inch SDR11 Sections connected using electrofusion couplings 4-inch transition fittings and mechanical saddles installed	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections to be removed at 12 months and 24 months</li> </ul>
April 2008 – Wisconsin (WE Energies)  Pressure = 250 psig	Cold climate	4-inch SDR11 Sections connected using electrofusion couplings and butt fusion 4-inch transition fittings and mechanical saddles installed	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections to be removed at 12 months and 24 months</li> </ul>
May 2008 – Detroit, MI (DTE, MichCon)  Pressure = 330 psig	Cold climate and high pressure limit	4-inch SDR11 Sections connected using electrofusion couplings and butt fusion 4-inch transition fittings and mechanical saddles installed	<ul style="list-style-type: none"> <li>• No leaks</li> <li>• Sections to be removed at 12 months and 24 months</li> </ul>

**Table 2: Summary of the field demonstrations simulating increased stress levels**

## **AGEING CHARACTERISTICS OF THE PA12 PIPING SYSTEMS**

Based on the preceding discussions, the cumulative results of the laboratory testing and simulated field trials amply demonstrate the ability of the PA12 piping systems to operate at the intended higher operating pressures under the combined influence of internal pressures and various types of secondary in-service stress states. However, an additional key piece of information was to quantify the ageing characteristics of the PA12 piping system as a function of various geographic and climatic conditions. Consequently, samples have been removed from the initial GTI trial performed during February 2006 and the National Fuel trial and subjected to a comprehensive battery of tests to investigate potential impact to the mechanical and physical properties of the PA12 pipe material.

The results of the ageing testing were consistent with expectations. There was no evidence of premature oxidative degradation, and the cumulative data amply demonstrates that the overall stability of the respective PA12 resin suppliers product is technically sound with respect to the mechanical and physical properties. The results of the testing are summarized in Appendix A and B for both Evonik-Degussa and UBE Industries PA12 pipe material, respectively.

## **SUMMARY AND CONCLUSIONS**

Since 2004, both Evonik-Degussa and UBE have been engaged in a comprehensive program to perform the necessary testing in order to validate the safe long term performance of PA 12 piping systems for high pressure application.

Based on the comprehensive results of the testing to characterize the mechanical, chemical, and physical properties of the PA 12 piping material by both suppliers, the PA 12 material has been successfully incorporated into ASTM D2513 in Annex A5 during 2006.

While the results of laboratory testing are necessary and help to characterize the material specifications, an important technical consideration for any material relates to its in-service performance for the intended application. That is, while the material specifications help to ensure that a product has good overall stability with respect to short term and long term properties, the real issue is how well it perform under actual field conditions and if there are any special construction and maintenance requirements which need to be established.

As a result, a comprehensive series of tests under both laboratory conditions and actual in-service conditions were performed to evaluate the effects of various types of secondary stress states and in-service conditions. The results of the testing were consistent with expectations. Specifically,

- The results demonstrated that the PA12 piping systems (pipe, fittings, and appurtenances) can safely operate at pressures up to 250 psig (SDR 11 pipe sizes and use of a 0.40 design factor) in conjunction with various types of secondary stresses acting on the pipe.

- The use of existing construction and maintenance practices specific to PE piping systems can be readily used with PA 12 piping systems. Results of the testing demonstrated that strong PA 12 joints can be made using the qualified PA 12 joining procedures; the results confirmed the ability that the use of “squeeze-off” does not adversely impact the long term performance of the pipe; the results conformed the safe use and operations of other types of appurtenances installed on the piping systems including transition fittings, mechanical fittings, and electrofusion fittings.
- There were no adverse reactions to the PA 12 piping systems as a result of exposure to various types of in-service stresses acting on the piping system or environmental considerations such as premature oxidative degradation after nearly three years of exposure to in-service conditions

**Appendix A**  
**EVALUATION OF THE AGEING CHARACTERISTICS OF THE**  
**VESTAMID LX9030 PA 12 PIPE MATERIAL**

The cumulative results of these respective installations amply demonstrate the effectiveness of using PA12 piping systems over a range of sizes (2-inch through 6-inch) and increased operating pressures (250 psig). Moreover, the installation in Europe also demonstrated the applicability of using coiled pipe which provides additional installation cost savings due to the reduction in the number of joints that are required over the length of the installation.

While the ability to safely install and operate the PA 12 piping systems is critical, an equally important consideration is to ensure that there are no deleterious effects to the PA12 pipe material over the intended design life as a result of exposure to various types of in-service conditions or environmental factors once installed.

## **HEAT AGEING CHARACTERISTICS**

### **Laboratory Simulations – Evonik-Degussa VESTAMID LX9030**

During January 2006, Arkema issued a technical report which outlined unexpected degradation of its Polyamide 11 (PA 11) natural gas piping material. The report indicated that after being installed for slightly over two years, the PA 11 pipes which were installed at several locations throughout the United States were strongly discolored and revealed brittle fracture in burst pressure tests.

A detailed investigation by Arkema demonstrated that the pipes experienced oxidative degradation which started at the outside of the pipe and moved over time into direction of the inner side. The results of the testing performed by Arkema clearly identified that this phenomenon was a direct result of the high contents of phosphoric acid in the PA 11 base material which together with the yellow bismuth vanadate pigment and air-born oxygen led to a heavy catalytical decomposition of the polymer.

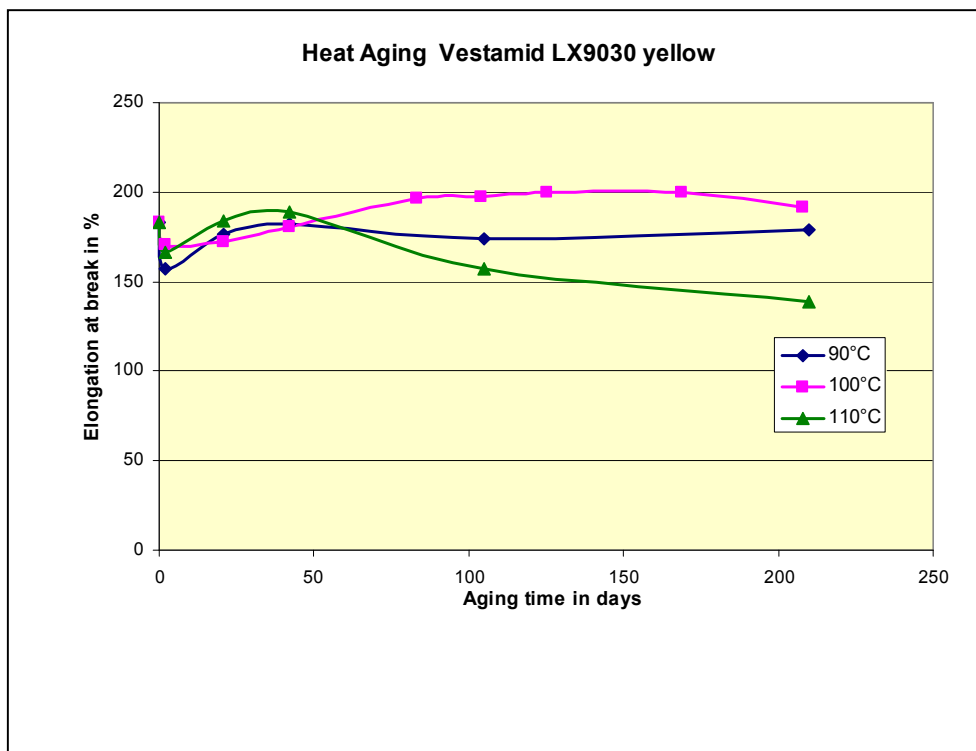
This rapid decomposition of the PA 11 resin was simulated in the laboratory by simple heat aging tests on the material at 110°C using tensile test bars as specimen. The data showed that there was a dramatic loss of elongation at break values within just 8 days. In order to resolve this issue, Arkema subsequently reformulated its PA 11 resin by replacing the bismuth vanadate pigment with a yellow Cd-pigment<sup>1</sup>.

In order to demonstrate that this particular issue is specific to PA 11 and not Polyamides in general, Evonik Degussa performed comprehensive testing of its Polyamide 12 (PA 12) piping to verify that there are no adverse degradation issues with its PA 12 resin formulation..

Comprehensive heat ageing tests in air of its VESTAMID LX9030 yellow material in accordance with accepted methodology for performing ageing exposure experiments. Several injection moulded tensile specimens of VESTAMID LX9030 yellow were subjected to heat ageing at 90, 100 and 110 °C under air, exactly as done for the critical PA 11 formulation . Specimens were removed after 2, 21, 42, 105 and 208 days, respectively, and tensile tests performed. The results of the testing are summarized in Figure 1 below.

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<sup>1</sup> In conformity with international environmental protection legislation, Evonik Degussa does not use pigments containing heavy metals like lead or cadmium compounds)



**Figure 1: Heat ageing of VESTAMID LX9030 yellow**

Figure 1 presents the mean value of the elongation at break as a function of the ageing time. The results confirm the excellent resistance of the VESTAMID LX9030 yellow material. Specifically, the data demonstrates that the LX9030 yellow material does not experience a significant loss in the elongation at break property following exposure to a temperature of 110 °C after 208 days.

## **2” SDR-11 pipes from field installation on GTI pipe farm**

While the preceding discussions clearly demonstrates the outstanding heat ageing resistance of the VESTAMID LX9030 yellow resins, additional tests were performed using actual pipe specimens from the first GTI installation which was installed during 2005 (see previous section) and specimens were recovered following 30 months of exposure to buried underground conditions.

After approximately 30 months of exposure to in-service conditions (two complete seasonal cycles), several sections of the VESTAMID PA 12 pipe were removed during August 2007. In order to characterize the impact of the in-service condition on various types of construction practices, additional specimens were removed which included butt heat fusion joints and sections which were intentionally “squeezed”. A new pipe section was spliced in and the entire test section was re-pressurized back to 250 psig.

The removed pipe sections were visually inspected for any evidence of premature ageing or oxidative degradation. No visual change of the color was observed as compared to an uninstalled pipe section which was stored indoors. Following the visual inspection, comprehensive mechanical and ageing tests were performed as outlined in the sections to follow.

#### **Mechanical test – 2” SDR-11 pipes from GTI pipe farm**

Tensile testing was performed on Type I specimens in accordance to ASTM D638. The results of the testing were compared to tensile testing results presented in the original GTI test report (control specimens). In addition to the tensile testing, additional testing was performed to characterize the moisture content. This is an important consideration in being able to interpret the mechanical test results because moisture has a plasticizing effect on Polyamides; however, this is entirely reversible. For the various test specimens, the water content measured by Karl Fischer Method was 0.8%, which corresponds to the equilibrium for PA 12 at 73 °F (23 °C).

The results of the tensile testing were consistent with expectations. There was a small-scale reduction in the tensile strength at yield. This is a direct consequence of the hygroscopic nature of Polyamides. The tensile test results demonstrated that the stress at yield was equal to 4630 psi (34 MPa) with an elongation at break of greater than 200%. This corresponds to a reduction of the stress at yield by 8% compared to the value reported in the GTI report on non-aged specimens. Again, it can be reasonably inferred that this is a direct consequence of the plasticizing effect due to the influence of moisture over two complete seasonal cycles.

Based on the measured moisture content values of 0.8% corresponding closely to the equilibrium point, it can be reasonably inferred that the reduction in the mechanical strength has reached an asymptotic limit. This inference can be validated based on data developed on pipe specimens from subsequent planned removals.

In addition to testing pipe specimens, additional test specimens were prepared from the butt fusion joint and tested in accordance to ASTM D638 requirements. The results were again consistent with expectations. The stress at yield was 5003 psi (34.5 MPa) and clearly demonstrates that there is no significant difference in the mechanical strength of the PA12 butt heat fusion joint as compared with data from pipe samples. All of the specimens failed in a ductile manner outside the welding area.

#### **Ageing Characterization**

##### **2” SDR-11 pipes from GTI pipe farm**

To determine the potential for polymer degradation of the VESTAMID PA 12 pipe material removed from in-service caused by oxidation or other impacts, the viscosity number (VN) was measured along various points in the pipe wall in accordance to ISO 308. The VN correlates with the molecular weight of Polyamide 12 and is an established procedure in the industry. Samples were prepared from the outer and inner surface and from the center of the pipe wall to investigate any surface effects. The measured VN shows little to no change in comparison to the control samples (Fig. 2). Also, there was no discernable difference in the VN along the various points in the pipe wall.

### **6-inch SDR11 pipes from National Fuel – Buffalo, NY**

As previously discussed, the premature oxidative degradation experienced by the PA11 resin was not isolated to a particular geographic territory, rather, the PA11 pipes from each of the respective installations throughout the United States were removed due to the premature oxidative degradation.

To ensure that the VESTAMID LX9030 pipe material maintained excellent overall stability regardless of the installation setting, additional tests were performed on PA 12 pipe specimens recovered from the National Fuel installation in Buffalo, NY following 14 months of in-service experience. Like the pipe specimens from the GTI installation, the viscosity number (VN) was measured at three points along a cross section of the pipe. The results were consistent with expectations. There was no significant change in the VN along the pipe wall from the samples recovered from National Fuel: VN is 98% of the reference data as shown in Table 2 below.

Cumulatively, this data along with the mechanical testing clearly demonstrates that there is no adverse effect of the VESTAMID LX9030 yellow formulation and its ability to safely perform under various types of in-service conditions and in various geographic areas.

### **UV - Weathering under real conditions**

Additionally, UV - Weathering tests are also concurrently ongoing at Atlas Weathering in Phoenix, AZ which represent worst-case conditions. The first set of pipe samples have been tested after real-time exposure of 6 and 12 month. There was no visual evidence of any deleterious effects after real time ageing, i. e. no change in color was observed.

### **Mechanical testing - U.V. Weathering and Real Conditions**

Like the previous discussions, tensile tests were also performed. The water content of the pipe sample was also measured. The tensile test shows a stress at yield of 39 MPa and an elongation at break of >200 % (Fig. 3). This again is consistent with expectations. As previously discussed, the absorption of moisture by Polyamide materials tends to have a plasticizing effect which is reversible. The higher strength of the samples compared to the GTI samples may be a direct results of the “drying” of the specimens under intense UV exposure leading to a reduced moisture content value.

### **Ageing – U.V. Weathering under Real Conditions**

The ageing of the pipe sample was also tested by determining the VN as described in 3.1.2. The measured VN shows little to no change in comparison to the control sample (Fig. 2). Also, there were no differences in VN number values along the pipe wall.

				2" SDR-11 pipe	2" SDR-11 pipe 2,5 years	6" SDR-11 pipe 14 months
Properties	Unit	Standard	Require- ments	GTI report	Installation on GTI pipe farm	Installation at National Fuel
<b>Tensile Strength, 74 °F</b>						
- Strength at yield	psi / MPa	ASTM D2513/ D638		5370 / 37	4931 / 34	-
- Elongation at yield	%			12	14	-
- Strength at break	psi / MPa			6457 / 44	5800 / 40	-
- Elongation at break	%		> 150	219	> 200	-
<b>Water content</b>	%				0,80	-
<b>Relative Viscosity number (VN) along wall thickness</b>						
outer surface	%	ISO 307			100	98
center	%			100*	100	98
inner surface	%				99	98

**Fig. 2: Test results after installation at GTI and National Fuel VESTAMID LX9030 yellow**

			0,5 years at Atlas	1 year at Atlas
Properties	Unit	Specification		
<b>Tensile Strength, 74 °F</b>				
- Strength at yield	psi / MPa	ASTM D2513 / D638	5656 / 39	5661 / 39
- Elongation at yield	%		13	13
- Strength at break	psi / MPa		6960 / 48	7628 / 52
- Elongation at break	%		> 200	> 200
<b>Relative Viscosity number (VN) along wall thickness*</b>		ISO 307		
outer surface	%		100	100
center	%		100	100
inner surface	%		100	100

\* compared to result of the control sample

**Fig. 3: Test results after UV weathering at Atlas in Phoenix VESTAMID LX9030 yellow**

# **Appendix B**

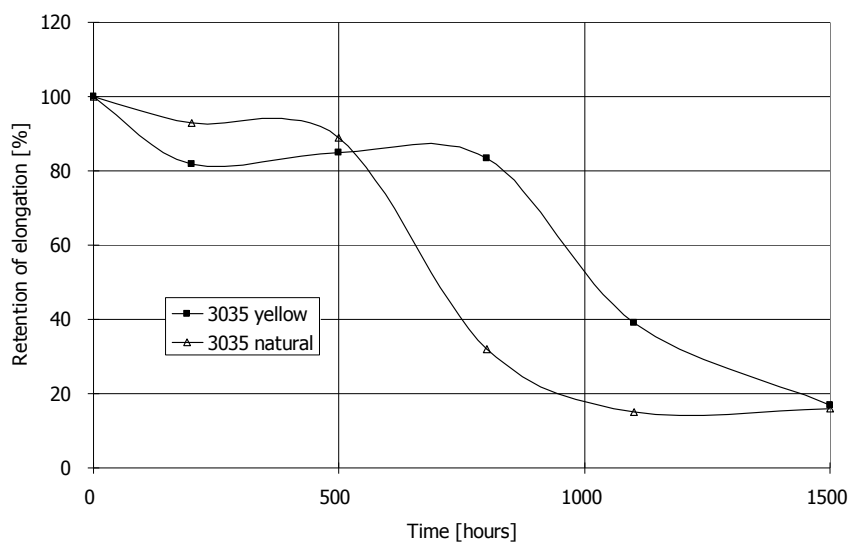
## **EVALUATION OF THE AGEING CHARACTERISTICS OF THE UBESTA 3035 PA 12 PIPE MATERIAL**

## EVALUATION OF AGEING CHARACTERISTICS – UBESTA 3035

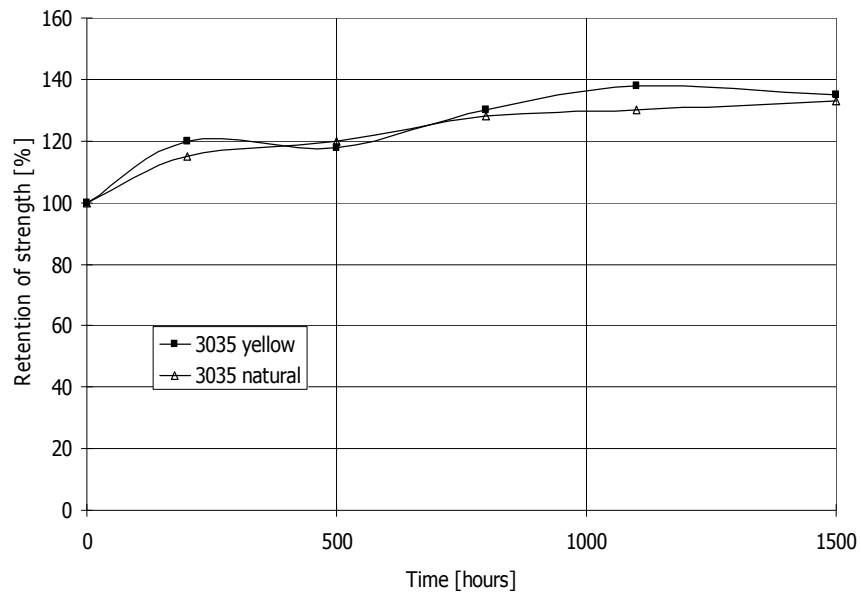
### Accelerated Weathering

In order to demonstrate that premature oxidative degradation due to the reaction of components of the formulation do not occur with UBESTA 3035, a series of accelerated weathering studies were performed. Both heat ageing and accelerated weathering testing were performed. The results indicate no premature oxidative degradation of UBESTA 3035. A description of the testing follows.

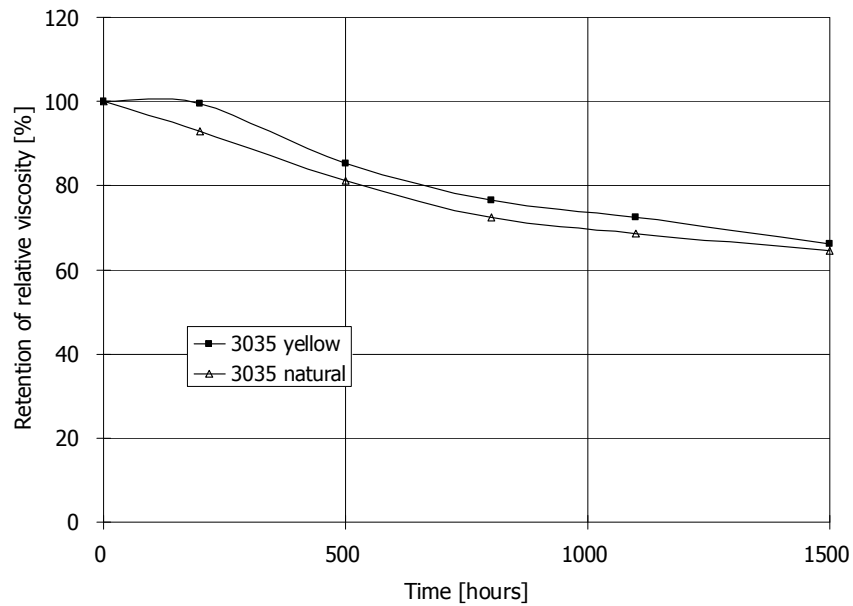
ASTM D638 tensile test specimens were exposed to hot air ageing at 130°C. After exposure the samples were characterized for tensile properties according to ASTM D638 and relative viscosity according to ASTM D789. Figures 1, 2 and 3 present the results.



*Figure 1. Heat Ageing @ 130°C – Retention of Elongation*



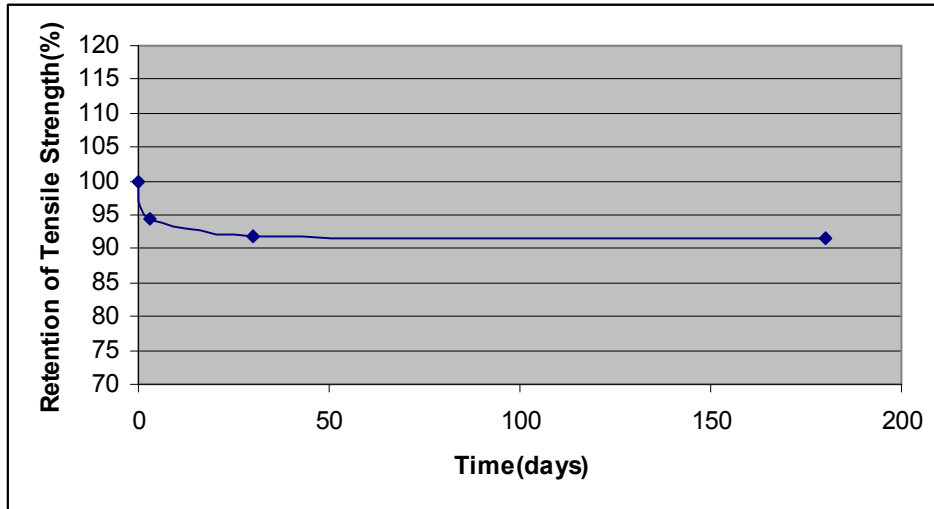
*Figure 2. Heat Ageing @ 130°C-Retention of Tensile Strength*



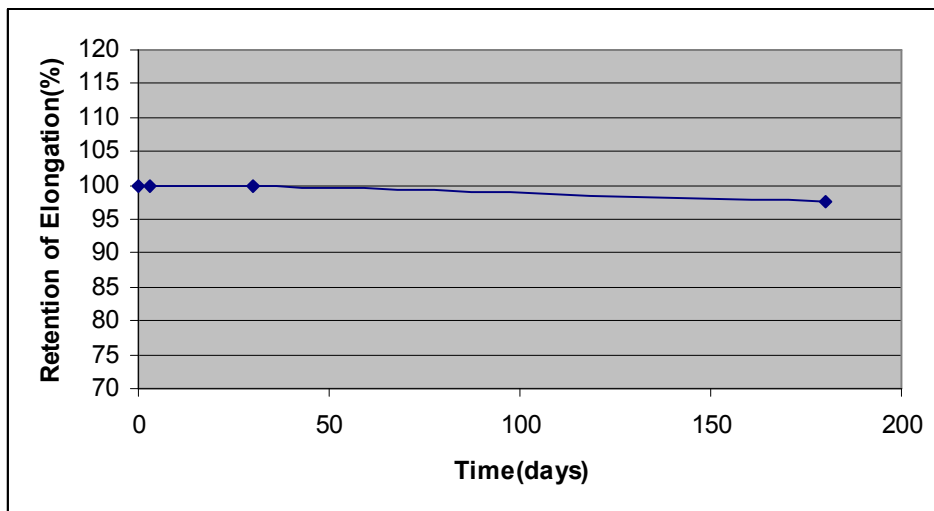
*Figure 3. Heat Ageing @ 130°C – Retention of Relative Viscosity*

Figures 1 and 2 indicate that there is no significant reduction in tensile properties due to the presence of the yellow pigment. Figure 3 indicates that there is no significant deterioration in molecular weight as compared to an unpigmented grade of UBESTA 3035. From the data, the conclusion can be drawn that there is no unexpected reduction in properties due to thermal oxidative degradation.

Additionally, Xenon Arc weathering according to ASTM D2565-99 was performed to assess the effect of exposure to UV irradiation. ASTM D638 Type 1 specimens were exposed for a total of 180 days. This corresponds to 645 MJ/m<sup>2</sup> total irradiation. Figures 4 and 5 present the results of testing on the exposed specimens.



*Figure 4. Xenon Arc Weatherometer Exposure – Retention of Tensile Strength*



*Figure 5. Xenon Arc Weatherometer Exposure – Retention of Elongation*

The data indicates no significant degradation of tensile properties after accelerated UV exposure.

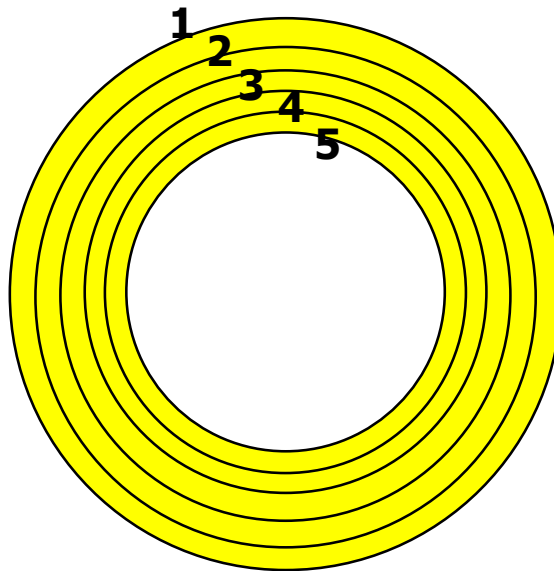
Overall, the results indicate that there is no degradation of properties of UBESTA 3035 after exposure to accelerated ageing conditions.

### Characterization of Samples Removed from Field Installations

As described in a previous section of this document, a series of field installations have taken place as part of the Phase 1 GTI project. Subsequently, aged samples have been removed from two installations for evaluation. Two inch SDR 11 pipe samples of UBESTA 3035 were removed from the February 2005 installation at GTI's pipe farm in August 2007, 30 months after the original installation. In January 2008, 6" IPS SDR 11 pipe samples of UBESTA 3035 were removed from the National Fuel private property installation. These samples were removed 15 months after the original installation.

Both 30 month aged samples from the GTI pipe farm and 15 month aged samples from the National Fuel installation were forwarded to UBE Industries, Ltd. laboratories in Japan. Samples were characterized for relative viscosity to determine if any deterioration in molecular weight had occurred since the original installation. Additionally, samples were characterized for tensile properties according to ASTM D638. The results were compared to test results from uninstalled reference pipe produced from the same lot of base material from the same extrusion run. The test results are presented below.

Samples were characterized for relative viscosity according to the Japanese standard JIS K6920. A modification to the normal procedure was employed. Rather than determine the relative viscosity of the bulk sample, 5 samples were microtomed through the wall of the sample and the relative viscosity of each respective layer determined. The intent was to determine if any degradation gradient was present through the pipe wall due to environmental exposure. Figure 6 illustrates the sample preparation scheme. Table 1 and 2 presents the relative viscosity measurements for reference pipe and samples from the GTI installation and National Fuel installation respectively.



*Figure 6. Layers of Pipe Sample for Relative Viscosity Measurement – Layer Thickness = 1.2mm*

<b>Zone</b>	<b>Description</b>	<b>Relative Viscosity, Uninstalled Reference Pipe</b>	<b>Relative Viscosity, 30 Months In-Service</b>
1	0-1.2mm from outside wall	2.44	2.34
2	1.2 – 2.4 mm from outside wall	2.47	2.39
3	Center 5.4 mm	2.42	2.46
4	1.2-2.4 mm from inside wall	2.43	2.43
5	0-1.2 mm from inside wall	2.41	2.44
Cross Section	Entire wall thickness	2.43	2.41

*Table 1. Relative Viscosity – 30 Month GTI Samples; 2” IPS SDR11 Pipe*

<b>Zone</b>	<b>Description</b>	<b>Relative Viscosity, Uninstalled Reference Pipe</b>	<b>Relative Viscosity, 15 Month In-Service Pipe</b>
1	0-3.1mm from outside wall	2.45	2.45
2	3.1-6.2mm from outside wall	2.46	2.45
3	Center 153mm	2.44	2.44
4	3.1-6.2mm from inside wall	2.45	2.47
5	0-3.1mm from inside wall	2.47	2.49
Cross Section	Entire wall thickness	2.45	2.46

*Table 2. Relative Viscosity – 15 Month National Fuel Samples; 6” IPS SDR11 Pipe*

The data would indicate no significant degradation of the molecular weight of either samples removed from the GTI installation after 30 months or the National fuel samples removed after 15 months.

The moisture content of the 2” IPS SDR 11 pipe removed from the installation was measured and determined to be 1.03%. The slight differences in the relative viscosity of the outer two layers of the removed pipe as compared to the reference pipe is due to the effect of absorbed moisture on the sample. This is consistent with expectations and does not indicate any significant degradation of the material nor deterioration in performance. The effect of moisture in the solid state on Polyamide 12 is completely reversible.

Additionally, tensile testing according to ASTM D638 was performed on the 30 month GTI samples. The results are presented in Table 3. Testing on the 15 month National Fuel samples is ongoing.

	<b>Control</b>	<b>Reference</b>	<b>Pipe removed after 30 months</b>
Tensile stress @ yield	6612 psi	5684 psi	5235 psi
Elongation @ yield	10.0%	9.7%	10.5%
Tensile strength @ break	7772 psi	7410 psi	6656 psi
Elongation @ break	254%	272%	227%
Relative Viscosity	2.42	2.43	2.41
Moisture content	-	0.78%	1.03%

*Table 3. Physical Property Profile*

The control data is taken from 2: SDR 11 samples submitted to GTI in April 2004 at the onset of the initial evaluation. The reference pipe data was generated on uninstalled pipe samples from the same lot of base material and the same extrusion run as the installed samples. The slight difference in tensile properties between the control, reference and removed samples can be attributed to the difference in moisture content between the three samples. Once more, this change in tensile properties does not indicate a permanent reduction in physical properties. The effect of moisture is completely reversible. The consistency of the relative viscosity values for the three samples supports the contention that no permanent degradation has taken place.

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

Technical Reference Report  
April 2004 – December 2005

Prepared by

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OTD Project No. 20042

for

Operations Technology Development Group

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December 15, 2005

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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 be a very promising candidate material for high pressure gas distribution applications.

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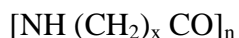
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## Section 1

### Polyamide 12 and History of Use

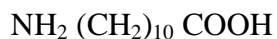
#### 1.1 Polyamides

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:



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:



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

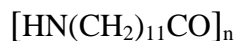
The development of Polyamide 12 was started in the 1960's. The first commercial production of Polyamide 12 began in the 1970's at what is now Degussa in Marl, Germany. At the present there are four commercial suppliers of Polyamide 12 worldwide:

- 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:



## 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:



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<sup>1</sup> 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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<sup>1</sup> 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:

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

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.

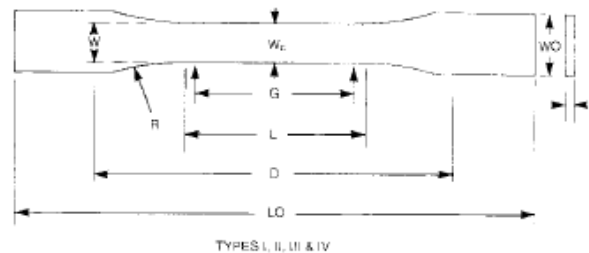
## 2.2 Tensile Strength Determination

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)	$\pm 0.5$ (0.02)
L – length of narrow sections	57 (2.25)	$\pm 0.5$ (0.02)
WO – width overall	19 (0.75)	$\pm 6.4$ (0.25)
LO – length overall	165 (6.5)	No max
G – gage length	50 (2.00)	$\pm 0.25$ (0.010)
R – radius of fillet	76 (3.00)	$\pm 1$ (0.04)
D - Distance between grips	115 (4.5)	$\pm 5$ (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 °F 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  $9f^{1/2}$  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.

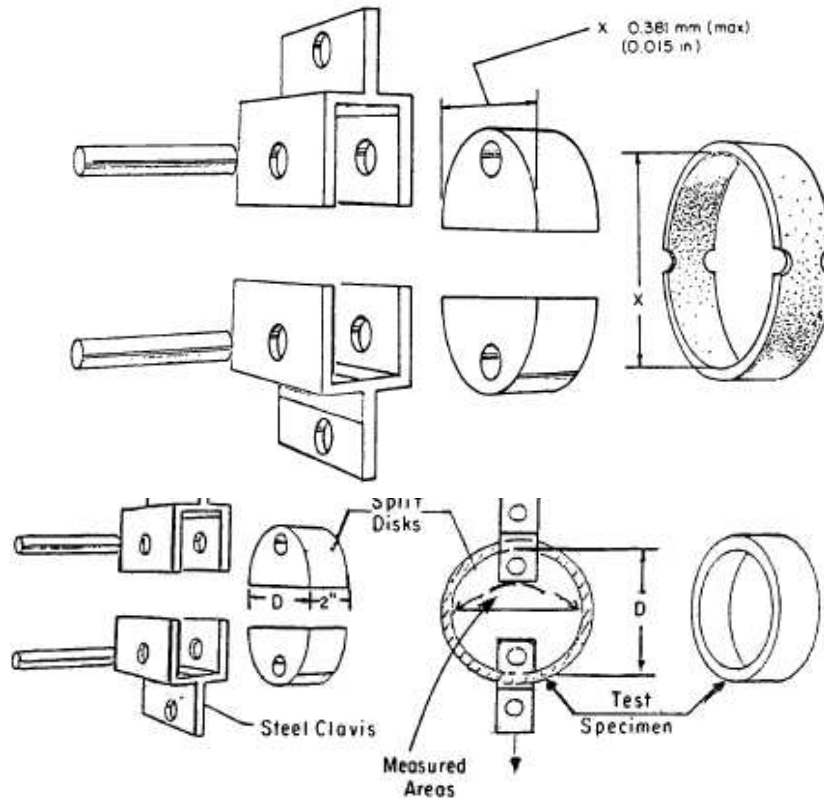
PA12 Supplier	Flexural Modulus
UBE	231.6 ksi
Degussa	213.6 ksi
EMS	173 ksi

**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 °F 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 Glycol	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.

Chemical	Polyamide 11 (PA11)	
	Change in Weight (%)	Change in Tensile Yield Strength (%)
Mineral Oil	< 0.5	- 12
Tertiary Butyl Mercaptan	< 0.5	- 12
Methanol	< 5	- 35
Ethylene Glycol	< 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**

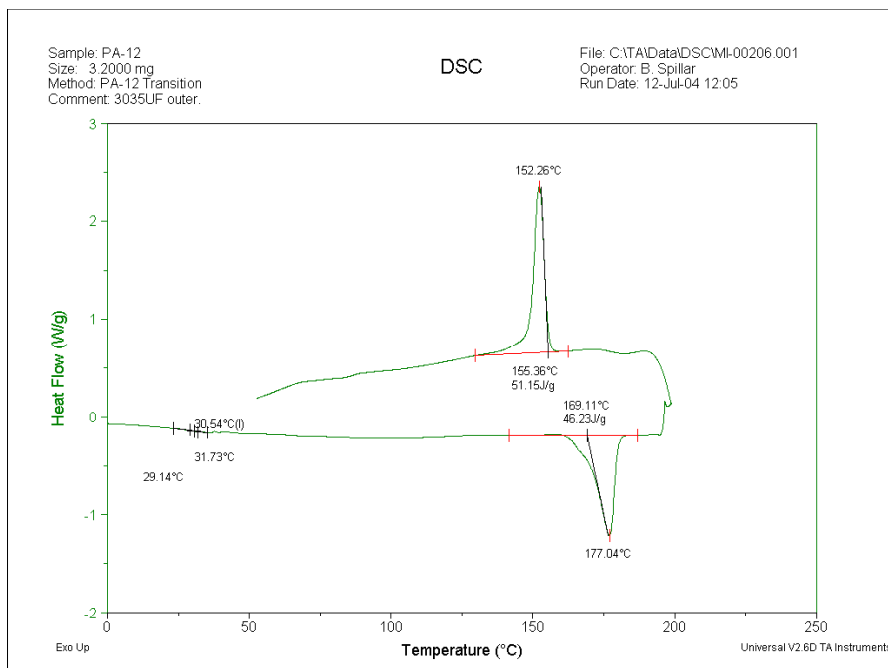
From Tables 10 and 11, it can be seen that the most significant reduction in tensile strength occurred under exposure to methanol and toluene in methanol. This is as expected given that methanol is a polar solvent. From fundamental chemistry, polar solvents tend to have a chemical affinity to polar materials. 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.

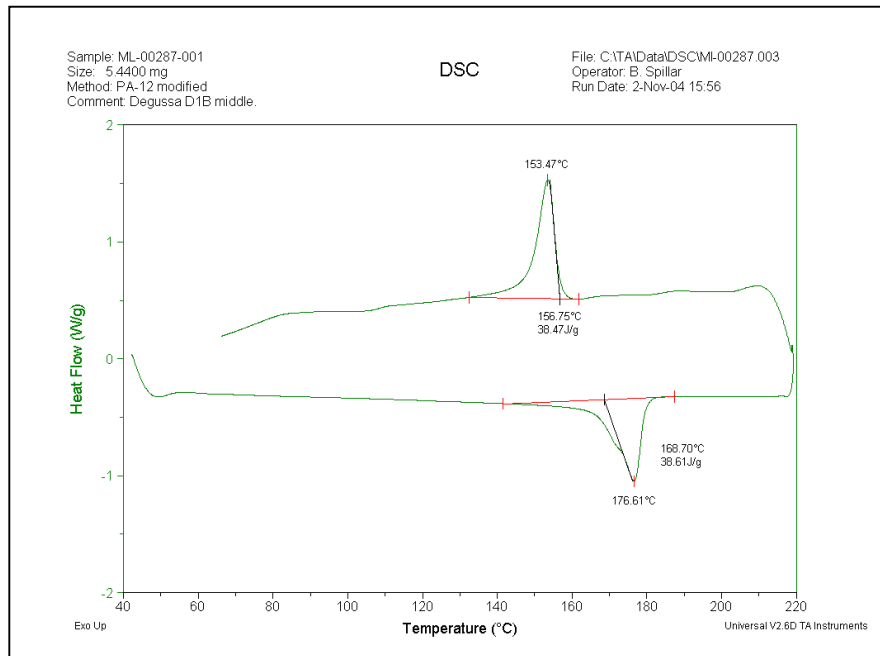
## 2.6 Melt Characteristics

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  $-50^{\circ}\text{C}$  and  $700^{\circ}\text{C}$ . 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.

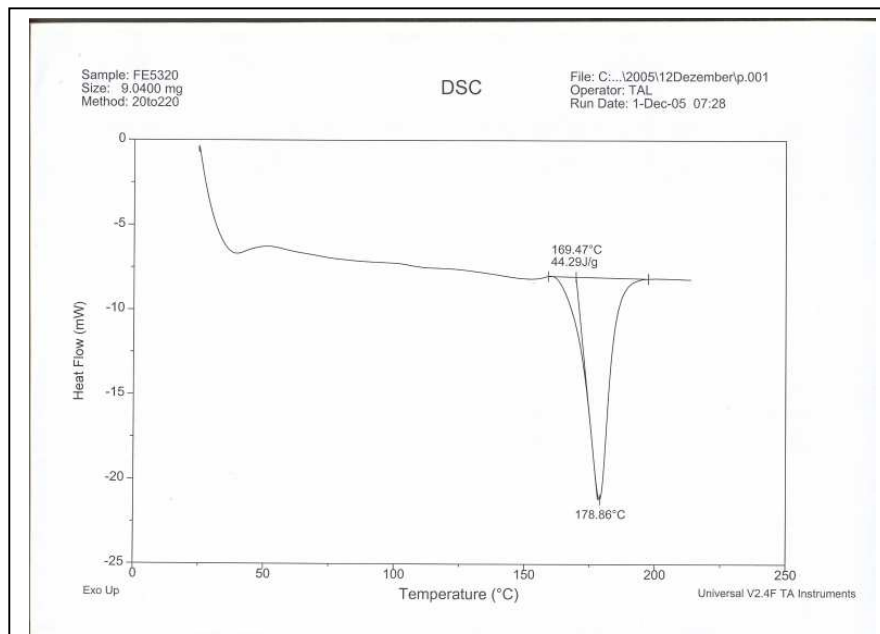
The key property of interest for this study is the melting point of polyamide 12. All three lots were tested to determine their melting points. Measurement of the melting point of the pipe was performed in accordance with ASTM D 3418 [14]. A 12.0 mg sample size was tested using  $350^{\circ}\text{C}$  at  $10^{\circ}\text{C}/\text{min}$ . 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**

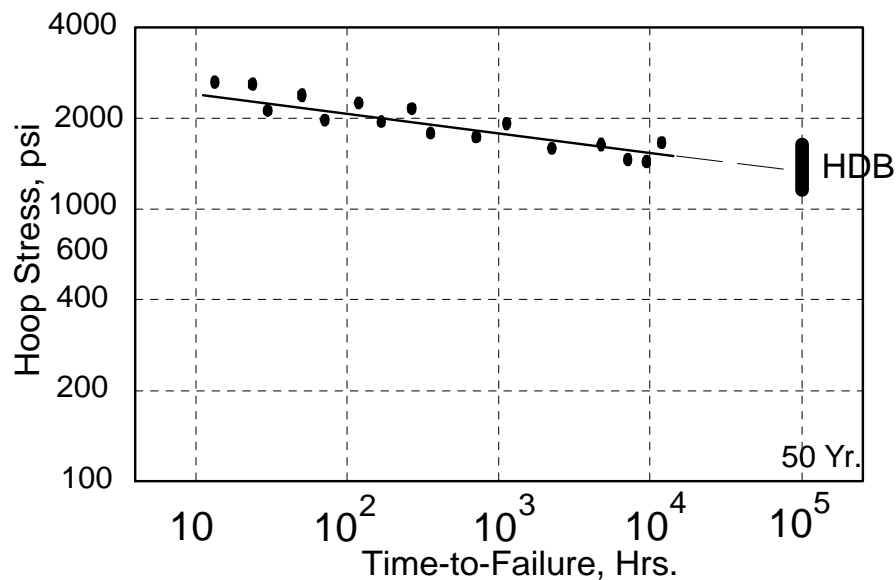
During the early 1960's, the Plastics Pipe Institute (PPI) proposed a new method for forecasting the long term strength of thermoplastic pipe materials. Soon after the industry adopted this method to stress rate their materials. In 1967, after the addition of some refinements, ASTM adopted the PPI proposal 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°F (23°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 (8.62 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]:

$$MAOP = \frac{2 \bullet HDB \bullet F}{SDR - 1} \quad (2)$$

where:

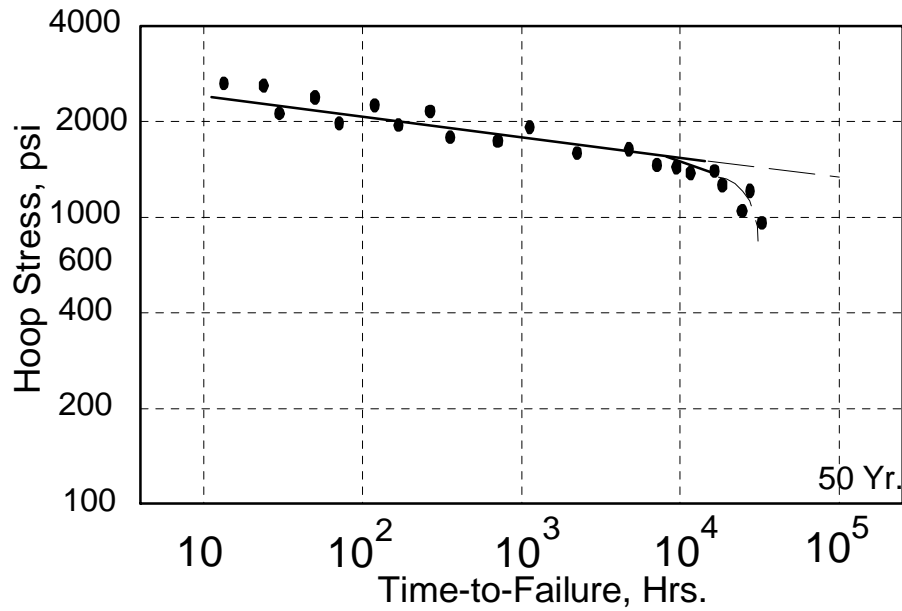
HDB = Hydrostatic Design Basis, psi  
F = Design Factor, 0.32 for gas piping  
SDR = Standard Dimension Ratio defined as the  
ratio of the mean outside diameter to the  
minimum wall thickness

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.

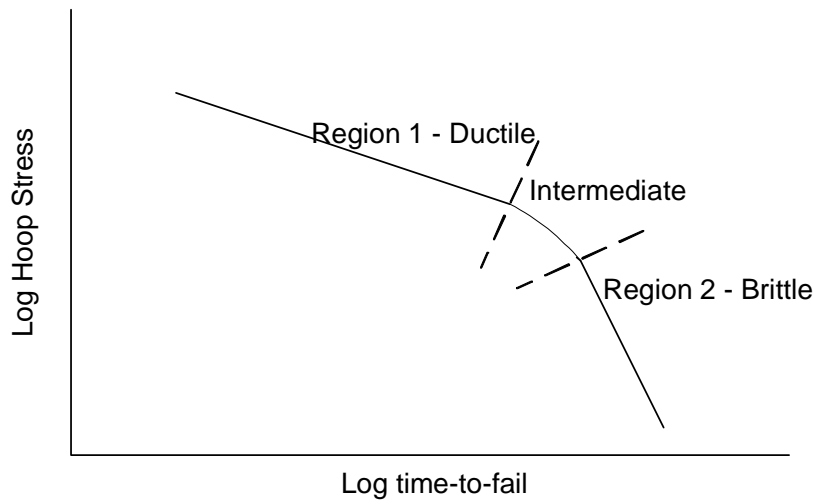
### 3.2 Validation of the Hydrostatic Design Basis

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:

$$\begin{aligned} a_T &= \exp[0.109(T_i - T_s)] \\ t_s &= t_i a_T \end{aligned} \quad (1)$$

$$\begin{aligned} b_T &= \exp[-0.0116(T_i - T_s)] \\ p_s &= \frac{p_i}{b_T} \end{aligned} \quad (2)$$

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.

$$\begin{aligned} a_T &= \exp[0.109(T_i - T_s)] \\ a_T &= \exp[0.109[80 - 23]] \\ q_T &= 499.2 \end{aligned} \quad (3)$$

$$\begin{aligned} \frac{t_s}{a_T} &= t_i \\ \frac{100,000}{499.2} &= t_i \\ t_i &= 200.3 \end{aligned}$$

and,

$$\begin{aligned} b_T &= \exp[-0.0116(T_i - T_s)] \\ b_T &= 0.516 \\ p_s &= \frac{p_i}{b_T} \\ p_i &= 825.9 \end{aligned} \quad (4)$$

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 criteria 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).

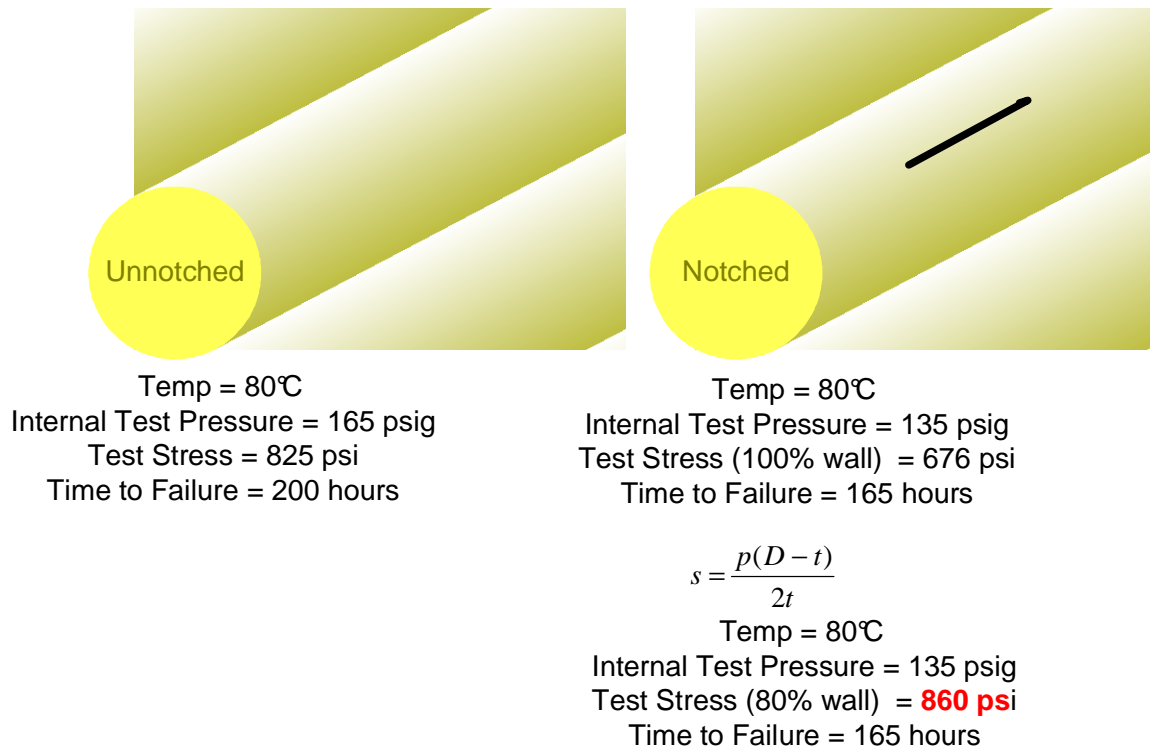
### **3.3 Notched Pipe Testing**

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 underscored by the fact that the test specimens within ASTM D2837 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.

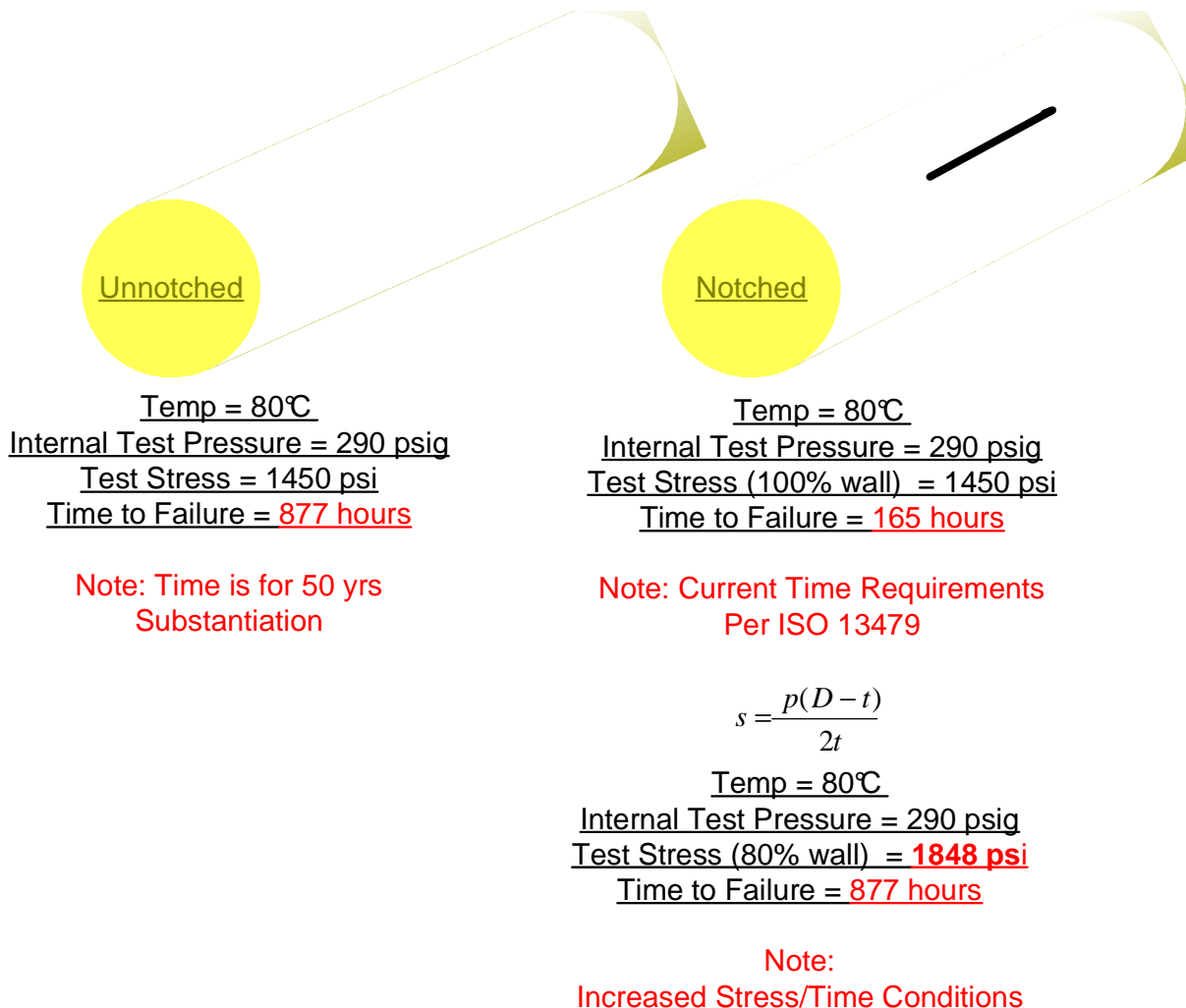
In order to gain a better understanding of the test protocol and its meaningfulness, consider the case of high density PE piping: the validation protocols within ASTM D2837 require that the pipe specimens must not fail prior to 200 hour test time at an applied stress of 825 psi (165 psig). In the case 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.



**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.



**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 than 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:

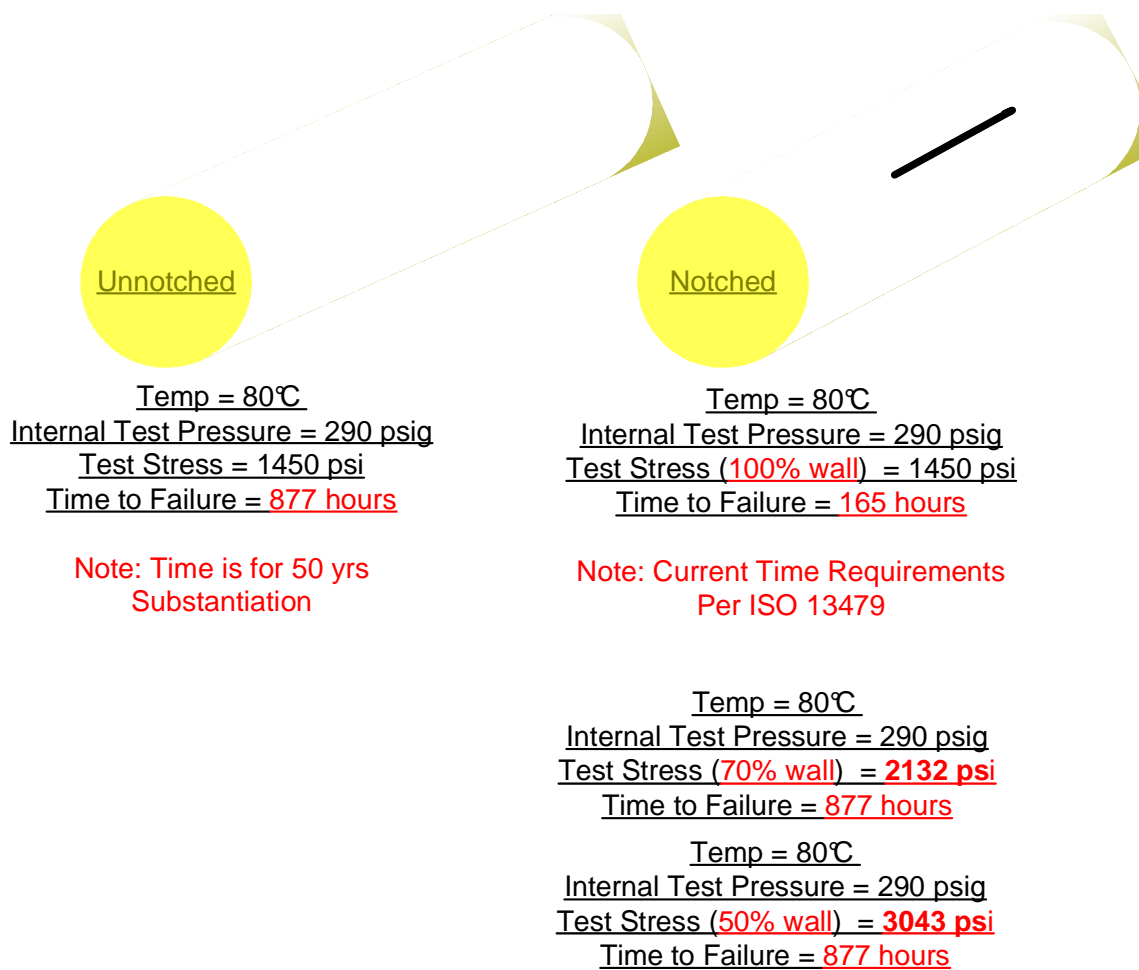
PA 12 Supplier	Test Criterion	Time to Failure (hrs)
UBE	Test Pressure: 290 psig (20 bars) Notch Depth: 20% Stress at remaining ligament: 1848 psi Test Temperature: 80°C 50-year substantiation time: 877 hours	> 2000
Degussa		> 2000
EMS		> 2000

**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.



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

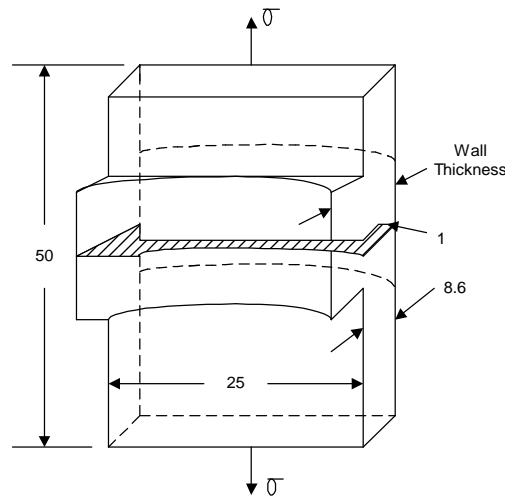
Conditions	Test Conditions	Time to Failure (hrs)
Condition 1 (UBE PA12)	Test Pressure: 290 psig (20 bars) <b>Notch Depth: 30%</b> <b>Stress at remaining ligament: 1848 psi</b> Test Temperature: 80°C 50-year substantiation time: 877 hours	> 2000 hours with no failures
Condition 2 (UBE PA12)	Test Pressure: 290 psig (20 bars) <b>Notch Depth: 50%</b> <b>Stress at remaining ligament: 1848 psi</b> 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 ( $\sigma$ ). 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

In 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  $\pm$  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 <b>Applied Torque: 10 ft-lbs</b> Torque Hold: 60 sec Clamp Time: 10 min
2		Heater Iron Temp: 500F Heat Soak: 60 sec <b>Applied Torque: 20 ft-lbs</b> 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 <b>Applied Torque: 10 ft-lbs</b> Torque Hold: 90 sec Clamp Time: 10 min
4		Heater Iron Temp: 500F Heat Soak: 60 sec <b>Applied Torque: 20 ft-lbs</b> 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 <b>Heat Soak: 60 sec</b> Applied Torque: 10 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min
6		Heater Iron Temp: 500F <b>Heat Soak: 90 sec</b> 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 <b>Heat Soak: 60 sec</b> Applied Torque: 20 ft-lbs Torque Hold: 60 sec Clamp Time: 10 min
6		Heater Iron Temp: 500F <b>Heat Soak: 90 sec</b> 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.

<b>Evaluation of Fusion Parameters – UBE PA12 Pipe</b>				
	<b>Condition 1</b>	<b>Condition 2</b>	<b>Condition 3</b>	<b>Condition 4</b>
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
<b>Evaluation of Fusion Parameters – Degussa PA12 Pipe</b>				
	<b>Condition 1</b>	<b>Condition 2</b>	<b>Condition 3</b>	<b>Condition 4</b>
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<sup>2</sup> 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  $3,5 \text{ GJ/m}^2$ . Therefore, the selected irradiance output of the Xenon Test Chamber was set to  $0.35 \text{ W/m}^2$  at  $340 \text{ nm}$ , with a typical irradiation value of  $41.5 \text{ W/m}^2$  between  $300 - 400 \text{ nm}$ , 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<sup>2</sup>, i.e., 120 minutes (2 hours) of exposure per one cycle.

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<sup>2</sup> In Florida, the UV solar radiation per year at a 45-degree tilt angle is about  $286 \text{ MJ/m}^2$  or about 4.76% of the total solar irradiation ( $6000 \text{ MJ/m}^2$ ). Therefore, the minimum total UV irradiation is about  $166 \text{ MJ/m}^2$ . If the cycle is 2 hours long and puts out an irradiance of  $41.5 \text{ W/m}^2$ , the number of cycles to reach  $166 \text{ MJ/m}^2$  is 555. Assuming 12 cycles/day, the total irradiation of  $166 \text{ MJ/m}^2$  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 <sup>2</sup> )
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**

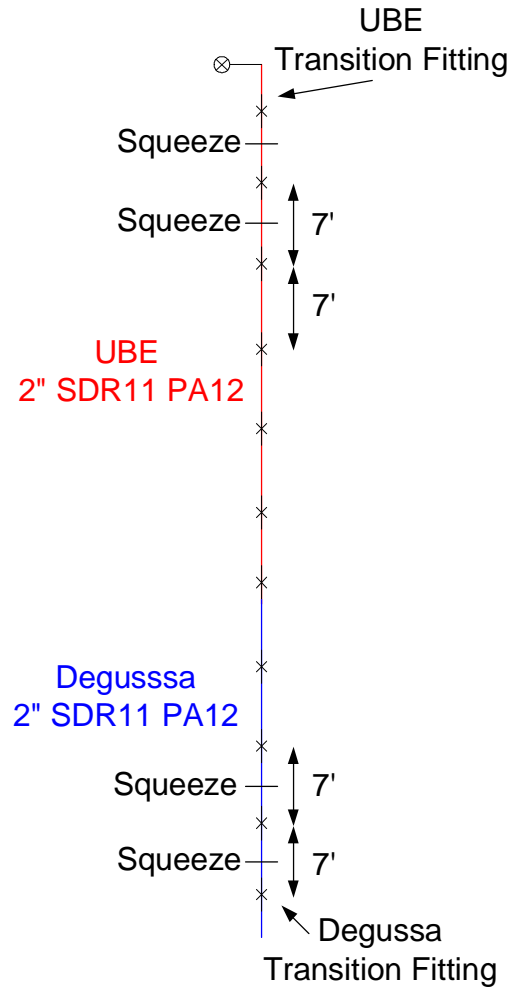
## **Section 5**

### **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.

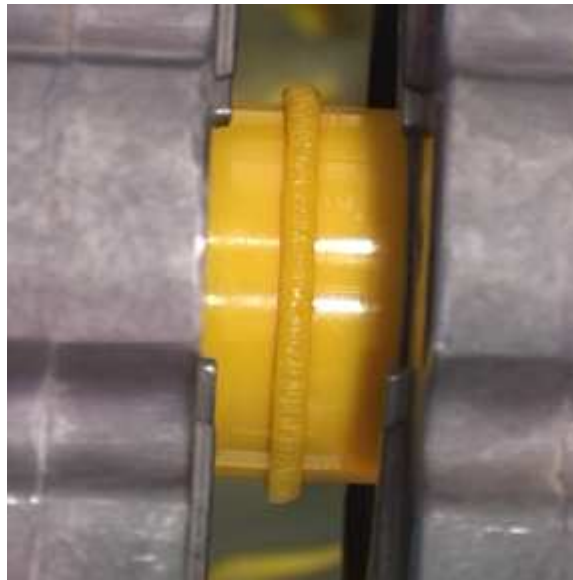


The PA12 pipes were supplied in 7 foot stick lengths, which were fused together using PA12 joining procedures developed as part of this program, See Section 4.1:

- 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.

# **EVALUATION OF POLYAMIDE 12 (PA12) FOR HIGH PRESSURE GAS DISTRIBUTION APPLICATIONS**

Final Report  
April 2004 – December 2006

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for

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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. While the increase in the maximum design pressure limitations represents some level of improvement, conventional grades of polyethylene (PE) materials still cannot operate at significantly higher pressures without adversely compromising flow capacity – the corresponding wall thickness increases significantly. As a result, there exists a desire on the part of utilities to leverage the benefits of thermoplastic piping materials and extend the range of operating pressures and use of larger diameter piping systems which does not sacrifice flow capacity.

One promising family of thermoplastic materials is the Polyamide materials. Since 2004, with the support of Operations Technology Development (OTD) group and respective PA12 resin suppliers (Degussa, UBE, and EMS), the Gas Technology Institute (GTI) has been engaged in a comprehensive program to validate the feasibility for the use of Polyamide 12 (PA12) in high pressure gas distribution applications through comprehensive laboratory testing and field experiments. Specifically, the program was aimed at developing the required short term mechanical property data per applicable ASTM and industry standards and specifications, characterizing the long term performance considerations, evaluating various construction, maintenance, and operating practices, and obtaining valuable in-service performance related experience.

Based on the cumulative results of the overall program, the PA12 material from Degussa and UBE appears to be a technically feasible candidate material for high pressure gas distribution applications. In particular, the test data demonstrates that their respective PA12 material conforms to all relevant requirements contained within ASTM D2513 and its respective annexes specific to polyamide. Moreover, the results of long term sustained pressure testing at elevated temperatures demonstrate that these respective PA12 materials exhibit very high slow crack growth resistance characteristics. Specifically, the results demonstrate that these respective PA12 materials perform very well under the combined influence of internal pressure and other secondary stresses including surface scratches, rock impingement, earth loadings, and bending strain. Evaluation of construction, maintenance, and operating considerations demonstrates that conventional practices already in use for PE materials can be readily transferred to PA12 piping systems. In the context of this program, it has been shown that there are no deleterious

effects of squeeze-off with respect to long term performance considerations. In addition, qualified PA12 joining procedures have been developed consistent with the both industry and code requirements. Finally, the results of several installations under actual conditions validate the use of PA12 piping systems at operating pressures up to 250 psig.

This report presents a comprehensive summary of each of the technical aspects of the program: technical data for short term and long term performance characteristics, development and evaluation of O&M practices, and results of field evaluations. The cumulative results demonstrate that the PA12 materials from the various resin suppliers appear to be a very promising candidate material for high pressure gas distribution applications.

It is important to emphasize that only an abbreviated summary of the technical discussions are presented here. Detailed discussions with respect to the testing protocols and results are contained within a separate report entitled “*Technical Reference on the Physical, Mechanical, and Chemical Properties of Polyamide 12 (PA12) for High Pressure Gas Distribution Applications*” issued to OTD and the respective PA12 resin suppliers during December 2005.

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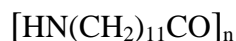
<b>OTD -</b>	<b>Operations Technology Development</b>
<b>AGA -</b>	<b>American Gas Association</b>
<b>PPI -</b>	<b>Plastics Pipe Institute</b>
<b>GTI /GRI -</b>	<b>Gas Technology Institute/ Gas Research Institute</b>
<b>DOT -</b>	<b>Department of Transportation</b>
<b>PHMSA -</b>	<b>Pipeline Hazardous Materials Safety Administration</b>
<b>ASTM -</b>	<b>American Society of Testing and Materials</b>
<b>ISO -</b>	<b>International Standards Organization</b>
<b>CFR -</b>	<b>Code of Federal Regulations</b>
<b>MAOP -</b>	<b>Maximum Allowable Operating Pressure</b>
<b>SDR -</b>	<b>Standard Dimension Ratio (OD/wall thickness)</b>
<b>ID -</b>	<b>Inside Diameter</b>
<b>OD -</b>	<b>Outside Diameter</b>
<b>SCG -</b>	<b>Slow Crack Growth</b>
<b>RCP -</b>	<b>Rapid Crack Propagation</b>
<b>LTHS -</b>	<b>Long Term Hydrostatic Strength</b>
<b>HDB -</b>	<b>Hydrostatic Design Basis</b>
<b>HDS -</b>	<b>Hydrostatic Design Stress</b>
<b>LCL -</b>	<b>Lower Confidence Limit</b>
<b>MDPE -</b>	<b>Medium Density Polyethylene (PE2708)</b>
<b>HDPE -</b>	<b>High Density Polyethylene (PE4710)</b>
<b>PA11 -</b>	<b>Polyamide 11</b>
<b>PA12 -</b>	<b>Polyamide 12</b>

## Section 1 Introduction and Background

Prior to detailed discussions about the overall program, it is important to present a high level overview with respect to the chemical make up of Polyamide 12, its potential use, and the technical approach that was utilized throughout the program.

### 1.1 What is Polyamide 12 and History of Use?

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. Polyamides are named by the number of carbon atoms in the monomer unit. The general formula for polyamides like Polyamide 12 is:



The properties of PA12 are significantly affected by the presence of amide groups in the polymer backbone which gives them their unique property profile. The amide group is characterized by the following formula:



The frequency of occurrence of the amide groups (amide density) differentiates 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 also affects the moisture absorption characteristics.

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

In the late 1970's, The Australia Gas Light Company (AGL) identified a need to rehabilitate corroded cast iron service lines in New South Wales, Australia. At the time, polyamide 11 (PA11) 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 PA11 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-1980s, 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. At present, 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.

## 1.2 Polyamide 12 High Pressure Gas Distribution Applications

Recent demographic changes and rapid urbanization have presented significant challenges for gas utility companies to safely and effectively satisfy the Nations' ever growing energy needs. As a result, there is a tremendous desire on the part of gas utility companies to operate their gas distribution infrastructure to its maximum capabilities. This has been underscored by recent initiatives to increase the pressure limitations for plastic piping system up to 125 psig, as limited by its hydrostatic design basis and geometric characteristics. Moreover, the industry has been actively working to increase the design factor which is used in the formula for determining the design pressure. These initiatives notwithstanding, conventional grades of polyethylene piping will not be able to operate over a range of desired pressures and flow capacity considerations.

In general, Polyamide 12 (PA12) offers significant potential given its inherent mechanical, physical, and long term performance characteristics. Based on empirical test data for its long term hydrostatic strength, PA12 has an established hydrostatic design basis (HDB) rating of 3150 psi. Using this particular HDB rating for an SDR 11 pipe size with either a 0.32 or 0.40 design factor, the following maximum design pressure can be potentially realized:

DF = 0.32	DF = 0.40
$p = \frac{2S}{SDR - 1} \times 0.32$	$p = \frac{2S}{SDR - 1} \times 0.40$
$p = \frac{2(3150)}{11 - 1} \times 0.32$	$p = \frac{2(3150)}{11 - 1} \times 0.40$
$p = \frac{6300}{10} \times 0.32$	$p = \frac{6300}{10} \times 0.40$
$p = 200 \text{ psig}$	$p = 250 \text{ psig}$

(1)

Where:

P = design pressure, psig

S = long term hydrostatic strength as represented by the HDB rating, psi

SDR = standard dimension ratio (OD/t)

From Eqn (1), it is shown that an SDR11 PA12 piping system can potentially operate at pressures up to 250 psig on the basis of their inherent long term hydrostatic strength characteristics and the use of a 0.40 design factor. For additional capacity, gas utility companies can also choose to utilize an SDR13.5 (thinner wall for added capacity) piping system which can operate at 200 psig with a 0.40 design factor. Either scenario will permit gas utility companies to utilize PA12 piping systems as an effective alternative to steel piping systems.

### 1.3 Technical Approach

Title 49, Part 192 of the Code of Federal Regulations governs the minimum requirements for the safe use of plastic piping systems. While all of the respective sections are important, Part 192, through reference, requires that all thermoplastic piping materials suitable for use in gas distribution applications must conform to the requirements contained within ASTM D2513-98<sup>1</sup> specification entitled “*Standard Specification for Thermoplastic Gas Pressure Pipe, Tubing, and Fittings*” [1]. Within the main body of the ASTM D2513, there are several requirements that are applicable to all thermoplastic materials. Additional requirements are also 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. Finally, additional guidance for the introduction and use of new thermoplastic materials is also provided in a non-mandatory Appendix within ASTM D2513. These guidelines include the following:

- Conformity to ASTM D2513 requirements and establishing a ASTM product specification
- Establishing the materials’ long term hydrostatic strength through comprehensive long term sustained pressure testing at elevated temperatures per ASTM D2837 requirements and PPI TR-3 policies and procedures used to establish the hydrostatic design basis (HDB) rating
- Demonstrating at least 3-years of service-related experience to demonstrate that a particular material can safely be used for underground gas pressure piping without significant changes to its long term performance characteristics

In the context of this program, a comprehensive approach was utilized to address each of these key considerations. Specifically, the objective was to develop the necessary technical data to validate the feasibility for the use of PA12 piping systems at higher pressures consistent with the requirements and recommendations contained within ASTM D2513. Technical discussions with respect to the comprehensive testing which addresses each of these key considerations are presented in the respective sections to follow.

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<sup>1</sup> 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

## **Section 2**

### **Characterization of Performance Requirements**

In order to demonstrate conformity to ASTM D2513-98 requirements and its applicable Annexes/Appendices, GTI performed comprehensive testing and evaluation of the PA12 pipe materials supplied by each of the three respective PA12 resin suppliers including UBE (Japan), Degussa (Germany) and EMS (Switzerland). Arkema (France), the fourth supplier of PA12 did not participate in the program.

It is important to reiterate that only an abbreviated summary of the technical discussions are presented here. Detailed discussions with respect to the testing protocols and results are contained within a separate report entitled “*Technical Reference on the Physical, Mechanical, and Chemical Properties of Polyamide 12 (PA12) for High Pressure Gas Distribution Applications*” issued to OTD and the respective PA12 resin suppliers during December 2005.

#### **2.1 Material Performance Characteristics**

Per ASTM D2513, there are several test requirements to characterize the mechanical, physical, and chemical characteristics of a given thermoplastic material. These tests include:

- Minimum hydrostatic quick burst strength (ASTM D1598)
- Tensile strength determinations (ASTM D638 and ASTM D2290)
- Flexural Modulus (ASTM D790)
- Chemical resistance testing (ASTM D543)
- Melt point and Oxidation induction times (ASTM 3418)

The cumulative results of the various short term testing used to characterize the mechanical, chemical, and physical properties indicates that the PA12 materials from each of the three PA12 resin suppliers conforms to the requirements of the main body of ASTM D2513 and its respective Annexes. Specifically, the respective PA12 materials from each of the three resin suppliers either meets and/or exceed the requirements and compares well with the established PA11 requirements contained within Annex A5. On the basis of this and other test data provided in the following sections of this report, the PA12 material from UBE was successfully integrated within ASTM D2513 (2006 version).

#### **2.2 Determination of Long Term Hydrostatic Strength (HDB Rating)**

In addition to determining the mechanical, chemical, and physical properties of the PA12 material, additional comprehensive tests were performed to establish the long term hydrostatic strength, as represented by the materials' hydrostatic design basis (HDB) rating, pursuant to the requirements contained within ASTM D2513.

It is important to note that this particular activity was not a part of the OTD program and was carried out by each of the respective PA12 resin suppliers independently. The data and information has been provided to OTD within the context of the co-funding

arrangements established at the onset of the program. It is important to emphasize that the HDB rating is a necessary prerequisite for any new thermoplastic material in order for it to be used for gas distribution applications. The HDB rating is the long term strength rating that is substituted within the design formula used to calculate the design pressure.

For a given thermoplastic material, the long term hydrostatic strength (LTHS) is determined on the basis of comprehensive tests as outlined per ASTM D2837 requirements and PPI TR-3 policies and procedures. The LTHS is determined by subjecting several pipe specimens to long term sustained pressure testing at elevated temperatures over a 10,000 hour test time. The resulting data (stress-rupture) are analyzed by linear regression analysis to yield a best-fit log-stress versus log time-to-fail straight-line equation. On the basis of this equation, the material's mean strength at the 100,000 hour intercept (LTHS) is calculated. The resultant LTHS is correlated to an appropriate Hydrostatic Design Basis (HDB) category, as shown in Figure 1 below from ASTM D2837.

NOTE 1—The LTHS is determined to the nearest 10 psi. Rounding procedures in Practice E 29 should be followed.

Range of Calculated LTHS Values		Hydrostatic Design Basis	
psi	(MPa)	psi	(MPa)
190 to < 240	( 1.31 to < 1.65)	200	( 1.38)
240 to < 300	( 1.65 to < 2.07)	250	( 1.72)
300 to < 380	( 2.07 to < 2.62)	315	( 2.17)
380 to < 480	( 2.62 to < 3.31)	400	( 2.76)
480 to < 600	( 3.31 to < 4.14)	500	( 3.45)
600 to < 760	( 4.14 to < 5.24)	630	( 4.34)
760 to < 960	( 5.24 to < 6.62)	800	( 5.52)
960 to <1200	( 6.62 to < 8.27)	1000	( 6.89)
1200 to <1530	( 8.27 to <10.55)	1250	( 8.62)
1530 to <1920	(10.55 to <13.24)	1600	(11.03)
1920 to <2400	(13.24 to <16.55)	2000	(13.79)
2400 to <3020	(16.55 to <20.82)	2500	(17.24)
3020 to <3830	(20.82 to <26.41)	3150	(21.72)
3830 to <4800	(26.41 to <33.09)	4000	(27.58)
4800 to <6040	(33.09 to <41.62)	5000	(34.47)
6040 to <6810	(41.62 to <46.92)	6300	(43.41)
6810 to <7920	(46.92 to <54.62)	7100	(48.92)

**Figure 1: Hydrostatic Design Basis Categories as a function of LTHS**

Using the aforementioned approach, as of preparation of this report, both the UBE and Degussa PA12 materials have an established HDB rating. The UBE material has undergone the complete 10,000 hours of required testing and has already received a *standard* listing of 3150 psi at 73F and 2500 psi at 140F within PPI TR-4/2006 listings. The Degussa PA12 material has completed nearly 8,000 hours of the required 10,000 hours of testing. It also has received an *experimental grade* listing (E-2) of 3150 psi at 73F and 2500 psi at 140F within PPI TR-4 listing. As of preparing this report, EMS has not provided any information with respect to their status of testing and/or the results.

### 2.3 Characterization of Slow Crack Growth Resistance

Over 40 years of field experience using polyethylene materials has demonstrated that the primary mode of in-service failures are due to the slow crack growth (SCG) failure mechanism.

Because 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 - viscoelastic behavior. As a result, 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.

Therefore, in order to ensure that only those materials exhibiting excellent SCG characteristics are utilized for gas distribution applications, ASTM D2513 prescribes several tests including validating the long term hydrostatic strength over a 50-year theoretical design life, PENT test, etc.

In the context of this particular program, several tests were performed consistent with the ASTM D2513 requirements. A brief summary of the results includes:

- Because there are no validation protocols specific to polyamide materials within ASTM D2513, the project team employed the same theoretical considerations that were used for PE materials. The primary assumption within ASTM D2513 is that materials which are used for gas distribution applications must demonstrate ductile performance over their intended design life – 50 years. Subsequently, ASTM D2513 requires additional long term sustained pressure testing at elevated temperature using specified test conditions for test time, temperature, and pressures. As a result, six specimens from both UBE and Degussa were subjected to comprehensive long term sustained pressure testing at 290 psig and 80C. The threshold test time was determined to be 1000 hours. It is important to note the significant degree of conservatism inherent within this approach – the test conditions are greater than the calculated values using the bidirectional shift functions, and are consistent with proposed International Standards Organization (ISO) requirements for PA materials. The results of the testing demonstrated that

there were *no failures* from either the Degussa or UBE pipe specimens at test times greater than 1500 hours.

- In addition to LTHS validation testing, comprehensive PENT tests were performed on injection molded plaques. The PENT test is useful relative index for a particular materials' resistance to the SCG failure mechanism. For all of the PA12 test specimens from both the Degussa and UBE pipe, there were *no failures* at times greater than 1000 hours at a test stress of 2.4mPa and 80C.

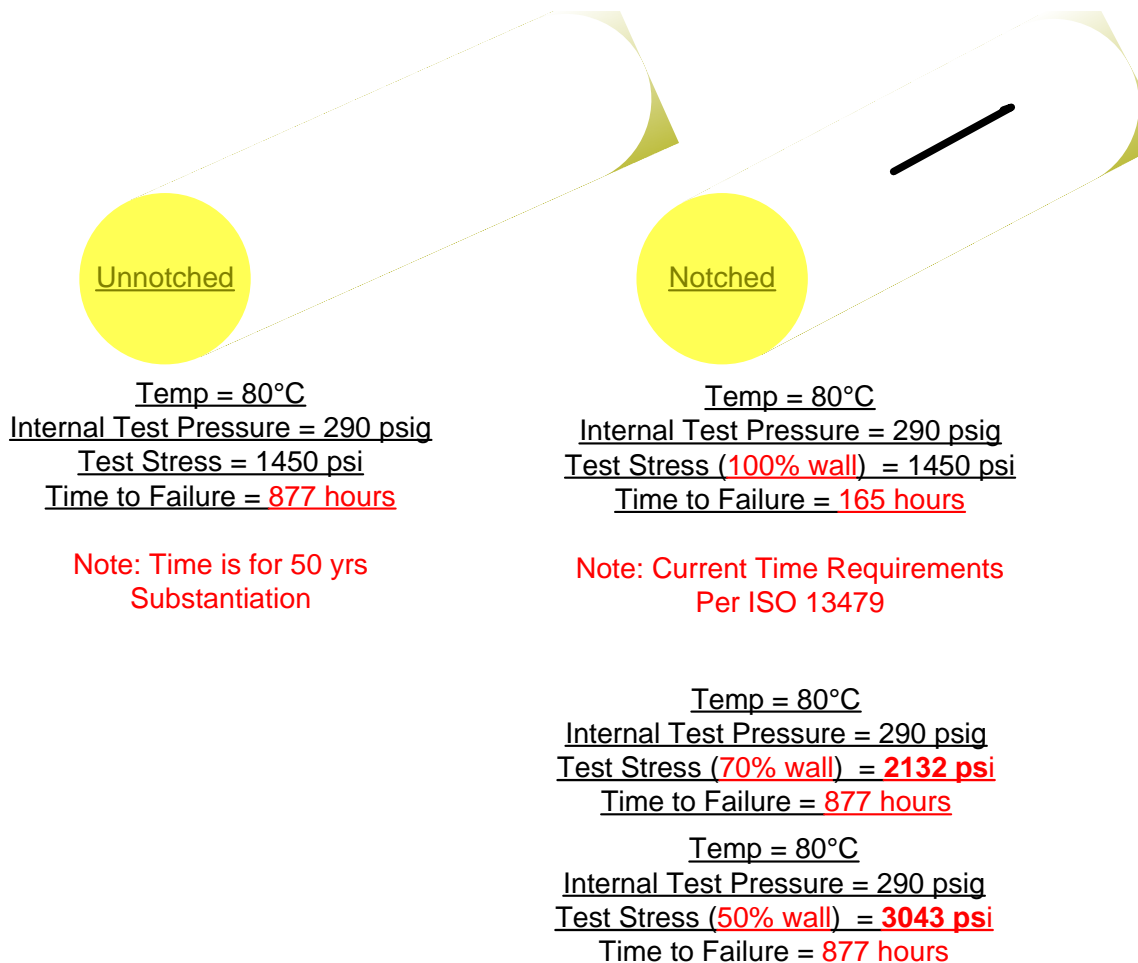
While the results of the aforementioned testing effectively addressed the SCG performance requirements contained within ASTM D2513, the ISO specification for thermoplastic piping materials contains additional test requirements to characterize the influence of surface scratches on the outside diameter of the pipe - the notched pipe test (ISO 13479).

The notched pipe test is somewhat analogous to the validation testing required under ASTM 2837 whereby actual pipe specimens are subjected to long term sustained pressure testing at elevated temperatures. However, the ISO 13479 notched pipe test provides for intentionally introducing a controlled notch (20% of the wall thickness) along the axial direction of the pipe specimens located 90° apart circumferentially. The notched pipe specimens are then subjected to constant internal pressure based on the respective material and the time to failure is recorded. It is important to note that this is a very aggressive test in that the actual stress at the remaining ligament is extremely high. Because ISO 13479 only prescribes test conditions for PE materials, a slightly modified approach was used to establish the appropriate test conditions. It was reasoned that while gas utility companies employ effective construction practices to minimize the potential for installation induced scratches, they invariably do occur. However, once installed, the pristine pipe and the pipe with the scratches both operate at the same pressures. Therefore, the project team agreed to use the same test pressure which was used to validate the long term hydrostatic strength – 290 psig. Six (6) specimens from each of the three suppliers PA12 pipe were subjected to notched pipe testing per ISO 13479. As expected, the results of the notched pipe test for each of the three suppliers' PA12 product were positive. There were no failures for any pipe specimens with a 20% notch depth at test pressures of 290 psig at 80C at test times greater than 2000 hours.

While these respective tests provided excellent insight into the SCG performance characteristics of PA12 materials, additional tests were requested by UBE to further characterize the influence of varying degree of notch depths. As a result, GTI performed comprehensive long term sustained pressure tests using notch depths of 30% and 50% of the wall thickness and a test pressure of 290 psig at 80C. In order to illustrate the extreme degree of conservatism inherent within this approach, Table 1 and Figure 2 presents the comparative illustration of the actual applied hoop stress at the location of the each respective notch depths based on the test conditions. From Figure 2, it is important to note that, at 50% notch depth, the corresponding test stress is approximately 2 times greater than the stress used to validate the HDB rating.

Conditions	Test Conditions	Time to Failure (hrs)
Condition 1 (UBE PA12)	Test Pressure: 290 psig (20 bars) <b>Notch Depth: 30%</b> <b>Stress at remaining ligament: 2132psi</b> 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) <b>Notch Depth: 50%</b> <b>Stress at remaining ligament: 3043 psi</b> Test Temperature: 80°C 50-year substantiation time: 877 hours	> 500 hours with no failures of 3/6 specimens

**Table 1: Notch pipe testing of UBE PA12 pipe specimens with at 30% and 50% notch depth**



**Figure 2: Comparison of notch pipe test criterion with 30% and 50% notch depths**

Cumulatively, the results of comprehensive tests to characterize the SCG characteristics for PA12 materials were extremely positive.

## **2.4 Effects of Secondary Stresses**

In addition to characterizing the SCG performance characteristics and influence of surface scratches, additional tests were performed to characterize the influence of secondary stresses. The motivating factors for performing these tests were two-fold. First, as previously discussed, ASTM D2513 suggests (non-mandatory requirement) that new thermoplastic materials must demonstrate at least 3-years of in-service experience through either field demonstrations and/or suitable tests which simulate the effects of in-service conditions. Second, more importantly, all of the previous tests discussed thus far only take into account the stress contribution due to internal pressure. However, under actual field conditions, the piping systems are subjected to the combined effects of both internal pressure and other secondary stresses including rock impingement, earth loading, and bending. Often, these secondary stresses, not internal pressure, are the root cause of many in-service field failures.

Therefore, comprehensive long term sustained pressure tests were performed at elevated temperatures to characterize the effects of various types of secondary stresses. It is important to note that these tests are not a part of either the ASTM or ISO standard. The test methodology is an extension of previous research performed by Dr. Charles Bargraw – DuPont and further refined by Dr. Michael Mamoun – Gas Technology Institute to study the performance characteristics of older generation PE materials. For the case of the rock impingement, the intent is to evaluate the performance of pipe materials subjected to indentations by a ½” rock. For the case of the earth loading, the typical safe deflection limit that is specified is 5%. For the case of the bending strain, the typical bend radius limits for a pipe specimen without any joints or appurtenances is 20 times the outside diameter.

Six (6) 2-inch SDR11 PA12 pipe specimens from Degussa and UBE were placed in appropriate test rigs to simulate the effects of rock impingement, earth loading, and bending strain. The entire test assembly was placed under long term sustained pressure testing at 290 psig at 80C. Again, note the significant degree of conservatism – the 290 psig test pressure is the same pressure used to validate the LTHS values. Like the case of the notched pipe test, Like the case of the notched pipe test, the applied secondary stresses in combination to the circumferential stresses resulting from the internal pressure significantly increases the overall applied stress beyond the stress value used to validate the HDB rating. The results of the testing demonstrated that there were no failures after 1000 hours of testing, as presented in Table 2 below.

Secondary Stress	Test Criterion	Results
Rock Impingement	<i>1/2" Indentation</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Earth Loading	<i>5% Deflection of Outside Diameter</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Bending Strain	<i>20 times OD</i> Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures

**Table 2: Summary of test conditions to simulate effects of secondary stresses**

## 2.5 Rapid Crack Propagation Characteristics

In 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 effective nature of the S4 test as compared to the FST, 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  $\pm$  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 constant temperature conditions (32F) 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 3 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 3: 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 formulas used to correlate the S4 test results with the full-scale critical pressure values and maximum allowable operating pressure.

Additional work has been on-going at the ISO level to perform full scale RCP testing of the PA12 materials by the various PA12 suppliers, which again is outside the scope of this project. Based on information provided by the various PA12 suppliers and as of preparing this report, there are no definitive conclusions derived from the test data. Additional meetings at the ISO have been scheduled for March 2007 to investigate the matter further.

### **Section 3**

#### **Characterization of Critical Operating Considerations**

Based on the preceding discussions, the results of comprehensive laboratory testing effectively demonstrated that the respective PA12 materials from both Degussa and UBE conforms to relevant ASTM standards and specifications with respect to performance considerations, both through short term and long term tests. However, additional tests were also performed to evaluate the impact of critical operating practices including impact of squeeze-off and joining characteristics / procedures for PA12 piping. Other than information related to the 6-inch butt heat fusion joining, the following sections contain only an abbreviated discussion with respect to each point – detailed discussions can be found in the Technical Reference Report.

##### **3.1 Effects of Squeeze-off**

A commonly used practice to safely and effectively 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 of the pipe is not compromised following the use of the squeeze-off technique, ASTM D2513 Annex A1 specifies that the pipe subjected to squeeze-off shall exhibit no leakage or visual evidence of splitting, cracking, breaking, or reduction in 1000-hour sustained pressure tests at elevated temperatures values.

In order 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 was 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 2.3 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 were consistent with expectations. There were no failures in any of the PA12 test specimens from each of the three PA12 resin suppliers pipe at times greater than 1000 hours.

##### **3.2 Polyamide 12 Joining Procedures**

A critical construction and maintenance concern involves the safety and integrity of various types of joints on plastic piping systems. 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 and Degussa PA12 material for 2-inch pipe sizes. On the basis of the test result, “qualified” PA12 joining procedures were developed. For the PA12 materials, the joining parameters were determined to be:

- Butt Fusion Interface Pressure Rang: 60 - 90 psi
- Heater Surface Temperature Range: 495 – 505°F
- Time of contact with Heater Face: 60 – 75 sec
- Melt Bead: 1/16” – 1/8”

It is important to note, like PE butt heat fusion, the PA12 butt heat fusion process is also a visual process. The specified times are an estimate and ambient temperature conditions must be taken into account.

Given that the primary intended application for PA12 piping system is for higher pressures and larger diameters, additional tests were performed to qualify these procedures for 6-inch IPS pipe specimens – the following discussion contains new information not found in the Technical Reference Report. Comprehensive tests were performed on parametrically controlled fusion joints made in accordance to the previously developed PA12 joining procedures with exception of varying the interfacial pressures and heater iron temperatures. Moreover, the compatibility of cross-fusions between each of the PA12 resin suppliers’ product was also investigated.

With the assistance of McElroy Manufacturing, several 6-inch PA12 butt heat fusion joints were prepared using the specific PA12 joining procedures. Specifically,

- 36 fusion joints were made from each resin supplier (UBE, Degussa, and EMS)
- 3 base materials – pipe only for use as control specimens
- 3 cross fusions – different materials

Four (4) coupons were machined from each fusion joint and subjected to McSnapper™ testing, as shown in Figures 3. The McSnapper™ is a high speed tensile-with-impact testing machine which combines the Tensile Impact Test ASTM D1822 and High Speed Tensile Test ASTM D2289. The McSnapper™ unit uses a hydraulic cylinder to provide the necessary force and velocity and uses a piezoelectric load cell to measure resistance forces on the samples. The unit then measures or calculates and records the force, energy, velocity, and position of the data for the respective test specimen. Figure 4 illustrates the progression of the McSnapper™ testing apparatus.



The results of the McSnapper™ testing on the 6-inch PA12 fusion joints from the various resin suppliers were consistent with expectations. There was an excellent degree of corroboration between the tensile strength values of the PA12 joints as compared to PA12 pipe specimens. Figures 6-7 presents a summary of the test data for various joints and control specimens – this is for illustrative purposes only as there were over 216 data points in total.

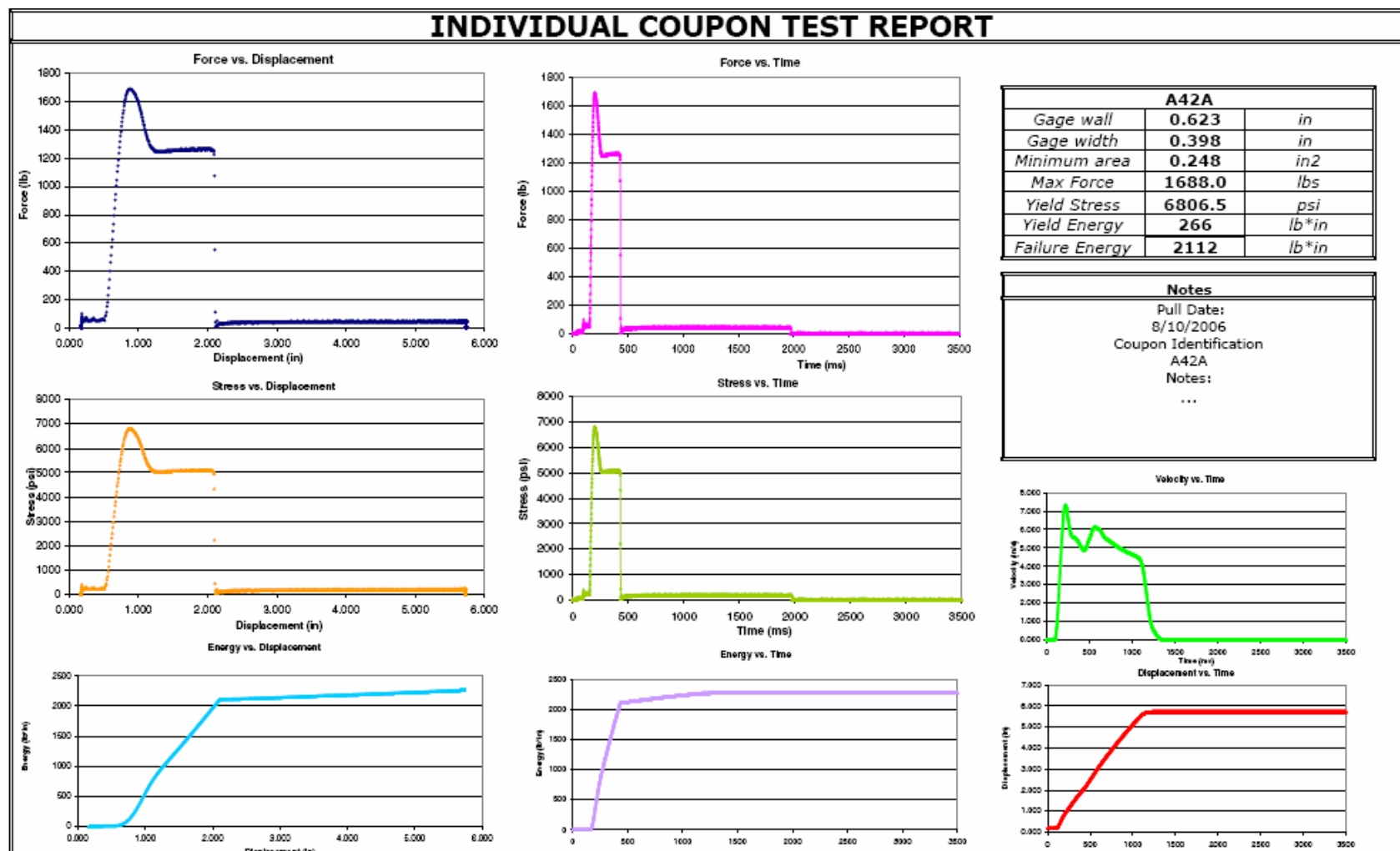
In addition to validating the performance of PA12 joints from each of the respective PA12 resin suppliers using their materials, various cross fusions were made using PA12 pipes from different suppliers – unlike pipe. The test results confirmed the ability to make strong and effective cross-fusion joints, i.e. joints made using PA12 pipes from different suppliers such as Degussa pipe to UBE pipe. It was reported that in all of the test specimens, with the exception of one coupon, all of the failures originated outside the fusion interface, as shown in Figure 5 below. The exact cause for this particular failure is unknown. Regardless, the overall results demonstrated that strong effective 6-inch PA12 joints can be made using the qualified PA12 joining procedures.



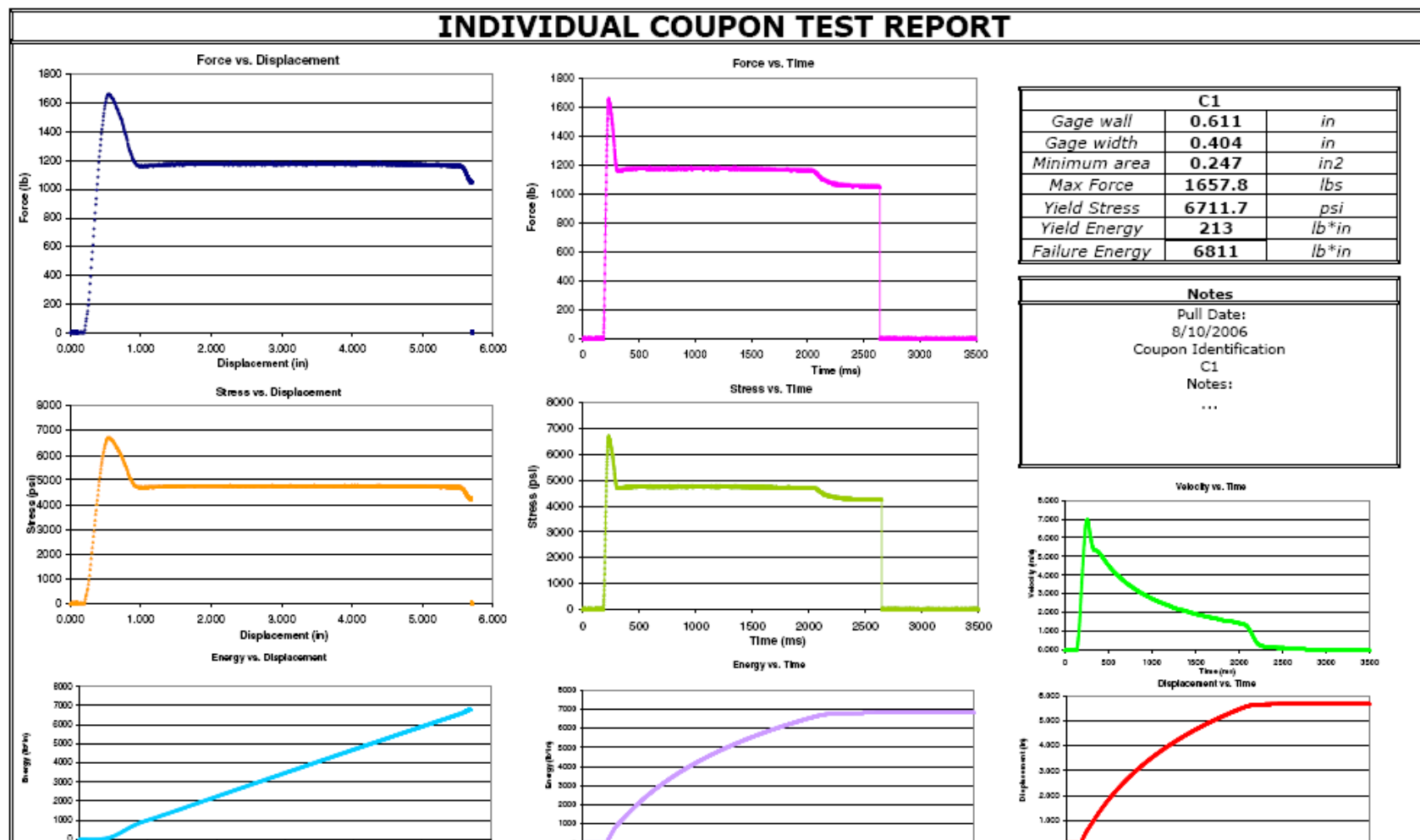
**Figure 5: Illustration of the fracture surface of a PA12 joint which did not satisfy the visual criteria for an acceptable joint**

In addition to the McSnapper™ testing, additional long term sustained pressure testing at elevated temperatures were performed on 6-inch PA12 pipe specimens using 290 psig at 80C. As expected, the results of the testing were consistent with expectations. There were no failures in any of the test specimens from each of the three PA12 resin suppliers' products at times greater than 1000 hours.

Cumulatively, the results of the testing amply demonstrated the ability to make strong joints using the qualified PA12 joining procedures consistent with CFR Part 192 requirements.



**Figure 6: Illustration of test results from McSnapper testing for a typical PA12 heat fusion joint**



**Figure 7: Illustration of test results from McSnapper testing for a typical PA12 pipe specimen**

## **Section 4**

### **Evaluation of Appurtenances**

Another critical element of this particular program was to evaluate various types of fittings and appurtenances. While there are numerous types of fittings that may be required to construct an overall PA12 piping system, the intent of this program was to test and evaluate transition fittings at a minimum. The following sections presents detailed discussions with respect to the evaluation of transition fittings, mechanical fittings, and electrofusion fittings. It is important to emphasize that the development and evaluation work specific to mechanical and electrofusion fittings was outside the OTD scope of work. Both the development efforts and the evaluations were performed independently by the PA12 resin suppliers and the information contained herein has been provided to OTD as part of the co-funding agreements.

#### **4.1 Transition Fittings**

In the context of this program, transition fittings are a critical element for the development of an overall PA12 piping system. Because the intended application for PA12 piping systems are at high pressures (150 – 250 psig), it is important to have a means to safely facilitate tie-in to steel piping systems.

The qualification and use of transition fittings for use in gas distribution applications is governed by ASTM F1973 entitled “*Standard Specification for Factory Assembled Anodeless Risers and Transition Fittings in Polyethylene (PE) Fuel Gas Distribution Systems*”. Although the requirements contained within this specification are specific to PE, this standard was used as guidance for the relevant testing of PA12 transition fittings.

Per ASTM F1973 requirements, there are several tests which are prescribed. The two most important tests include: temperature cycling and tensile testing. Given the limited quantities of transition fittings that were manufactured, the consensus decision of the project team was to evaluate the temperature cycling requirement which was believed to be the more critical between the two. The temperature cycling requirement states that the joint shall be leak-free after exposure to 10 temperature cycle tests ranging between 20F and 140F.

As a result, several 2-inch PA12 transition fittings were developed by Continental Industries using their existing PE designs and tooling. Six PA12 transition fittings from both Degussa and UBE were subjected to the range of test temperatures (20F to 140F). Three samples were then leak tested at 7 psig, and all of the specimens passed. The remaining three specimens were then tested at 1.5 times the MAOP (375 psig). For the PA12, the MAOP was chosen to equal to 250 psig assuming a HDB rating of 3150 psi and using a 0.40 design factor, see section 1.2. Under these conditions, there were no failures that were observed for the transition fittings made from the Degussa and UBE PA12 pipe.

## **4.2 Mechanical Fittings**

The qualification and use of mechanical fittings for use in gas distribution applications is governed by ASTM F1924 and ASTM F2129 depending on the operating pressure and choice of material for the plastic mains. ASTM F1924 provides the qualification requirements for mechanical fittings to be installed on PE piping system, and ASTM F2129 provide qualification requirements for mechanical fittings to be installed on Polyamide (PA) mains. It is important to emphasize that the requirements for both of these standards is the same with the exception of minor changes to certain test conditions.

Like the case with the transition fittings, targeted tests were performed on 2-inch mechanical fittings made from injection molding trials at Continental Industries using the UBE and Degussa PA12 resin. Specifically, mechanical fittings installed on PA12 piping were subjected to long term sustained pressure tests at elevated temperatures and temperature cycling tests.

Six pipe/fitting assemblies were subjected to long term sustained pressure tests at 290 psig and 80C. The mechanical fittings were tapped prior to test to ensure that entire joint is under test. The results of the testing were consistent with expectations. There were no failures that were observed in any of the test specimens at test times greater than 1000 hours.

In addition to the long term sustained pressure tests, six specimens from each supplier was subjected to temperature cycling testing. Like the previous testing on the transition fittings, the temperature cycling tests for mechanical fittings require that the mechanical joint be leak tight following exposure to 10 alternating temperature cycles ranging in temperature from 20F to 140F at pressures of 7 psig to 1.5 times MAOP. Like the case with the transition fittings, the MAOP was assumed to be 250 psig based on the use of a 3150 psi HDB rating and a 0.40 design factor. Again, as expected, there were no failures observed with any of the mechanical fittings that were tested.

## **4.3 Electrofusion Fittings**

In addition to transition fittings and mechanical fittings, another valuable component(s) that was also developed for PA12 piping systems are electrofusion fittings. It is important to emphasize that this particular activity was performed by Degussa independently and the information included herein is made in the context of the co-funding arrangement between Degussa and OTD.

The qualification for the use of electrofusion fittings is governed by ASTM F1055. Degussa, in conjunction with Friatech, developed the necessary electrofusion fittings made from PA12 resin for use on PA12 piping using the universal electrofusion box. It is important to note, the primary factor for performing this developmental activity is that given the inherent chemical make-up of PA12 materials, they will not bond with PE using heat. As a result, conventional PE electrofusion fittings will not work on PA12 piping systems. Moreover, the differences in the range of operating pressure also preclude the use of conventional PE electrofusion fittings.

Based on information provided by Degussa, the electrofusion fittings made at Friatech using the PA12 resin conforms to all relevant requirements contained within ASTM F1055 requirements.

## Section 5

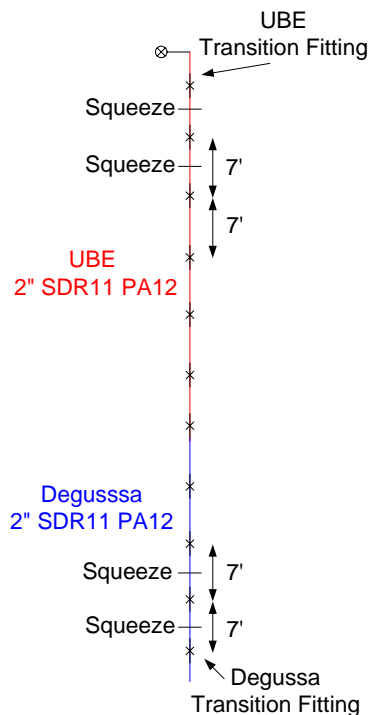
### Field Demonstration

In addition to comprehensive laboratory evaluations, another critical element of the overall program was to validate the technical feasibility for the use of PA12 piping systems and to ensure safe performance under field testing.

#### 5.1 GTI Installation – 2-inch piping with surrounding soil

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. 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.

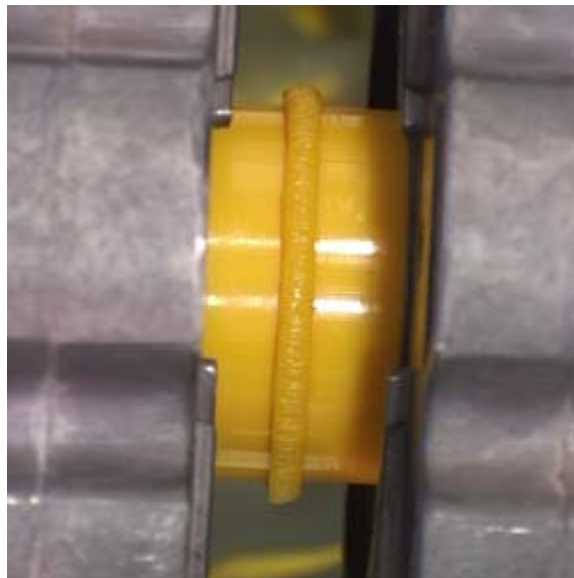
Two inch IPS SDR 11 PA12 pipes from UBE and Degussa were used for the installation. Approximately 70' of PA12 pipe was installed, of which 42' was UBE and 28' was Degussa. The schematic below is a layout of the PA12 piping material used for the field installation.



The PA12 pipes were supplied in 7 foot stick lengths, which were fused together using the qualified PA12 joining procedures as shown below.

- Butt Fusion Interface Pressure Range 60 - 90 psi
- Heater Surface Temperature Range 495 – 505°F
- Estimated Time of contact with Heater Face 60 – 75 sec
- Melt Bead 1/16" – 1/8"

All of the 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 approximately 60 seconds until the desired melt pattern was observed (1/16 – 1/8" bead). Upon removing the heat source, the pipe ends were fused together using an applied torque of 10 ft-lb. This force was applied for approximately 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 8. Two transition fittings, one of each of the respective suppliers, were also installed. The transition fittings were heat fused to the end pipe lengths from each of the respective suppliers.



**Figure 8: Typical 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 evaluated. Therefore, the pipe specimens were squeezed at various intervals over the length of pipe to be installed. 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 9.



**Figure 9: Application of squeeze-off procedure on PA12 pipe**

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 10.



**Figure 10: Completed squeeze-off on PA12 pipe**

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 entire line segment was then pressure tested at 1.5 x MAOP, or 375 psig. After the pressure testing, the pressure of the flow loop was decreased and installed operating at 250 psig, as shown in Figure 11.



**Figure 11: Installation of 2" PA12 pipe at GTI test facilities**

Following the installation, the trench was backfilled with indigenous soil. Approximately 6" of sand were placed above and below the pipe to mark the location of the flow loop for future excavations.

## **5.2 GTI Installation – 6-inch piping with different backfill materials**

Having confirmed the ability to install and operate the PA12 piping systems at higher operating pressures using 2-inch piping, additional installations were performed on GTI private property to evaluate the use of larger diameter PA12 pipe sizes – 6-inch. Specifically, two separate installations were performed to evaluate the effects of various types of backfill including rocky soil and flowable fill during October, 2006.

This particular installation(s) was extremely challenging and difficult due to the adverse weather conditions. However, it was also an excellent test installation given that these conditions represented the worst case conditions that could be possible in many portions of the United States – cold, rain/snow, and windy conditions. As a result, the entire installation took several days longer than expected to complete.

Two separate PA12 lines (one from Degussa and one from UBE) were installed in a single joint trench which was 120' long, 3' deep, and 3' wide. There were a total of two separate trenches running perpendicular to one another using two different backfill materials respectively – rocky soil and flowable fill. The 6-inch PA12 pipes were supplied in 40-foot straight lengths by both Degussa and UBE and were extruded overseas at DEKA Systems in Germany. The respective pipe segments were joined using both PA12 butt heat fusion procedures and Friatech electrofusion couplings made from the PA12 resin. Given the limited availability of additional fittings such as end caps and reducers, 6-inch PA12-steel transition fittings were installed on both ends of the line segments with the appropriate steel end connections (steel end caps) to facilitate pressurization. Figure 12 presents representative illustrations of the overall installation.



(a)



(b)



(c)



(d)

**Figure 12: Representative illustrations of the 6" PA12 installation at GTI pipe farm during October 2006. (a): Illustration of the butt heat fusion using the McElroy 28 machine. (b): Illustration of end connections used to facilitate pressurization and tie-in. The end connections consist of a 6-inch PA12 transition fitting and schedule 40 steel fittings and end caps. (c): illustration of pipe connected using both PA12 electrofusion fittings and butt heat fusion joints. (d): Illustration of pipe to be installed using flowable fill as the backfill material.**

The respective PA12 lines were installed in each of the respective trenches and subjected to a 48-hour leak test at 150 psig (Note, this extended test time was due to rain delays). Following the leak test, the lines were then pressure tested at 375 psig for a period of 24 hours, as shown in Figure 13 (again, the extended test time was due to rain). The pressure was then reduced for each line segment to 250 psig and installed at a 3' feet burial depth. Each trench was then backfilled with the respective materials, and the test installation was completed on 10/11/2006.



(a)



(b)

**Figure 13: Representative illustrations of installed PA12 pipe segments using both (a)flowable fill and (b)rock soil mixture**

While the overall installation was successful, there were some minor issues that were observed. Specifically, there were minor difficulties installing the 6-inch electrofusion couplings on the UBE pipe outdoors. Following the completion of the heating and cooling cycles, significant amount of the melt extruded outside the ends of the couplings, as shown in Figure 14. This was inconsistent with previous experience. All of the fusions which were performed indoors under controlled laboratory conditions did not exhibit this kind of behavior. It is believed that the most likely cause of these particular failures were as a result of operator error given the adverse weather conditions and lack of proper tooling.



(a)



(b)

**Figure 14: Representative illustrations of the inside and outside surfaces of the failed electrofusion couplings using on the UBE PA12 pipe**

### 5.3 National Fuel Installation – 6” piping with surrounding soil

Besides the installations at GTI, an additional test installation was performed by National Fuel on its private property located at their Mineral Springs facility. Like the GTI installation, this particular installation was also impacted due to adverse weather conditions. During the week prior to the installation, record snowfalls (20 inches) fell on the Buffalo, NY area. Regardless, the installation took place as planned.

The primary objective(s) of this particular installation was to evaluate the impact of bending strain on the in-service performance characteristics of the PA12 piping system and gain critical feedback from an operator’s perspective. Approximately 500 feet of 6-inch straight length PA12 pipes supplied in 40-foot sections from Degussa and UBE were installed under the influence of two different bend radius. For the UBE pipe with no joints or appurtenances, the bend radius for the test installation was 20 times the OD. For the Degussa pipe, the test bend radius was 90 times the OD at the location of the heat fusion joint. This is significantly more conservative than the specifications prescribed for PE piping. Figure 15 presents an illustration of the overall test installation site.



**Figure 15: illustration of National Fuel installation with varying bend radii**

Like the installations at GTI, the PA12 pipe segments were joined using both heat fusion and electrofusion couplings, as shown in Figure 16. Given the previous experience with the electrofusion coupling and the UBE pipe, there was additional scrutiny during the installation of these respective pipe/coupling joints. There were no failures that were observed. This underscores the possibility that the electrofusion fitting failures that were observed during the GTI installations were a result of operator error and/or improper tooling.



**Figure 16: Representative illustrations of the National Fuel installation at its Mineral Spring facilities.**

After overcoming some minor installation issues not related to the PA12 piping systems (poor welds on the steel end connections), the lines were installed and were pressure tested using compressed natural gas at a test pressure of 375 psig for approximately 4 hours. Following its completion, the line pressure was reduced to 250 psig. Additional 6x2" mechanical fittings from Continental Industries were installed using their recommended installation procedures. The fittings were tapped against line pressure and leak tested, as shown in Figures 17. The installation was completed on 10/25/06.



**Figure 17: Illustration of the installation and leak testing of a 6x2” Continental Industries mechanical fitting. The mechanical fittings were installed at 250 psig and tapped with no observed issues.**

#### **5.4 Additional High Pressure Installations – Europe**

In addition to this particular program, additional installations have been performed in a parallel effort that this on-going in Europe with similar objectives and technical approach as the OTD program.

In order to demonstrate safe operations at high pressures, Degussa has worked with Germany’s largest utility company, E. ON Ruhrgas to install a 60m line of 4-inch SDR11 pipe from coiled pipe using both the electrofusion and butt heat fusion process. The line was installed by E. ON Rhurgas operators using their company’s approved installation and operating practices. Since November 2005, the line has been pressurized to 345 psig (24 bar) and has not experienced any failures. In parallel, a 3m test line has also been pressurized at 515 psig (36 bar). Both installations have performed well without any reported failures to date. Figure 18 are some illustrations of the 345 psig installation. Figure 18 presents a few illustrations from the E.ON Rhurgas installations, as shown below.



**Figure 18: illustrations of Ruhrgas installation operating at 345 psig**

The cumulative results of these respective installation alongside the installations as part of the OTD program amply demonstrate the effectiveness of using PA12 piping systems over a range of sizes (2-inch through 6-inch) and increased operating pressures. Moreover, the installation in Europe also demonstrated the applicability of using coiled pipe which provides additional installation cost savings due to the reduction in the number of joints that are required over the length of the installation.

## **Section 6**

### **Summary and Conclusions**

Since 2004, through the support of Operations Technology Development (OTD) group and PA12 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 material conforms to all of the requirements contained with ASTM D2513 and its respective Annexes.
- 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. Over 40 years of field experience has demonstrated that the primary mode of in-service field failures is as a result of the SCG mechanism. Given the positive results of testing, it is reasonable to infer that the PA12 piping systems will be able to sufficiently withstand the combined effects of both internal pressure and the influences of secondary stresses resulting from outside surface scratches, rock impingement, earth loadings, and bending strain.
- 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. Specifically, the test data demonstrates that strong and effective joints can be made using PA12 joining procedures. Moreover, electrofusion joints also can produce strong and effective joints.
- The results of the RCP testing were slightly inconsistent with expectations. The calculated maximum operating pressure based on the S4 test data is lower than the target range of 200 psi; however, the meaningfulness of the test procedure, the efficacy of the correlation functions, and the implicit safety factor are at best questionable at this time. 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.
- The results of various field installations and feedback from operators has been positive. The feedback indicates that the operators did not realize any difficulties with the use of the PA12 materials as compared to PE piping systems.

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



## Evaluation of Polyamide 12 (PA12) for High Pressure Gas Distribution Applications

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Washington, D.C.  
August 26, 2008

## Polyamide 12 (PA12)



- First commercial production of PA12 began in the 1970s in Marl, Germany – Degussa AG
- At present, four commercial suppliers
  - Evonik-Degussa AG (Germany)
  - UBE Industries (Japan)
  - EMS Grivory (Switzerland)
  - Arkema (France)
- The frequency of amide groups (amide density) differentiate various polyamide materials from each other
  - Influences strength and physical characteristics
  - Influences moisture absorption characteristics

## Comparison to PA11



- In general, similar but enhanced material performance characteristics

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	350
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

Source: OTD Technical Reference Summary Report issued December 2005

## History of Use



- Australia Gas Light Experience
  - Conversion to PA12 during 1990s from PA11 for low pressure gas distribution applications
    - Thinner wall – mostly insertion applications for rehabilitation
    - Nearly 80% of AGL systems (mains) use Polyamide materials
    - Mostly operate at 58 psig
    - 32 mm SDR25 sizes
- Additional positive installation experiences with PA12 piping systems - Poland and Chile
- Under evaluations in North America and Europe for higher pressures (>150 – 250 psig systems)

## Program Objective (OTD/PA12 Suppliers)



- Since 2004, comprehensive program to validate the technical feasibility for use of Polyamide 12 (PA12) at higher operating pressures and larger diameters without sacrificing flow capacity in a cost effective manner through both laboratory testing and field demonstrations
  - Specifically,
    - Ensure conformity to existing code requirements, industry guidelines and practices, and ASTM standards and specifications
- Note: Parallel efforts being performed in Europe under the GERG Consortium

## Key Accomplishment to Date



- PA12 has...
  - An established ASTM production designation per ASTM D4066
  - Comprehensive test data demonstrates conformity to ASTM D2513 requirements (Main body and Annex A5 for PA materials)
    - At present, ASTM D2513-06 contains annex specific for PA12
  - Has an established Hydrostatic Design Basis Rating within PPI TR-4 listings
    - **3150 psi at 73F**
    - **2000 psi at 140F**
  - Over 3 years of actual in-service performance at operating pressures up to 250 psig
    - Results of in-service ageing demonstrates excellent retention of mechanical and physical properties

## Performance Characteristics



- Evaluation of Performance Characteristics
  - Develop comprehensive data to characterize pertinent properties of PA12 to ensure structural integrity and safe long term performance for the intended application
    - Short term testing
    - HDB Rating
    - Resistance to Slow Crack Growth (SCG)
    - Characterization of RCP Characteristics
    - Weathering

## SCG Characteristics (1/5)



- Typical short term tests help to preclude propensity for failures in the “ductile” manner
- Field experience demonstrates that majority of in-service related field failure occur as a result of SCG...
  - Various tests in place to validate excellent SCG characteristics of PA12 resins
    - HDB validation per ASTM D2837
    - PENT Testing per ASTM F1474
    - Notched Pipe Testing per ISO 13479 (ASTM F1474 withdrawn)
    - Influence of secondary stresses including rock impingement, earthloading, and excessive bending strain

## SCG Characteristics (2/5)

### HDB Validation



- Validation Req. Per TR-3

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

HDB to be Validated (psi)	193°F (80°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

- Use of bi-directional shift functions

> 50-year substantiation per ASTM D2513

$$a_r = \exp[0.109(t_r - T_s)] \quad b_r = \exp[-0.0116(t_r - T_s)]$$

$$t_s = t_r a_r$$

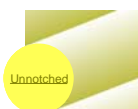
$$a_r = 499.1966$$

$$p_s = \frac{p_r}{b_r}$$

$$\frac{1}{b_r} = 1.937115$$

For Test Temp = 80°C  
Validation of 50 yrs = 877 hours  
For Same Stress Under Test

- Validation Test Conditions for PA12



Temp = 80°C  
Internal Test Pressure = 290 psig  
Test Stress = 1450 psi  
Time to Failure = 200 hours

**Results: NO Failures at test times greater than 2000 hours!!!**

## SCG Characteristics (3/5)

### Notched Pipe Testing



- Notched Pipe Testing per ISO13479 – No PA Requirements

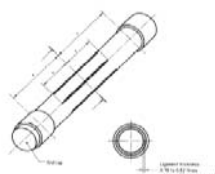


Table A.1 — Test-pressure levels

SDR	S	Test pressure, p	
		PE 80	PE 100
41	20	2	2.3
33	16	2.5	2.86
26	12.5	3.2	3.68
21	10	4	4.6
17.8	8.3	4.82	5.54
17	8	5	5.75
13.8	6.3	6.35	7.3
11	5	8	9.3
9	4	10	11.5
7.4	3.2	12.5	14.38
6	2.5	16	18.4

- Test conditions: test at same internal pressure as the HDB validation testing noting increased stress at notch location (290 psig at 80C)
- **Results: NO Failures at test times greater than 1000 hours**

## SCG Characteristics (4/5)

### PENT Testing



- PENT Testing per ASTM F1473

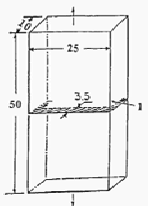


TABLE 1 Notch Depth as a Function of Specimen Thickness\*

NOTE 1—This table is based on the stress intensity being the same for all thicknesses.

Thickness, mm	Notch Depth, mm
4.00	1.90
5.00	2.28
6.00	2.60
7.00	2.85
8.00	3.05
9.00	3.20
10.00	3.30
11.00	3.40
12.00	3.50
13.00	3.60
14.00	3.70
15.00	3.80
16.00	3.90
17.00	4.00
18.00	4.10
19.00	4.20
20.00	4.30

\*For an intermediate thickness, linearly interpolate to obtain the notch depth. The notch depth in the specimen shall be within 0.02 mm of the interpolated value.

- Condition A: Test Stress = 2.4 MPa, Test Temp = 80C
- Condition B: Test Stress = 4.8 MPa, Test Temp = 80C
- Results: NO Failures after 2000 hours of testing at both respective test conditions (Conditions A and B)**

## SCG Characteristics (5/5)

### Effects of Secondary Stresses



- Evaluate the long term performance of PA12 piping systems under the combined influence of both internal pressure and secondary stresses (rock impingement, earth loading, and bending strain)

Secondary Stress	Test Criterion	Results
Rock Impingement	$\frac{1}{2}$ " Indentation Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Earth Loading	5% Deflection of Outside Diameter Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures
Bending Strain	20 times OD Test Pressure = 290 psig Test Temperature = 80C	Test Time > 1000 hour with No failures

- Results confirm excellent long term performance characteristics of PA12 materials and provide laboratory corroboration of field demonstrations**

## RCP Characteristics <sup>(1/2)</sup>



- Determine susceptibility to Rapid Crack Propagation
  - Two prevalent test methodologies
    - Small-scale steady state per ISO 13477
    - Full-scale RCP test per ISO 13478
  - In the event of dispute, Full-scale RCP is the referee test method
- Comprehensive testing through both S4 and full-scale testing demonstrate that conventional correlations are specific to PE and do not apply to PA12 piping systems
- Based on comprehensive data, new correlations have been developed for PA12

$$p_{c,PA12} = ((7.8 \times p_{c,S4}) + 6.8) \times 14.5 = \text{psig}$$

## RCP Characteristics <sup>(2/2)</sup>



- Full-Scale Results developed at Advantica

Supplier	Pipe Size	Full-Scale Critical Pressure
1	110 mm SDR-11 (4-inch)	≥ 30 bar (≥ 435 psig)
2		
1	170 mm SDR-11 (6-inch)	≥ 25 bar (≥ 363 psig)
2		

- Supports use of PA12 piping systems at operating pressures up to 250 psig

## Operating Considerations



- Characterization of Relevant Construction, Maintenance, and Operating Considerations....
  - Evaluate (develop, as necessary) applicability of various operating practices
    - Effects of squeeze-off (flow control)
    - Joining
    - Evaluation of appurtenances
  - Critical in terms of ensuring streamlined acceptance within gas distribution companies

## Flow Control



- Critical need to be able to “shut-off” flow of gas in a safe manner
  - Most prevalent technique: Squeeze-off
  - Per ASTM D2513 Requirements,
    - Squeeze pipe specimens, maintain for 4 hours, re-round
    - Subject squeezed specimens to sustained pressure testing at elevated temperatures for 1000 hours
  - Six (6) specimens subjected to 290 psig at 80C
  - Results: NO Failures of squeezed pipe specimens after 1000 hours of testing

## Joining (1/5)



- Butt Heat Fusion (2-inch and 6-inch)
  - Developed qualified joining procedures consistent with the requirements contained within Part 192 requirements
    - Per 192.283, must perform: (Quick burst testing or sustained pressure testing) and tensile testing
    - Several joints using 2-inch pipes were prepared using parametrically controlled joining parameters
    - Joints then subjected to prescribed tests to yield qualified procedures
    - Additional evaluations to verify the ability to cross fuse PA12 pipe from various suppliers

## Joining (2/5)



- PA12 Joining Parameters
  - Heater Iron Temperature: 490 – 510F
  - Interfacial Pressure: 60 – 90 psi
  - Approx. Contact Time: 60 – 75 sec
  - Melt Bead: 1/16" – 1/8"
  - Visual process and must take into account ambient conditions
- Results of quick burst and tensile strength of joints compare well with control pipe specimens
- Results of long term sustained pressure testing at 290 psig and 80C – NO Failures after 1000 hours of testing
- Procedures also qualified for 6-inch SDR11 pipe sizes
- Procedures validated for cross-fusion among different PA12 suppliers

## Joining (3/5)



- 6-inch SDR11 VESTAMID PA12 McSnapper Testing



All failures terminated outside fusion interface

## Joining (4/5)



- Electrofusion Joining
  - Friatech has developed EF fittings (couplings, saddles, reducers, end caps, etc) for use on PA12 piping systems
  - EF Fittings have been evaluated by Friatech and are consistent with the requirements of ASTM F1055 specifications



## Joining (5/5)



- Mechanical Joining
  - Several 2-inch Mechanical Fittings have been molded at Continental Industries using the PA12 Resin with existing tooling
  - Mechanical Fittings subjected to sustained pressure testing and thermal cycling testing
  - Results:
    - No Failures under sustained pressure testing at 290 psig and 80C after 1000 hours of testing
    - Ability to maintain leak tight seal after 10 alternating temperature cycling (-20F to 140F) at pressures between 7 psig and 375 psig.

## Appurtenances



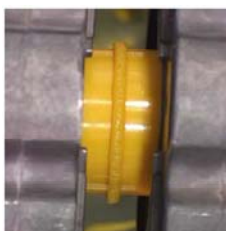
- Must ensure complete ability to safe construct an entire PA12 piping system
  - Transition fittings
    - Evaluated 2-inch and 6-inch transition fittings made by Continental Industries and R.W. Lyall using PA12 pipe
    - Performed thermal cycling testing and pull out resistance testing
    - Results: NO Failures under both testing requirements
  - Valves (In-line and EFV for service lines)
    - Under development

## Field Demonstrations



- Several field demonstrations were performed to evaluate the storage and handling techniques, effects of various geographic and climatic conditions, in-service stress conditions, etc.
  - 2-inch PA12 (GTI Pipe Farm) at 250 psig
  - 6-inch PA12 (GTI Pipe Farm – Rocky Soils) at 250 psig
  - 6-inch PA12 (GTI Pipe Farm – Flowable Fill) at 250 psig
  - 6-inch PA12 (National Fuel – Wet/Cold Conditions) at 250 psig
  - 4-inch PA12 (City of Mesa – Hot Climates) at 160 psig
  - 4-inch PA12 (WE Energies – Cold Climates) at 250 psig
  - 4-inch PA12 (DTE – Cold Climates) at 330 psig

## 2" GTI Installation 250 psig Installation



### Notes:

- Approx: 100 ft long
- 2-inch SDR11 pipe
- Operating Pressure = 250 psig
- Press. Test = 1.5 MAOP = 375 psig
- Installation: 01/05
- Backfill: Indigenous soil
- Note Cross-fusion of Degussa/UBE

## 6" GTI Installation

Rocky Soil and Flowable Fill Backfill Materials – 250 psig

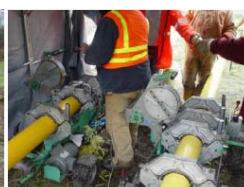


### Notes:

- Approx: 200 ft long
- 6" SDR11 pipe
- Pressure = 250 psig
- Press. Test = 1.5 MAOP = 375 psig
- Installation: 10/06
- Backfill: Rocky Soil (80/20) mix and Flowable fill
- Use of butt fusion joining and EF couplings
- 6" Transition fittings with steel end connections

## 6" National Fuel Installation

Wet and Cold Environments plus tight bend radius – 250 psig

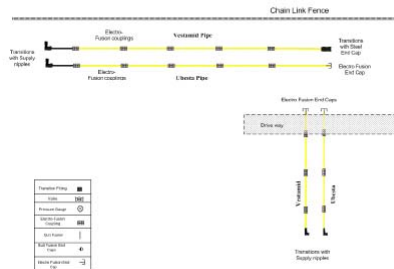


### Notes:

- 6" SDR 11 pipe approximately 800 in length operating at 250 psig using compressed natural gas
- Connected using qualified PA12 joining procedures and 6" PA12 EF Couplings
- Pressure test to 375 psig
- 6x2" Continental mechanical fittings installed, leak tested, and tapped at 250 psig
- Indigenous soil: wet and cold conditions
- Installation completed on 10/06

## 4" City of Mesa

Hot Climates and Use of a elevated temperature HDB Rating



City of Mesa – Vestamid & Ubesta PA12 Pipe



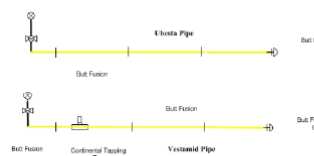
City of Mesa PA12 Material List	
Description	Manufacturer
4" SDR 11 PA12 pipe	George Fischer Piping Systems
4" PA12 to steel transition fittings	R.W. Lyall
4" PA12 electrofusion couplings	Friatec
4" PA12 electrofusion end caps	Friatec
Mechanical Eliminator purge fitting	Continental Ind.
Steel end cap	misc.
Steel nipple	misc.

### Notes:

- 4" SDR 11 pipe approximately 1000 in length operating at 160 psig using air
- Connected using qualified 4" PA12 EF Couplings
- Pressure test to 320 psig
- 4x2" Continental mechanical fittings installed, leak tested, and tapped at 160 psig
- Indigenous soil: Hot and Dry conditions
- Installation completed on 03/08

## 4" WE Energies

Cold Environments – 250 psig



WE Energies - Vestamid & Ubesta 4" PA12 Installation

WE Energies PA12 Material List	
Description	Manufacturer
4" SDR 11 PA12 pipe	George Fischer Piping Systems
4" PA12 to steel transition fittings	R.W. Lyall
4" PA12 end caps	George Fischer Piping Systems
Mechanical Eliminator purge fitting	Continental Ind.
Steel end cap	misc.
Steel nipple	misc.

Transition Fitting	■
Steel	□
Pressure Change	○
But Fusion	■
But Fusion	■
But Fusion End Cap	■
Steel Tapping Tee	■
Tapping Tee	■

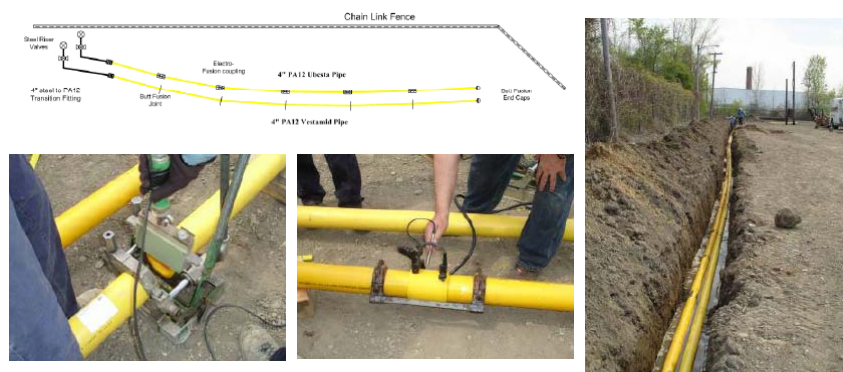


### Notes:

- 4" SDR 11 pipe approximately 250 in length operating at 250 psig using air
- Connected using qualified PA12 joining procedures and 6" PA12 EF Couplings
- Pressure test to 375 psig
- 4x2" Continental mechanical fittings installed, leak tested, and tapped at 250 psig
- Indigenous soil: wet and cold conditions
- Installation completed on 04/08

## 4" DTE Installations

Cold Environments – 330 psig



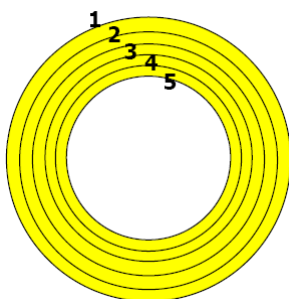
### Notes:

- 4" SDR 11 pipe approximately 250 in length operating at 250 psig using air
- Connected using qualified PA12 joining procedures and 4" PA12 EF Couplings
- Pressure test to 450 psig
- 4x2" Continental mechanical fittings installed, leak tested, and tapped at 330 psig
- Indigenous soil: wet and cold conditions
- Installation completed on 04/08

## Effects of In-service Ageing



- Important to validate impact of in-service ageing
  - Samples were removed from GTI installation (30 months) and National Fuel (15 months)
  - Layers of PA12 pipe material were removed to ensure the retention of key properties throughout the wall thickness



Results demonstrate that both the VESTAMID and UBESTA PA12 materials retain their physical and mechanical properties

-- Little to no change in the measured Properties throughout wall thickness

## Weathering



- Subject PA12, MDPE, and HDPE tensile specimens to accelerated Xenon Arc UV degradation
- Remove specimens after 3 days, 1 month, 2 month, 3 month, and 6 month exposure times
- Subject specimens to ASTM D638 tensile testing
- Measure change in tensile properties
- Data indicates no appreciable change in the tensile properties after exposure to accelerated weathering up to 6 months in Xenon Arc testing apparatus
- Additional **real-time weathering** studies are in-progress in various climates (Phoenix and Florida) at ATLAS Weathering

## Key Take Away



- Cumulative results validate the potential use of PA12 piping system for high pressure gas distribution applications (>150 psig)
  - Excellent short term and long term performance characteristics consistent with ASTM D2513 requirements
  - PA12 has an HDB Listing in PPI TR-4
    - 3150 psi at 73F
    - 2000 psi at 140F
  - Existing construction, maintenance, and operating practices specific to PE are readily transferable to PA12 piping systems
  - Field demonstrations validate the ability to operate PA12 piping systems up to 250 psig under various geographic and climatic conditions and backfill types
  - Excellent retention of mechanical and physical properties following exposure to in-service conditions

## Proposed Next Steps



- Initiate waiver (special permit) process to help support revised NPRM filing based on the following:
  - Seek to operate PA12 piping systems at maximum design pressures between 150 - 250 psig as limited its HDB rating, 0.40 design factor, and respective pipe sizes shown below using the design formula contained with CFR Part 192.121:

<u>Nominal Pipe Size in inches</u>	<u>Minimum Wall Thickness in inches</u>	<u>Corresponding SDR Values</u>
½" through 1-1/2"	0.090 in.	Varying
2-inch	0.216 in.	11
3-inch	0.259 in.	13.5
4-inch	0.264 in.	17
6-inch	0.390 in.	17



## **POLYAMIDE 12 (PA12) FIELD EVALUATIONS**

### **UTILITY FIELD DEMONSTRATION PROGRESS REPORT**

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August 2008

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The author appreciates the contributions of the following entities:

City of Mesa Gas, Gas Division

DTE Energy Company (MichCon Gas, business unit)

WE Energies

## Executive Summary

The natural gas industry has long understood the advantages associated with plastic piping materials. In addition to being easier to handle and join, plastic piping materials eliminate the need for long-term corrosion control and the associated costs. This is evident by the exponential increase in the amount of plastic pipe that has been installed in the past few decades. At present, the majority of the new installations utilize polyethylene piping materials at pressures up to 125 psig.

Since the mid-1990's, there has been an on-going effort to utilize the plastic piping system at higher operating pressures. The Department of Transportation Office of Pipeline Safety (DOT OPS) has amended the Federal Code to allow the use of the polyethylene (PE) materials at increased pressures of up to 125 psig. Concurrently, the industry has also sponsored research into new materials that can operate at pressures of up to 250 psig while maintaining the overall benefits of plastic piping materials. One of the most promising candidates is polyamide 12 (PA12).

In order to be proactive, Gas Technology Institute (GTI) with support from Operations Technology Development, NFP (OTD), Evonik (Degussa), and UBE have initiated a research program to validate the feasibility for the use of Polyamide 12 (PA12) in high pressure gas distribution applications through comprehensive laboratory testing and field experiments. Specifically, the program was aimed at:

1. Developing the required short-term mechanical property data per applicable ASTM and industry standards and specifications
2. Characterizing the long term performance considerations
3. Evaluating various construction, maintenance, and operating practices
4. Obtaining valuable in-service performance related experience.

The results of this recently concluded research program demonstrated the PA12 materials, as compared to other resin suppliers, appear to be a promising candidate material for high pressure gas distribution applications.

In order to further test and evaluate the PA12 piping system, installations were planned at additional utilities including, City of Mesa of Arizona, WE Energies in Wisconsin, and MichCon Gas in Michigan. The objective of the installations was to further evaluate the performance of PA12 in various environments across different practices and procedures found in the utilities around the country. This will provide utility personnel and product manufacturers with a better understanding of the design, construction, and maintenance necessary for future PA12 installations and to support the on-going regulatory initiatives, e.g. Notice of Proposed Rulemaking.

GTI has solicited the above mentioned utility candidates for installation/field demonstration evaluations with the support of the PA12 suppliers. GTI has assisted the utility companies in identifying, planning, coordinating, and overseeing the execution of installations.

## Introduction

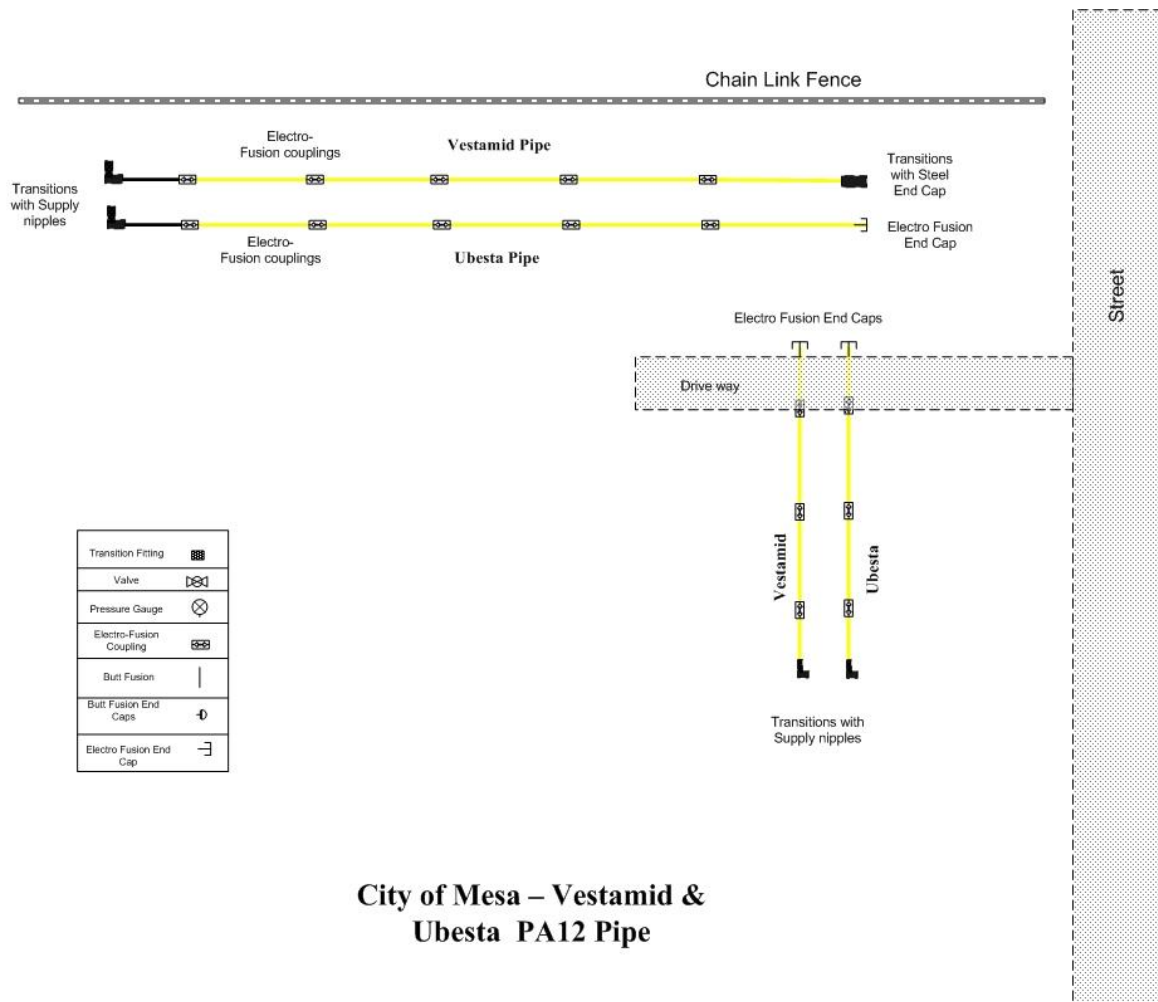
In order to validate the overall performance and to validate the technical feasibility for the use of PA12 piping system, GTI has solicited several utilities to participate in field trials. The objective of these installations was to further evaluate the performance of PA12 in various environments across different practices and procedures found in the utilities around the country. It also provides a basis for revising 49 CFR Part 192.123 to permit the use of PA12 at higher operating pressures. Several small-scale installations were carried out to evaluate the PA12 system in various types of operating environments.

GTI, along with three utilities – City of Mesa Utilities in Arizona, MichCon Gas in Michigan, and WE Energies in Wisconsin, worked together to plan small scale backyard evaluations of PA12 systems produced from resins from both Evonik and UBE. The overall installations were planned and executed considering safe, reliable, and the varied construction practices of the three natural gas distribution utilities.

## Installation Details

### ***City of Mesa Utilities – Arizona***

The first installation took place on City of Mesa Utility's property in Arizona. The installation schematic is shown below in Figure 1. The installation was initiated and completed during March 2008.



### City of Mesa – Vestamid & Ubesta PA12 Pipe

**Figure 1: Installation Schematic – City of Mesa Utility – Mesa, Arizona - March 2008**

This particular installation provides an excellent opportunity to evaluate the PA12 piping systems in a hot weather climate. Arizona is often considered to be one of the worst climate conditions for plastic piping systems the United States. The hot air temperatures lead to elevated ground temperatures.

Two separate 4-inch IPS SDR 11 PA12 systems (one from Evonik and one from UBE) were installed in two perpendicular joint trenches with a sloping depth. The depth was approximately one foot deep at one end of the trench to five foot deep at the other end. The 4-inch PA12 pipes were supplied in 40-foot straight lengths by both Evonik and UBE. The respective pipe segments were joined using Friatech electrofusion couplings and end caps made from the PA12 resin (Figure 5). PA12-steel transition fittings were produced by R.W. Lyall and installed on at least one end of each line segment with the appropriate steel-end connections (steel end caps and nipples) to facilitate pressurization (Figure 4). In addition, a Continental Industries Eliminator fitting (mechanical purge fitting) was installed for evaluation purposes (Figure 3).

City of Mesa PA12 Material List	
Description	Manufacturer
4" SDR 11 PA12 pipe	George Fischer Piping Systems
4" PA12 to steel transition fittings	R.W. Lyall
4" PA12 electrofusion couplings	Friatec
4" PA12 electrofusion end caps	Friatec
Mechanical Eliminator purge fitting	Continental Ind.
Steel end cap	misc.
Steel nipple	misc.

**Table 1: Materials used on the City of Mesa PA12 small scale installation**

Representatives from Evonik, UBE, and Friatec were onsite to observe and assist with the proper installation of their various components. Approximately 260 feet of each pipe (Evonik and UBE) was assembled using the electrofusion end caps and couplings adjacent to the open trench as illustrated in Figure 7 and then lowered in the trench fully assembled. The entire system was then leak tested at 50 psig and pressure tested with nitrogen to 320 psig (see Figure 2). After a successful pressure test, the pressure was reduced and secured at the current operating pressure of 160 psig. The 160 psig level is the maximum allowable operation pressure at 140°F (80°C) calculated using a 0.4 design factor.

Thermocouples were attached to the PA12 piping system in order to record pipe temperature data at various locations along the pipe (see Figure 6). Ground temperatures in Arizona are considered to be some of the highest in the country. The pipe was then backfilled with native soil. Periodic inspections will be conducted to record pressure and temperature of the PA12 system.



**Figure 2: Leak and pressure test of PA12 piping system**



**Figure 3: Installation of Continental Eliminator fitting**



Figure 4: PA12-steel transition fittings w/ supply nipples



Figure 5: Installation of Friatec electrofusion coupling



Figure 6: Thermocouples attached to pipe surface



Figure 7: PA12 Pipe coupled together

## ***WE Energies - Wisconsin***

In order to further test and evaluate the PA12 piping system, a small scale installation was planned at WE Energies' Racine, Wisconsin operating facility. The following summarizes the overall installation at WE Energies.

The installation of the PA12 IPS SDR 11 piping system took place on a cold day (approx. 32 °F) in April 2008. Two separate 4-inch diameter PA12 mains (one from Evonik and one from UBE) were installed in the same joint trench. The 4-inch PA12 pipes were supplied in 40-foot straight lengths by both Evonik and UBE. Three sticks of each (approximately 140 foot) were joined together using butt heat fusion techniques. A McElroy 14 machine was used to fuse the ends of the pipe together based on existing PA12 butt fusion procedures. The qualified PA12 joining procedures included the following parameters:

- Butt Fusion Interface Pressure Range 60 – 90 psi
- Heater Surface Temperature Range 495 – 505°F

- Estimated Time of contact with Heater Face 60 – 75 sec
- Melt Bead 1/16 inch – 1/8 inch

Each line contained a butt fused PA12 end cap on one end and a PA12-steel transition fitting butt fused to the other end. The PA12 transition fitting was produced by R.W. Lyall. A steel end cap and nipple were welded to the transition fitting. A Continental Eliminator mechanical fitting was also installed for evaluation purposes.

<b>WE Energies PA12 Material List</b>	
<b>Description</b>	<b>Manufacturer</b>
4" SDR 11 PA12 pipe	George Fischer Piping Systems
4" PA12 to steel transition fittings	R.W. Lyall
4" PA12 end caps	George Fischer Piping Systems
Mechanical Eliminator purge fitting	Continental Ind.
Steel end cap	misc.
Steel nipple	misc.

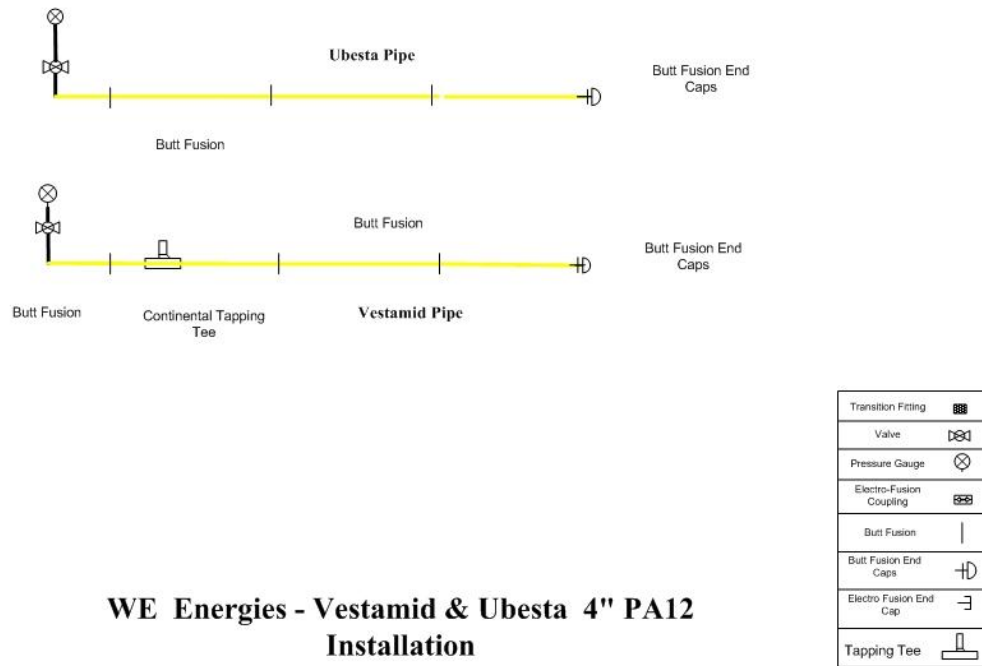
**Table 2: Materials used on the WE Energies PA12 small scale installation**

The installation plan called for both butt fusion joints and electrofusion couplings to be used in joining the sections of PA12 pipe together. However, WE Energies' universal electrofusion processor would not fit the Friatec couplings. The electrical connection nodes on the couplings were of a different size than the processor and the processor's various adapters. This may need to be further investigated with Friatec so the nodes on the fittings can fit all commonly used processors and not just Friatec's.

Once the pipes were assembled, they were both lowered into the open trench. The trench was approximately 3 foot deep. Both systems (Evonik and Ube) were then leak and pressure tested with nitrogen to 375 psig. After a successful pressure test, the pressure was reduced and secured at the current operating pressure of 250 psig. This is the maximum allowable operation pressure for the PA12 SDR 11 pipe calculated using a 0.4 design factor.

The pipe was then backfilled with native soil. Periodic pressure inspections are taking place during the evaluation period of the PA12 piping system.

A schematic of the installation is shown below:



**Figure 8: WE Energies Small Scale PA12 Installation Schematic**



**Figure 9: Open trench and PA12 pipe sections**



**Figure 10: Butt fusion of PA12 pipe sections**



**Figure 11: Fusion bead on PA12 pipe**



**Figure 12: Butt fused end cap installation**



**Figure 13: Butt fusion to install transitions**



**Figure 14: Continental purge tee installed**



**Figure 15: Initiating pressure test on system**



**Figure 16: Soap testing fusion joints for leaks**

## MichCon Gas – Michigan

The final small scale installation of PA12 pipe took place at MichCon Gas (a DTE Energy Company) in early May 2008. The objective of this installation was to evaluate the performance of PA12 and its components at an increased operating pressure of 330 psig.

Two separate 4-inch IPS SDR 11 PA12 mains (one from Evonik and one from UBE) were installed (approximately 240 foot of each) in a joint open trench with a depth of four to five feet deep. The 4-inch PA12 pipes were supplied in 40-foot straight lengths by both Evonik and UBE. Six of each supplier's pipe segments were joined using both butt fusion techniques and Friatech electrofusion couplings. The butt fusions were made using McElroy fusion equipment per the existing PA12 fusion procedures. The qualified PA12 joining procedures included the following parameters:

- Butt Fusion Interface Pressure Range 60 – 90 psi
- Heater Surface Temperature Range 495 – 505°F
- Estimated Time of contact with Heater Face 60 – 75 sec
- Melt Bead 1/16 inch – 1/8 inch

PA12-steel transition fittings were produced by R.W. Lyall and installed on one end of each line segment with the appropriate steel-end connections (steel end caps and nipples) to facilitate pressurization. In addition, a Continental Industries Eliminator fitting (mechanical purge fitting) was installed for evaluation purposes.

MichCon Gas PA12 Material List	
Description	Manufacturer
4" SDR 11 PA12 pipe	George Fischer Piping Systems
4" PA12 to steel transition fittings	R.W. Lyall
4" PA12 electrofusion couplings	Friatec
4" PA12 end caps	George Fischer Piping Systems
Mechanical Eliminator purge fitting	Continental Ind.
Steel end cap	misc.
Steel nipple	misc.

**Table 3: Materials used on the MichCon Gas PA12 small scale installation**

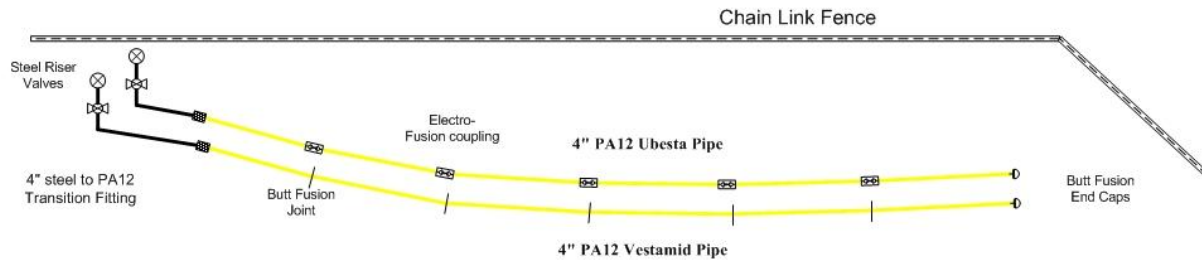
As with the previous small scale installation at WE Energies, problems were encountered with the electrofusion couplings. At first, a Central Plastics universal Easy Fuse processor was used without success. The coupling nodes would not fit the processors connectors. An Innogaz universal processor unit was then tried and one of the unit's adapters did fit the Friatec couplings. Another problem was encountered when trying to scan the bar code on the coupling with the scanning device on the processor unit. The bar code provides the processor the details of the fitting being fused for proper current and time requirements. It required 30 to 40 scans with the scanning device to finally read the bar code. Further investigation is needed to determine if this is a fitting bar code issue or a processor scanning device issue.

After all of the pipes and fittings were assembled, both pipe sections were lowered into the open trench. As depicted in Figures 18 and 25 below, the bottom of the trench contained about 6 to 8-







inches of water. The systems (Evonik and Ube) were then leak and pressure tested with nitrogen to 450 psig and held at this pressure for 1 hour. After a successful pressure test, the pressure was reduced and secured at the current operating pressure of 330 psig as requested by MichCon Gas.

The pipe was then backfilled with native soil. A pressure chart box was installed on the steel riser of each PA12 line in order to record the pressure throughout the evaluation period of the PA12 piping system.

A schematic of the installation is shown below:



- Approximately 240' of 4" PA12 pipe installed
- Both Butt Fusions and Electro-Fusion couplings were used to join the pipe and fittings
- Pressure tested to 450 psig with Nitrogen for 1 hour
- Operation Pressure = 330 psig

Transition Fitting	
Valve	
Pressure Gauge	
Electro-Fusion Coupling	
Butt Fusion	
Butt Fusion End Caps	

### **Michcon Gas - 4" PA12 Installation**

**Figure 17: MichCon Gas Small Scale Installation Schematic**



**Figures 18 and 19: Open trench and sections of PA12 strung out for installation**



**Figure 20 and 21: Facing and heating the pipe ends prior to joining the two pipe sections**



**Figure 22 and 23: Installing an electrofusion coupling – scraping and reading the bar code**



**Figures 24 and 25: PA12 system assembled and then installed in the open trench**



**Figure 26: Transition with end cap and nipple**



**Figure 27: Pressure test gauge at 450 psig**