

Polyethylene Gas Pipe.... Here Today, Here Tomorrow

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Abstract:

Solvay Polymers (now BP Solvay Polyethylene North America) has produced and sold polyethylene resins for gas distribution pipe manufacturing since the early 1980's. Recent developments at BP Solvay Polyethylene have resulted in the removal of numerous pipe specimens that have been on long-term hydrostatic test in excess of 70,000-80,000 hours at elevated temperatures. This paper examines the character of some of these pipe specimens and compares the physical properties and performance capabilities of these specimens before and after these extended, aggressive test conditions. As a result of this discussion, we gain insight into the affect of long-term stress on the durability of modern polyethylene piping systems and gain confidence in the ability of these materials to remain ductile despite prolonged periods of intensified stress.

Introduction:

The long-term strength of polyethylene (PE) pipe has traditionally been established using stress rupture data obtained on pipe specimens placed on test at specified conditions of stress and temperature. The data generated under this methodology is then analyzed using an industry established

stress regression algorithm to determine a time dependent long-term strength for the PE piping material.

Within the ASTM system prevalent throughout North American, we utilize ASTM D2837 to develop stress rupture data over a 10,000 hour time frame to determine a long-term hydrostatic strength (LTHS) at 100,000 hours which is subsequently categorized into one of a series of hydrostatic design bases (HDB's).(1) On a more global scale, the protocol established within ISO 9080 is used to generate stress rupture data that is then extrapolated to establish a 50-year minimum required strength (MRS) within the ISO system.(2)

While these analytical methods do differ at a technical level, both rely on the collection of stress-rupture data under controlled conditions of temperature and stress. In both systems, data is generated at various combinations of stress and temperature to determine the long-term performance capability of the piping material in accordance with industry established protocol.

In the paragraphs that follow, we will turn our attention to a slightly different aspect of long-term testing of PE pipe. Specifically, we will investigate the effect that long-term

exposure to these conditions of temperature and stress may have on PE pipe grade materials. In this paper, we will investigate various PE2406 and PE3408 developmental piping materials that have been under hydrostatic test at 60 degrees C for extensive time periods. Using these samples and comparing them to control specimens of the same pipe runs that were not hydrostatically tested will provide insight to the long-term performance capability of these PE piping materials.

The Data Set:

BP Solvay maintains an active pipe test lab at its technical center in Deer Park, Texas. This facility provides the capability of testing over 1100 pipe specimens at 5-6 different temperatures depending on the developmental needs of the organization.

Historically, pipe testing at this facility has been conducted in such a way as to allow testing to continue to failure of all pipe specimens at all temperatures for each data set developed. However, recent developments at BP Solvay Polyethylene have resulted in the removal of numerous pipe specimens that have been on long-term hydrostatic test in excess of 40,000 hours at elevated temperatures.

The sudden availability of these long-term test specimens combined with their respective control specimens that have been in storage since testing was initiated provides the basis for this paper. Table I provides an overview of the pipe run number, formulation, temperature, stress level and time under test for each of the pipe formulations discussed in the paragraphs which follow.

For those formulations in which sufficient 60 degree C data is available, an analysis is conducted on the impact that these long-term specimens have on the regression analysis established using the industry recognized 10,000-hour data. A comparative analysis is then made between long-term

stressed specimens and non-stressed specimens as it relates to basic physical properties and mechanical properties such as melt, index, density, oxidative induction time, tensile strength, quick burst, and slow crack growth resistance.

**Table I
Pipe Formulations Under Long
Term Stress at 60 Deg C**

Formulation	Color	Type	Stress, Psi.	Time, hours
A (R398)	Orange	PE2406	Control	NA
B (R398)	Orange	PE2406	700	115751
C (R443)	Black	PE2406	Control	NA
D (R443)	Black	PE2406	719	112840
E (R834)	Black	PE3408	Control	NA
F (R834)	Black	PE3408	826	52896
G (R761)	Yellow	PE2406	Control	NA
H (R761)	Yellow	PE2406	997	61488
I (R853)	Blue	PE3408	Control	NA
J (R853)	Blue	PE3408	853	46153
K (R833)	Black	PE3408	Control	NA
L (R833)	Black	PE3408	829	52940
M (R881)	Yellow	PE2406	Control	NA
N (R881)	Yellow	PE2406	798	42196
O (R202)	Orange	PE2406	952	147547

Long Term Strength:

As previously indicated, the long-term strength of PE piping materials is established in accordance with ASTM D2837 throughout North America. Using this methodology, the long-term strength of the material is determined by extrapolating a line based on data generated through 10,000 hours out to 100,000 hours on a stress versus time log-log plot. The intercept at 100,000 hours is the long-term hydrostatic strength (LTHS). The LTHS is then categorized into one of a series of hydrostatic design bases (HDB) that is subsequently used to stress-rate the piping product. The LTHS varies with temperature but is commonly determined at 23 degrees C.

In addition to establishing an LTHS for PE gas pipe material, ASTM D2837 also provides that the data set should be tested or “validated” to assure that the slope of the data generated in determining the LTHS

(and hence, the HDB) does not change. This change in slope would indicate a change in failure mode from ductile to brittle thereby lowering the actual 100,000-hour intercept.

ASTM D2513, the principal standard for plastic pipes in gas distribution applications, places additional requirements on PE materials used in the manufacture of pipe and fittings for these applications.(3) This standard requires that materials for these applications also maintain an elevated temperature HDB, such as at 60 degrees C, and that the HDB at 23 degrees C be substantiated as linear out to the 50 year time interval.

Table II
Additional Requirements for PE Materials in Gas Pipe

Standard	Section	Requirement
D2837	5.6 or 5.7	Validation Of HDB
D2513	5.6	Elevated Temperature HDB
D2513	A1.3.3	HDB Substantiation to 50 years

Based on the combination of these requirements, it is apparent that the nature of the stress regression extrapolation must be maintained as linear out through the 50 year design life of most gas piping systems even at elevated temperatures.

The test methods and mathematical models used to both validate and/or substantiate the long-term performance of PE gas pipe have been well researched and long established within the plastics piping industry. However, debate continues over the accuracy of these models as it relates to actual projection of long-term performance at elevated temperatures.

The data presented in this paper provides a rare opportunity to substantiate the performance of PE pipe at 60 degrees C using pipe failure data well beyond the 10,000-hour minimum established by

ASTM convention. Figure I and II present 60 degree stress rupture curves for data sets developed for formulations D(R443) and H(R761).

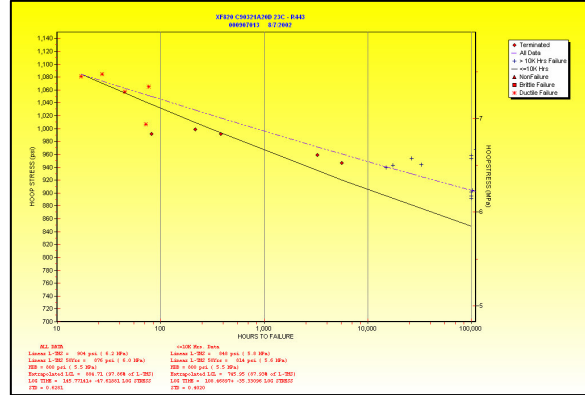


Figure I: 60 Degree C Regression Analysis of Specimen D(R443)

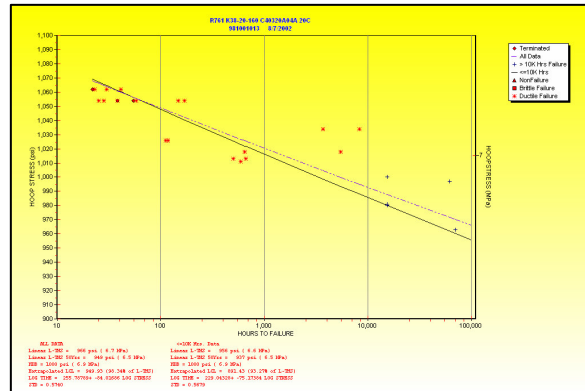


Figure II: 60 Degree C Regression Analysis of Specimen H(R761)

Both data sets for formulations D and H have been validated as being linear out through 100,000 hours at 60 degrees C. The solid lines in Figures I and II represent the 60 degree C 100,000 hour regression analysis of the data based on the traditional 10,000-hour data requirement. The dashed lines in both figures represent the 60 degree 100,000-hour regression of both data sets with inclusion of the additional data points from 20,000 to 100,000 hours.

Table III provides a summary of the 100,000-hour and 50-year intercepts with and without the inclusion of the extended data sets for both formulations D(R443) and H(R761).

Table III: The Effect of Extended Data Point Inclusion on LTHS Projection

Analysis	100,000 hour Intercept, psi	50-year Intercept, psi
D(R443) 10,000 data	861	828
D(R443) Full data set	905	877
H(R761) 10,000 data	956	937
H(R761) Full data set	966	949

Physical Property Analysis:

Representative samples of all the pipe formulations, both pressurized and control, were then analyzed for the following physical and mechanical properties.

- Melt index
- Density
- Quick burst
- Tensile properties
- Oxidative induction time
- PENT

Through a comparative analysis of these properties for both the stressed and the control specimens, the effect of exposure to long-term, sustained stress on PE pipe can be more fully investigated.

Melt Index and Density:

Representative samples of each formulation were first analyzed for melt index and density in accordance with ASTM D1238 and ASTM D1505, respectively. The data obtained as a result of these analyses are presented in Table IV that follows.

Table IV: Melt Index and Density Results

Formulation	Long Term Stress, Psi.	Time on LT test, hours	Melt Index gr/10m in	Density Gr/cc
A (R398)	Control	NA	0.209	0.9411
B (R398)	700	115751	0.205	0.9404
C (R443)	Control	NA	0.626	0.9395
D (R443)	719	112840	0.602	0.9394
E (R834)	Control	NA	0.095	0.9468
F (R834)	826	52896	0.095	0.9461
G (R761)	Control	NA	0.214	0.9404
H (R761)	997	61488	0.203	0.9398
I (R853)	Control	NA	0.156	0.9432
J (R853)	853	46153	0.162	0.9431
K (R833)	Control	NA	0.165	0.9445
L (R833)	829	52940	0.161	0.9441
M (R881)	Control	NA	0.206	0.9411
N (R881)	798	42196	0.216	0.9416
O(R202)	952	147547	0.218	0.9394

Quick Burst:

Representative samples from each formulation, both control and stressed, were submitted in duplicate for quick burst analysis at 23 degree C in accordance with the requirements of ASTM D1599.(4) The data generated from this testing is presented in Table V below.

Table V: Quick Burst Results

Formulation	Long Term Stress, Psi.	Time on LT test, hours	Avg Burst Press, psi	Burst Quality
A (R398)	Control	NA	945	Ductile
B (R398)	700	115751	935	Ductile
C (R443)	Control	NA	1144	Ductile
D (R443)	719	112840	1112	Ductile
E (R834)	Control	NA	1327	Ductile
F (R834)	826	52896	1202	Ductile
G (R761)	Control	NA	1292	Ductile
H (R761)	997	61488	NA	Ductile
I (R853)	Control	NA	1287	Ductile
J (R853)	853	46153	1205	Ductile
K (R833)	Control	NA	1397	Ductile
L (R833)	829	52940	1267	Ductile
M (R881)	Control	NA	1299	Ductile
N (R881)	798	42196	1249	Ductile

The reader will note that Specimen “O” was not submitted for quick burst analysis. As shown in Table I, Specimen O had undergone extensive elevated temperature testing, 147,547 hours, or roughly 17 years. At the time the specimen was removed from hydrostatic test there was only one pipe sample remaining. As such, this sample was exempted from the quick burst test regime to facilitate other physical and mechanical testing.

Tensile Properties:

Pipe samples from each formulation in the data set were then analyzed for tensile properties. This testing required that the pipe samples be cut into equally sized sections, roll-milled and then pressed into a plaque under standard laboratory conditions following ASTM procedures. Tensile coupons were then cut from the plaques and submitted for testing at 23 degrees C in accordance with the requirements of ASTM D638.(5) The data is presented in Table VI.

Table VI: Tensile Test Results

Formulation	Long Term Stress, Psi.	Tensile Strgth @ Yld, psi	Tensile Elong @ Yld, %	Tensile Elong @ Brk, %
A (R398)	Control	3020	13.3	839
B (R398)	700	3000	13.4	759
C (R443)	Control	2920	21.1	944
D (R443)	719	2910	15.1	641
E (R834)	Control	3410	9.4	895
F (R834)	826	3400	9.4	754
G (R761)	Control	2930	13.3	906
H (R761)	997	2920	13.2	856
I (R853)	Control	3120	10.2	825
J (R853)	853	3160	12.6	789
K (R833)	Control	3230	13.4	919
L (R833)	829	3270	9.4	577
M (R881)	Control	2960	10.8	779
N (R881)	798	3030	12.4	719
O(R202)	952	2920	14.0	852

Thermal Properties via OIT:

Representative samples of each formulation were then analyzed for

oxidative induction time at 210 degrees C via a differential scanning calorimeter (DSC). Each pipe sample was sectioned and OIT results were obtained on specimens from the inner, mid and outer wall of each sample. The results of this analysis are presented in Table VII below.

Table VII: Oxidative Induction Time at 210 Deg C

Formula	Long Term Stress, Psi.	OIT Inner Wall, Min	OIT Mid Wall, Min	OIT Outer Wall, Min
A (R398)	Control	27.0	51.0	10.0
B (R398)	700	12.0	16.0	14.0
C (R443)	Control	16.0	25.0	9.1
D (R443)	719	1.6	2.4	1.4
E (R834)	Control	24.0	35.0	19
F (R834)	826	0.5	4.6	1.6
G (R761)	Control	69.0	84.0	63.0
H (R761)	997	1.4	19	16
I (R853)	Control	78.0	85.0	69.0
J (R853)	853	3.5	5.2	11.0
K (R833)	Control	26.0	42.0	27.0
L (R833)	829	2.0	4.6	2.2
M (R881)	Control	55.0	48.0	29.0
N (R881)	798	7.0	15.0	0.9
O(R202)	952	2.5	6.4	6.2

Resistance to Slow Crack Growth (PENT):

The final test to be conducted on both the stressed and control pipe formulations is resistance to slow crack growth. To facilitate this, PENT specimens were pressed and machined from plaques produced from the remaining pipe specimens for the formulations shown in Table VIII. The PENT specimens (coupons) were placed on test in an instrumented PENT apparatus in accordance with ASTM F1473 and tested to failure or to a level in excess of 100 hours at 80 degrees C and a stress level of 2.4 MPa.(6) Individual times to failure and the average time to failure for each formulation are shown in Table VIII.

**Table VIII: PENT Results
at 80 degrees C, 2.4 Mpa Stress**

Formula	Long Term Stress, Psi.	PENT Coupon A, hours	PENT Coupon B, hours	Avg PENT hours
C (R443)	Control	167.6	152.5	160.8
D (R443)	719	501.2	451.2	476.1
E (R834)	Control	115.6	92.90	104.3
F (R834)	826	184.3	200.9	192.6
G (R761)	Control	IS*	IS*	IS*
H (R761)	997	IS*	IS*	IS*
I (R853)	Control	IS*	IS*	IS*
J (R853)	853	IS*	IS*	IS*
K (R833)	Control	> 240	> 240	> 240
L (R833)	829	> 240	> 240	> 240
M (R881)	Control	> 1275	> 1275	> 1275
N (R881)	798	> 1275	> 1275	> 1275

* Insufficient Sample

Discussion:

The test regime undertaken on the pipe formulations in this paper has resulted in a considerable amount of data of a highly diverse nature. The discussion that follows will focus on two essential aspects of the data generated. First will be a brief discussion on the significance of the 60-degree hydrostatic performance of two of the formulations; specifically D(R443) and H(R761). Secondly, we will take a closer look at the data generated on the physical properties of both the tested and the control specimens as it relates to any fatigue effect produced as a result of the extensive period of hydrostatic testing at 60-degree C.

Hydrostatic Performance

Clearly, it would have been beneficial to have had full 60-degree data sets for all of the pipe formulations included in this study. However, one should bear in mind that these formulations represent different stages and types of analytical or developmental initiatives within BP Solvay Polyethylene. As such, full 60-degree C curves were not necessarily the overall research goal for each formulation included in the test population investigated here.

That said, however, formulations D(R443) and H(R761) for which full 60 degree curves do exist provide a rare opportunity to investigate the impact of extended hydrostatic testing on the regression models used to establish the long-term hydrostatic strength of PE piping compounds. The extensive nature of the testing undertaken on formulations D(R443) and H(R761) allows us the opportunity to look well beyond the 10,000-hour requirement and investigate the validity or accuracy of the stress regression model itself.

Both the ISO and the ASTM method of regression analysis utilize data generated out through the 10,000-hour time frame to project a long term strength at a specified time interval. The ATSM methodology assumes that the failure mode for the data generated in this type of testing does not change; that it remains ductile in nature. To insure that the failure mode remains ductile, a test is conducted to insure that the slope of the curve does not change. This test is called “validation” or “substantiation” depending on the standard referenced.

The pipe test data generated for both D(R443) and H(761) were analyzed in accordance the requirements of ASTM D2837 in two separate and distinct manners. First, data for each formulation was submitted for regression analysis without the inclusion of the failure points that were out beyond the 10,000-hour level. The LTHS and 50-year intercepts generated for these regressions were then compared to those generated on the full data sets that included multiple data points out beyond the 10,000-hour requirement.

The results of the regression analyses were presented in Table III. From this we see that the inclusion of these points and the subsequent effect on the 100,000-hour intercept provides confirmation of the linearity of the regression curve and illustrates the conservative nature of the rate process method of stress rupture analysis.

If the regression is carried out to the 50-year intercept we once again see that the inclusion of the extended data sets increases the value of the 50-year intercept for both sets of data. The positive impact of the inclusion of these extended data points illustrates the conservative nature of the regression methodology and serves as confirmation of the mathematical model incumbent to the validation algorithm.

Physical Properties

The physical and mechanical testing presented in this discussion was undertaken to investigate any impact that extended exposure to the 60-degree C test conditions may have had on the integrity of the pipe specimens analyzed. We would anticipate that a reduction or degradation in the physical or molecular properties of the formulations would indicate deterioration in serviceability.

Embrittlement or heat aging of the formulations exposed to these test conditions would be indicated by a significant change in the melt index or the density of the formulations. A change in the melt index from the control specimen of each formulation could indicate molecular damage or alteration from the original formulation. Chain scission or potentially even molecular cross-linking which could occur as a result of extensive exposure to these test conditions would be indicated by a significant change in the melt index of the formulation.

Similarly, one could anticipate a change in density associated with strain hardening of the polymer over time or heat aging of the pipe specimens as a result of the elevated temperature exposure associated with the 60-degree C hydrostatic testing.

Analysis of both the exposed specimens and control specimens of each formulation shows no statistical change in either melt index or density. The data presented in Table IV clearly reflect little to no real

difference in molecular weight as evidenced by melt index. Similarly, for each formulation, the density of the exposed specimen is comparable and consistent with that of the original unexposed control specimen. Obviously, there is some variability in the data. However, this is felt to be consistent with the normal variability associated with this type of testing.

While melt index and density are key indicators of the polymeric structure of a plastic pipe formulation, mechanical tests also exist that would help provide evidence of deterioration of the piping formulations as a result of the extended hydrostatic testing. It is reasonable to assume that a loss in ductility could be expected as a result of the extensive 60-degree C hydrostatic testing. Two key tests, tensile strength and quick burst, were conducted in an attempt to discern a difference in ductility between the control and the exposed specimens for each pipe formulation.

From Table V, we see that quick burst testing of both the control and the exposed specimens of each formulation resulted in markedly similar results. All of the pipe specimens subjected to quick burst analysis resulted in ductile failures at similar ultimate burst strengths.

Photos of all of the quick burst specimens could not be reproduced here due to space limitations. However, two of the more photogenic failures obtained as a result of testing Specimens M(R881) and N(R881) are presented in Figure III. The reader will note the classic “parrot’s peak” or “fish mouth” appearance typical of a ductile quick burst failure mode as exhibited by Specimens M(R881) and N(R881).

Tensile testing conducted on both the control and the exposed specimens of each formulation provides further indication that despite the extended test period each formulation still exhibits a ductile character. In every case the tensile strength at yield is

consistent between the control and exposed specimens for each formulation. However, it can also be seen from the data that the elongation at break is somewhat reduced for each formulation. This is not unexpected as a slight reduction in elongation is anticipated as a result of sustained exposure to stress. It should be noted, however, that none of the specimens exhibited an elongation at break less than 500%, suggesting acceptable retention of ductile properties.



Figure III: Quick Burst of Specimens N(R881) on left and M(R881) on right

Thermal analysis is one area in which we see a significant difference between the exposed and control specimens of each formulation. Traditionally, analytical techniques such as differential scanning calorimetry (DSC) are employed to determine a polyethylene compound's resistance to the onset of oxidation. The oxidation induction time (OIT) that is obtained as a result of this technique provides a relative indication of the ability of the polymer's additive system to resist oxidation under controlled conditions of heat and time. The greater the OIT in minutes the greater the resistance to the onset of oxidation, or degradation due to oxidation.

Generally speaking, polyethylene pipe materials are formulated with a small amount of various UV stabilizers, anti-oxidants and/or heat stabilizers.(7) These

are generally separate and distinct from the pigmentation system with one notable exception being carbon black which serves a dual function role; both UV stabilization and pigmentation.

The additive systems are compounded into the base polyethylene resin to protect the polymer from degradation due to exposure to high temperature, oxidation or irradiation by ultra-violet light. The chemistry for these additives systems and the mechanisms by which they protect the polymer do vary to some degree. However, many of them rely on sacrificial consumption of the additive over extended periods of exposure to effectively protect the polymer.

For this reason, the difference in oxidation induction times between the control specimens and the specimens exposed to sustained 60-degree testing is not unexpected. It is reasonable to assume that a major portion of the additives system have been consumed or exhausted as a result of the long-term exposure to the elevated temperature. Taken in context of the physical testing results obtained in this study, it is clear that the additives systems utilized in these formulations have proven very effective.

The extremely low OIT numbers obtained in thermal analysis of the exposed specimens as compared to the control specimens may justify additional investigation that is beyond the scope of this writing. Visual inspection of each pipe specimen did not reveal any indication of brittle failure or surface crazing associated with the onset of stage III type phenomena.(8) However, a more thorough analysis is required to determine the significance of the extremely low OIT numbers as it relates to lifetime prediction or the potential effect that additive exhaustion may have on long term serviceability.

The final property to be analyzed in this test regime was that of resistance to slow crack growth. The premise here is that if the

pipings formulations had been negatively affected by the 60-degree hydrostatic testing then the inherent slow crack growth resistance of the exposed specimens would show some significant difference when compared to that of the original control specimens of each formulation. For this reason, control and exposed specimens from each formulation were placed on PENT testing as shown in Figure IV, below. The specific test conditions were 80 degrees C and 2.4 MPa. Unfortunately, however, PENT testing could not be concluded in time for inclusion in this paper submittal. It is anticipated that the PENT results will be available for presentation and review at the time of the symposium.



Figure IV: PENT Test Apparatus at BP Solvay Polyethylene

The data generated to date as a result of this investigation clearly supports the hypothesis that long-term exposure of these pipe specimens to extreme conditions of stress and temperature has resulted in very little deterioration in the serviceability of the pipe formulations under study.

While the entire test regime could not be completed in time for publication of this paper, a considerable amount of information can be drawn from the data generated to date. We fully anticipate that the completion of the slow crack growth studies (PENT) will support the premise that any fatigue effect produced by long-term exposure to these conditions of stress and

temperature will be negligible. However, this must be confirmed by completion of the test regime and analysis of the data generated.

Conclusions:

This discussion has taken advantage of the availability of pipe specimens which have been subjected to extensive periods of hydrostatic testing at 60 degree C to investigate any potential fatigue or deterioration in the serviceability of these polyethylene pipe formulations. We see that the polyethylene pipe formulations presented here show very little indication of deterioration in serviceability based upon a diverse array of physical and mechanical testing on both exposed (or stressed) specimens when compared to control specimens of the same pipe formulation.

From this investigation, the following conclusions are drawn:

- 1) The inclusion of the pipe specimens that were on test greater than 10,000 hours in the traditional ASTM D2837 stress rating protocol provides “real world” substantiation of the linearity of the stress regression model inherent to this rating method.
- 2) Comparative physical property testing of both the exposed pipe specimens and the control specimens suggests no impact on the molecular character of the base resins from which these formulations were produced.
- 3) Traditional tensile testing and quick burst testing provide no indication of degradation in the mechanical properties of these formulations as a direct result of exposure to long-term stress as exemplified by 60 degree C hydrostatic testing.
- 4) Thermal analysis of specimens subjected to the elevated temperature

testing does confirm some sacrifice in stabilization when compared to control specimens of the same formulation. While not unexpected in nature, the significance of this phenomenon may provide focus for future research and discussion.

- 5) Finally, any impact on the slow crack growth properties of these formulations could not be presented here due to time constraints. However, this data will be available for presentation at the time of the symposium.

The reader is advised that not all of the formulations investigated here were developed specifically for natural gas distribution applications. Many were, but some of the formulations were developed to address research needs in other piping end uses such as water, sewer or industrial applications. This does not, in the author's opinion, detract from the significance of the investigation and discussion generated here. Rather the diversity of the formulations investigated provides for a more robust investigation. One that provides confidence in the fact that properly designed, manufactured and installed polyethylene gas pipe will be here today....and here tomorrow.

Acknowledgements:

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