Final Report

Research Synthesis and Recommendations for Corrugated High Density Polyethylene Pipes

Prepared for the

Florida Department of Transportation

by

Richard D. Granata, Ph.D.
Florida Atlantic University
Department of Ocean Engineering
101 North Beach Road
Dania Beach, FL 33004

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ABSTRACT

A Technical Review Group (TRG) was constituted by the Florida Department of Transportation to consider the adequacy of specifications for high density polyethylene corrugated pipe (HDPE CP) used for drainage purposes and the related topics of research synthesis and direction. Technical challenges had been raised by competing industry groups. Upon consideration of the challenges and broader concerns, the findings of the TRG were that the current specifications have incorporated the state-of-practice test methods to evaluate the long-term properties of corrugated HDPE pipes. Three members were in favor of this finding, two were not and the facilitator concurred with the majority. While, members of the TRG regarding the extrapolation methods and the sensitivity of the test methods expressed different opinions, the current specifications were adequate, areas of improvement were possible and specific additional areas were identified for research synthesis and direction. These areas included:

1. Tests could be developed to determine the onset of HDPE oxidation versus antioxidant depletion and which is more relative to the oxidation mechanisms of HDPE corrugated pipe.
2. Alternate techniques capable of providing additional information on brittle surface layers could be considered for development, and the correlation of such surface oxidation phenomenon to the design life prediction for HDPE corrugated pipe.
3. Identify the primary stress mode in the corrugated pipe walls under compression loading. If other than tensile mode (K_I) is presented, multimode stresses should then be considered in more detail to determine the relevance of such stresses.

These proposed research areas have the potential to advance, in the sense of continuous improvement, existing FDOT specifications that considered technically adequate and representative of current state-of-the-practice of the industry and engineering community.

1. INTRODUCTION

Recent changes to FDOT's specifications set forth the requirements for high density polyethylene (HDPE) corrugated pipe to be used on projects requiring 100-year design service life. The specification is based on a FDOT sponsored study carried out by Drexel University. Concurrent with FDOT's aforementioned specification change, competing pipe manufacturing industry groups presented technical challenges to FDOT's sponsored study that formed the basis for Class II or 100-year performance requirements. The differences in technical opinions between those in the Drexel report and those contained in the technical challenges are complex and must be addressed drawing intensely upon fundamental knowledge of polymer science. There is a need for a synthesis representing the combined knowledge and technical guidance of a body of experts versed in materials fundamentals, polymer science and manufacturing processes.

The Drexel report [1], entitled, “Protocol for Predicting the Long-term Service Life of Corrugated High Density Polyethylene Pipe,” and related documentation are available at the following link:

http://www.dot.state.fl.us/statematerialsoffice/laboratory/corrosion/hdpe/index.htm

A list of these documents is provided in Appendix A.

Note: This research effort relates only to Part II of the above-referenced Drexel report.

2. METHODS

The Principal Investigator (PI) established and provided direction to a technical review group (TRG) comprised of five individuals with advanced knowledge in appropriate areas of materials science and/or manufacturing processes. The PI was permitted to supplement the technical review group with persons of related specialized knowledge as needed. TRG members were selected primarily on the basis of their technical capabilities as representing the contrasting views on the topic. The members were recommended by different parties, including Florida Department of Transportation, American Concrete Pipe Association, and Corrugated Polyethylene Pipe Association.

The TRG members were:

Grace Hsuan, Ph.D.
Associate Professor of Civil, Architectural and Environmental Engineering
Drexel University

Alexander Chudnovsky, Ph.D.
UIC Distinguished Professor
Professor of Mechanics and Materials
Director of Fracture Mechanics and Materials Durability Laboratory
University of Illinois at Chicago

Salvatore Stivala, Ph.D.
Rene Wasserman Professor Emeritus
Professor Chemistry and Chemical Biology
Stevens Institute of Technology (retired)

Clayton Ormsby, Ph.D.
Federal Highway Administration (retired)

Stephen Boros, Director
Plastics Pipe Institute

Richard Granata, Ph.D. (facilitator)
Professor of Ocean Engineering
Center for Marine Materials
Florida Atlantic University

Three meetings of the TRG were held:

1. March 10, 2006, at FDOT, Gainesville, FL, all members present.
2. April 7, 2006, Florida Atlantic University, Dania Beach, FL, all members present.
3. May 11, 2006, Florida Atlantic University, Dania Beach, FL, Dr. Stivala via speaker phone and Dr. Chudnovsky, unable to participate due to extensive overseas travel.

National Academy of Science procedures were followed regarding constitution of the panel and conflict of interest [2]. Based upon individual input, the TRG believed that the panel was properly constituted and that there appeared to be no conflict of interest.

Hard copies of documents listed in Appendix A were provided to each TRG member.

The TRG conducted a critical review of FDOT's revised Section 948 specification for corrugated HDPE pipe [3] along with the Drexel study, related testing protocols and the technical challenges posed by various industry interests. Based on the technical reviews, the TRG undertook:

1. To identify any technical flaws in the Drexel study, the Section 948 specification and related test protocols.
2. To provide a research synthesis that gives specific direction to FDOT on any needed revisions to specifications or test protocols.
3. To provide direction that ensures that the methodology used to predict service life of HDPE pipe is of at least comparative technical rigor as that used for other types of pipes and construction products.

All topics of concern pertinent to the review were raised and accepted for discussion followed by narrowing of those topics to critical issues. Literature of interest to the TRG was shared to provide adequate basis for discussions. In the course of the meetings, oxidation and cracking emerged as divisive topics.

The TRG facilitator selected Stephen Boros to write an account for the committee's report of the technical background and synopsis of the Section 948 specification based on the Drexel study. This assignment required detailed knowledge of the fabrication process and history of the material application development. The account served as the framework for the remaining deliberations to which other TRG members contributed regarding research synthesis and direction. The account is presented below followed by additional discussion and critique regarding oxidation and cracking.

3. TECHNICAL REVIEW

3.1. Account of Technical Background and Synopsis

3.1.1. Current FDOT Interim Specification.

Table 1

Stress Crack Resistance of Pipes			
Pipe Location	Test Method	Test Conditions	Requirement
Pipe Liner	FM 5-572, Procedure A	10% Igepal solution at 50°C 600 psi and applied stress 5 replicates	Average failure time of the pipe liner shall be ≥17 hours; no single value shall be less than 12 hours.
Pipe Corrugation* (molded plaque)	ASTM F 2136	10% Igepal solution at 50°C 600 psi applied stress	Average failure time shall be ≥24 hours; no single value shall be less than 17 hours.
Junction**	FM 5-572, Procedure B and FM 5-573 ASTM D 2837	Test temperature 80°C and applied stresses of 650 and 450 psi. Test temperature 70°C and applied stress of 650 psi 5 replicates at each stress level	Calculate three constants Failure time at 500 psi at 23°C ≥ 100 years (95% statistical confidence)
		Single Test: Test temperature 80°C and applied stress of 650 psi. 5 replicates	The failure time must be equal or greater than the calculated value using the three constants from the three points test
Longitudinal Profile**	FM 5-572, Procedure C, and FM 5-573 ASTM D 2837	Test temperature 80°C and applied stresses of 650 and 450 psi. Test temperature 70°C at applied stress of 650 psi 5 replicates at each stress level	Calculate three constants Failure time at 500 psi at 23°C ≥ 100 years (95% statistical confidence)
		Single Test: Test temperature 80°C and applied stress of 650 psi., 5 replicates	The failure time must be equal or greater than the calculated value using the three constants from the three points test
Oxidation Resistance of Pipes			
Pipe Location	Test Method	Test Conditions	Requirement
Liner and/or Crown	OIT Test (ASTM D 3895)	2 replicates (to determine initial OIT value)	25 minutes, minimum
Liner and/or Crown	Incubation test FM 5-574 and OIT test ASTM D 3895	Three samples for incubation of 195 days at 80°C and applied stress of 250 psi. One OIT test per each sample.	Average OIT value shall be ≥3 minutes (no single value shall be less than 2 minutes)

Note: FM= Florida Method of Test.
* Required only when corrugation resin is different than liner resin.
** A higher test temperature (90°C) may be used if supporting test data acceptable to the State Materials Engineer is submitted and approved in writing.

3.1.2. Design Life Factors of Corrugated HDPE Pipes

The test protocols in the current interim specification are designed to evaluate the potential limiting factors related to the design life of the corrugated HDPE pipes under service conditions which are:

- Analysis to identify governing stress in the pipe under service conditions
- Stress crack resistance (SCR) of the resin
- SCR of the pipe
- Testing of "critical" areas of the pipe where failure is likely to occur
- Oxidation resistance of the resin and pipe to assure mechanical properties will not decline to the level that can affect the performance of the pipe during the service life.

Background on these five factors has been described in the report, entitled, *"Protocol Prediction Long-Term Service of Corrugated HDPE Pipes"* [1]. Analysis of governing stresses has already been determined based on Dr. McGrath's work and will not be addressed further in this report.

3.1.3. Stress Crack Resistance of Corrugated PE Pipes: Liner Tests

In addition to American Association of State Highway and Transportation Officials (AASHTO) M294 requirements, the FDOT interim specification (Section 948) sets material requirements for the corrugated PE pipes. Both the liner and the corrugation material (if different) must pass a notched constant ligament stress (NCLS) test as an index of the material's resistance to stress cracking. The values set in the specification have been established based on the NCHRP 4-26 report for pipe liner (paper was presented in Transportation Research Board (TRB), 2007). Background on this report is included as Appendix B.

It is important that corrugated HDPE pipes have a certain ability to resist stress cracking under the slow crack growth mechanism. NCLS test has been proven to be a valid index test to quantify the SCR property of pipe resins and pipes (NCHRP 429 and TRB paper). This interim specification adequately addresses the initial qualification of the corrugated PE pipe for a Class II or 100 year service life.

3.1.4. Stress Crack Resistance of the Corrugated Pipe Including Critical Areas: Junction Tests

Pipe design, manufacturing, and service conditions will have an effect on the service life of HDPE corrugated piping systems. It is important to perform testing under conditions that fairly represent the service conditions of the product. It has been demonstrated that one of the critical areas of a corrugated PE pipe is where the corrugation joins with the liner, otherwise called the junction. Due to the geometry of the junction, it will act as an area of stress concentration and be the point where stress cracking is most likely to occur. This is supported by extensive field failure investigation reported by Hsuan and McGrath (NCHRP report 429) [4].

In order to evaluate the potential for the junction to survive the desired service life under expected service conditions, the interim specification requires the pipe junction be evaluated by the Rate Process Method (RPM). Additionally, the longitudinal profiles (vent hole and molded line) must also pass the RPM evaluation.

RPM uses Arrhenius principles to forecast design life under a given set of conditions of temperature and stress by performing tests at elevated temperatures. The RPM has been used for nearly 30 years to predict the long-term performance of PE piping, and lends itself very well for this application. Testing by Dr. Hsuan demonstrates that the RPM evaluation of junction specimens cut from corrugated PE pipe can also be assessed by this method. Data [1] shows that RPM evaluation of the junction does follow an Arrhenius response at elevated temperatures. The empirical model developed from the RPM evaluation can be used to forecast the service life of the most critical area of concern in corrugated HDPE pipe at the proposed service conditions.

The service conditions proposed by this FDOT interim specification is an average temperature of 23°C and a maximum tensile stress of 500 psi. This service temperature is supported by agricultural data showing that the average soil temperature at a depth of 6 inches is 23°C or less in the State of Florida – excluding the Keys. The maximum service stress was derived in a report from Dr. McGrath [1] using finite element analysis and indicates when the corrugated PE pipe is installed appropriately, “the long-term tensile strain in the pipe should be less than 1.6%, corresponding to a long-term stress of approximately 320 psi.” Applied to this value is a factor of safety of 1.5, resulting in a minimum tensile stress of 500 psi. These proposed long-term service conditions of 23°C and 500 psi tensile stress are further detailed in the report [1]

entitled, “Protocol for Predicting Long-term Service of Corrugated High Density Polyethylene Pipes “.

The proposed RPM test conditions of 80°C, 650 and 450 psi, and 70°C, 650 psi result in stress crack growth failures. This is the same expected failure mode for corrugated PE pipe in service – as seen in inspection of actual field failures. By testing specimens cut directly from the corrugated PE pipe and generating failure data under the specified conditions, the RPM evaluation of the junction and the longitudinal profile is able to forecast whether the corrugated PE pipe will exhibit a design life in excess of Class II or 100 years at the service conditions of 23°C and 500 psi tensile stress. This methodology is considered a sound and appropriately conservative approach for service life forecast of corrugated PE pipe.

The effect of scaling factors was discussed by the TRG. Scaling could be pertinent when testing a small sample and applying the results to a larger population. The stress crack resistance testing does not test the entire pipe, but rather multiple specimens cut directly from the pipe. Performing this type of testing on full samples of large diameter pipe is not always practical or possible. There is a possibility for these specimens to not reflect all potential performance deficiencies. This is true of any test that does not include 100% testing of a component. However, the RPM testing on the junction does require multiple specimens from around the circumference of the pipe to initially qualify the corrugated PE pipe. In addition to the initial qualification testing in the interim specification, there is an extensive program involving QA/QC which will continually monitor these properties. This program, known as the National Transportation Product Evaluation Program (NTPEP) [5] under the aegis of AASHTO, provides improved supervision of product Quality Control, Quality Assurance (QC/QA) through collaboration among manufacturers. Thus, there will be improved uniformity and improved reliability of test results with minimization or elimination of potential scaling factor effects.

Some discussion suggested that there may be stresses other than tensile stress in the corrugated PE pipe – such as shear stress or compressive stress. The finite element analysis performed by Dr. McGrath did not propose these other stresses and there is currently no accepted design methodology that includes shear or compressive stress as a major or limiting stress in corrugated PE pipe. However, this may be an area where further research could be considered.

3.1.5. Oxidation Resistance of Pipes

As presented in the previous background information, oxidative degradation is a well known phenomenon that can lead to failure of PE piping. If oxidation progresses to the extent that key mechanical properties are adversely affected, this is an indication that the polymer could develop stress cracking – leading to a loss of ductility and potentially brittle cracking. It is important to assure that oxidation degradation of the polymer does not progress to this extent over the desired service life of the pipe.

The PE resin must contain sufficient antioxidants (AO) to protect the resin during both processing (i.e. extruding into pipe) and over the design life of the product. The FDOT interim specification takes a multi-faceted approach to evaluating the PE resins and corrugated PE pipe. A common test to evaluate the AO in a polymer system is the oxidation induction time (OIT) test. This is an established ASTM standard test method so it has been thoroughly developed in a manner that makes it well understood and yields results that can be easily compared. There have been many papers written on the applicability of the OIT test and how it relates to the AO effectiveness. It is recognized that the OIT test can not be used to forecast the life of an AO package under specific service conditions due to the high testing temperature. However, this test can be used as a tool to evaluate the relative effectiveness and the residual effective AO remaining in the polymer over time. The FDOT interim specification utilizes multiple tests to determine if the AO package is capable of protecting the pipe over its design life by evaluating:

1– Initial OIT of the pipe and/or crown to determine that AO is present in sufficient levels after processing.
2– After incubation of the pipe specimen, that the AO will adequately resist extraction, hydrolysis, and degradation over the design life.
3– The requirement of a minimum residual OIT response after incubation to assure that the AO system has not been completely exhausted over the design life.

Firstly, the initial OIT minimum value of 25 minutes was determined based on previous work by Dr. Hsuan. By requiring the OIT be performed on the pipe liner and/or crown of the corrugated PE pipe, rather than on the PE resin, it is assured that a sufficient amount of AO is still present in the pipe and has not been overly consumed during processing.

Secondly, the incubation of samples cut from the corrugated PE pipe is designed to simulate a Class II or 100 year design life under the service conditions in Florida – 23°C average temperature in a water environment. There are several considerations that were involved in determining this incubation process. Based on research performed by Dr. Hsuan, a water environment was considered far more aggressive on an AO package than an air environment. While there is less oxygen in water than air, the water is much more aggressive for extraction and hydrolysis of the AO package. It is considered that this conservative approach is more appropriate since it is common for the corrugated PE pipe to be partially or completely submerged in water for most, if not all, of its service life. The conditions of the incubation are set at 195 days (4680 hours) in a circulated water bath at 85°C, while the specimen is under a constant stress of 250 psi. This time and temperature is based on using an Arrhenius relationship assuming an activation energy of unstabilized PE. There are some references that propose a higher activation energy; however, it was desired to use a very conservative approach for this aspect of the oxidation incubation test. Placing the specimen under stress during the incubation achieves two things: 1) the stress state in the specimen acts as a further accelerator for oxygen, water and AO migration through the polymer matrix, and 2) since the stress corrosion cracking mechanism is only minimally dependent on stress, if oxidative degradation occurs during the incubation period this stress will quickly progress to a failure.

Thirdly, the oxidation resistance test in the interim specification requires a final residual OIT value after incubation. It has been demonstrated that as long as there are still AOs present in the polymer, they can protect the polymer from oxidative degradation. The interim specification requires a three minute OIT result after incubation aging. This will assure that the pipe has not entered the stage III, or oxidation degradation phase and the pipe will continue to maintain critical physical properties.

There was some question on whether using the OIT on the “bulk” pipe specimen is appropriate, or whether the OIT test should be performed on the surface of the specimen. Work performed by Dr. Cheng at Exxon Mobile examined pipe specimens that had been incubated according to the interim specification requirements. Using FTIR, the specimens were analyzed for the presence of the carbonyl functional group which is a well known and accepted indication of PE degradation. Testing of the 2.5 mm thick specimens was done on the surface, 0.5 mm removed, and 1 mm removed. None of the specimens displayed any presence of the carbonyl

groups, indicating that degradation did not occur in these specimens – not even on the surface. Further discussion on the FTIR method suggested that the specific technique used may not be sensitive enough to detect very small levels of oxidation on the surface of the exposed specimens. While the TRG agreed that there is oxidation on almost every surface if examined closely enough, it is not the purpose of the interim specification to determine the onset of oxidation, but rather to assure that mechanical properties (as indicated by changes to elongation at break and melt index), are not affected to the degree that integrity of the pipe is compromised to the point that the pipe might be susceptible to cracking. This is further supported by previous work by Dr. Hsuan showing how the incubation process reduced the bulk OIT results, but did not affect tensile strength, elongation at break, or melt index. These are key properties commonly used to evaluate the effectiveness of antioxidant packages in polyolefins.

Other discussion centered around polybutylene (PB) pipe failures and that surface oxidation played a role in these failures. It is accepted that the major oxidation mechanisms are the same for both PE and PB. Some limited data was presented showing that PE is several orders of magnitude more resistant to these oxidation mechanisms than is PB, and the PB failures were exclusively related to potable water pipe failures where free chlorine was present and played a significant role in the oxidation degradation failures of the PB piping and polyacetal fittings. There are no free oxidizing agents such as chlorine in storm water in Florida. PE is generally considered to be very resistant to mildly acidic and basic environments; however, a possible area for more research may be the study of storm water constituents in the State of Florida and how they may play a role in the oxidation degradation of PE and other piping materials used in Florida.

3.1.6. Conclusions on Interim Specification 948

Discussions by the TRG over the course of three meetings addressed many areas that potentially could affect the long-term performance of corrugated PE pipe. The FDOT Interim Specification on Corrugated PE Pipe appears to adequately cover the known failure concerns – SCR of PE resin, SCR of corrugated PE pipe junction and profile, oxidation resistance including potential for extraction of AO in a water environment. In agreement with this statement were Dr. Ormsby, Mr. Boros and Dr. Hsuan, whereas Drs. Stivala and Chudnovsky disagreed. As noted in this report, there are a few areas where it may be beneficial for further basic research. Based on current science, standard test methods, and known failure mechanisms for PE, the FDOT

interim specification (Section 948) is adequate to qualify corrugated PE pipe for Class II service in the State of Florida.

3.2. Account of Research Synthesis and Direction

TRG members Drs. Stivala and Chudnovsky have made contributions to the report which fit most appropriately into definition of research synthesis and direction. This view of their contributions is taken due to the nature of their perspectives. While no documented failures of HDPE CP by the mechanisms they proposed have been observed on HDPE corrugated pipe, future work may be considered.

Dr. Stivala suggests that the level of surface oxidation be determined to ensure that no significant oxidation occurs at a depth of 5 mils (127 micrometers) [6]. For example, FTIR can determine surface oxidation to a depth of 0.5 to 4 micrometers and when combined with microtome sampling, depth information can be obtained. This determination can be made as a function of exposure environment with high oxygen concentration for direct oxidation of AO or liquid water for hydrolysis of AO. Tests should be made to determine the onset of oxidation versus AO depletion.

Dr. Chudnovsky concurs with determining details of surface oxidation to the extent that surface oxidation greater that 5 mils (127 micrometers) can result in brittle surfaces capable of forming microcracks which can initiate larger scale damage. References were provided that indicated FTIR was adequate to determine the presence of potentially brittle surfaces and included alternate techniques capable of providing additional information on brittle layers [7,8]. Further, multimode stresses should be considered in more detail to determine the relevance of such stresses. Current practice can not accommodate analyses of multimode stresses for corrugated pipe.

4. CONCLUSIONS

At the conclusion of three intensive meetings, it was clear that the TRG was divided and would not reach uniform conclusions. As in the case of most specifications, the TRG agreed that more work could be performed to improve the specification. A majority believe that interim

specification is adequate. Two members took the opposing view. The facilitator believes that the boundary between the current (interim) specification and future work is the key to consensus on these issues. The TRG members confirming the adequacy of the specification are those with the greatest experience specifically with HDPE CP. They have confidence that the material is improved over previously produced material and the installation requirements are better understood. Those changes coupled to the current specifications and on-going developments lead to the finding that the current FDOT specification for HDPE corrugated pipes (Section 948) is adequate. The dissenting TRG members have taken a different view and instead expound upon theoretical failure mechanisms of HDPE CP. Their views serve an appropriate purpose for identification of addition work intended to improve the current specification and materials application.

4.1 Consideration of Possible Technical Flaws

The current FDOT specification for HDPE corrugated pipes (Section 948) appears to adequately cover the known failure concerns – SCR of PE resin, SCR of corrugated PE pipe junction and profile, oxidation resistance including potential for extraction of AO in a water environment. As noted in this report, there are a few areas where it may be beneficial for further basic (as opposed to applied) research. Based on current science, standard test methods, and known failure mechanisms for HDPE CP, this interim specification is adequate to qualify corrugated PE pipe for Class II service in the State of Florida.

4.2 Consideration of Research Synthesis and Direction

- Tests could be developed to determine the onset of HDPE oxidation versus antioxidant depletion and which is more relative to the oxidation mechanisms of HDPE corrugated pipe.
- Alternate techniques capable of providing additional information on brittle surface layers could be considered for development, and the correlation of such surface oxidation phenomenon to the design life prediction for HDPE corrugated pipe.
- Identify the primary stress mode in the corrugated pipe walls under compression loading. If other than tensile mode (K_I) is presented, multimode stresses should then–be considered in more detail to determine the relevance of such stresses.

4.3. Recommendations

The following is a list of topics that are recommended for consideration in future research and/or specification development:

- Studies could be made of antioxidant depletion under different oxidation conditions such as water only, dissolved oxygen in water and dry air, each with and without stress on HDPE specimen.
- Tests could be developed to determine the onset of HDPE oxidation versus antioxidant depletion and which is more relative to the oxidation mechanisms of HDPE corrugated pipe. Determination of service lifetime may be more practical.
- Alternate techniques capable of providing additional information on brittle surface layers could be considered for development, and the correlation of such surface oxidation phenomenon to the design life prediction for HDPE corrugated pipe. Specifically, the issue is determination of surface brittleness relative to the tensile strength of the pipe.
- Identify the primary stress mode in the corrugated pipe walls under compression loading. If other than tensile mode (KI) is presented, multimode stresses should then be considered in more detail to determine the relevance of such stresses. This research is considered complex and would serve to validate the current view that tensile stress is dominant.

5. REFERENCES

[1] Hsuan, Y.G., and McGrath, T.J., Protocol for Predicting Long-term Service of Corrugated High Density Polyethylene Pipes, Final Report to Florida Department of Transportation, July 29, 2005.

[2] The National Academies, “Our Study Process – Ensuring Independent, Objective Advice,” National Academies Press, Washington, DC, 20001, http://www.nap.edu.

[3] Specification 948: Miscellaneous Type of Pipe - Corrugated Polyethylene Pipe (Rev 2-8-05) (FA 3-3-05) (7-05), Florida Department of Transportation State Materials Office, located at 5007 N.E. 39th Avenue, Gainesville, FL 32609

[4] Hsuan, Y.G, and McGrath, T.J., National Cooperative Highway Research Program, Report 429, HDPE Pipe: Recommended Material Specifications and Design Requirements, Transportation Research Board, National Research Council, National Academy Press, Washington, D.C. 1999

[5] National Transportation Product Evaluation Program, http://data.ntpep.org/home/index.asp (accessed February 20, 2007).

[6] Broutman, L., Surface Embrittlement of Polyethylene Pipe Grade Resins, GRI-81-0030, Final Report, Gas Research Institute, Chicago, IL, 1981.

[7] Gedde, U.W., Terselius, B., and Jansson, J.-F., A New Method For The Detection Of Thermal Oxidation In Polyethylene Pipes, Polymer Testing, Volume 2, Issue 2, April-June (1981): 85-101.

[8] Gedde, U.W., Terselius, B., and Jansson, J.-F., A Survey Of Methods For The Detection Of Thermal Oxidation In High-Density Polyethylene Pipes, Polymer Testing, Volume 2, Issue 3, July-September (1981): 211-222

6. Appendices

6.1 Appendix A

FDOT: State Materials Office Corrosion and Concrete Durability Laboratory - HDPE Protocols

Document Title	File Type-Size
Protocol for Predicting Long-term Service of Corrugated High Density Polyethylene Pipes (Final Report)	PDF - 1051.4KB
Florida Method of Test for Determining Slow Crack Growth Resistance of HDPE Corrugated Pipes (FM 5-572)	PDF - 71.84KB
Florida Method of Test for Predicting the Crack Free Service Life of HDPE Corrugated Pipes (FM 5-573)	PDF - 26.18KB
Florida Method of Test for Predicting the Oxidation Resistance of HDPE Corrugated Pipes (FM 5-574)	PDF - 34.41KB
Specification 948: Miscellaneous Type of Pipe - Corrugated Polyethylene Pipe (Rev 2-8-05) (FA 3-3-05) (7-05)	PDF - 76.9KB
Technical Comments and Responses	-
Contech, Inc.	PDF - 35.75KB
Contech, Inc./Gorban	PDF - 69.46KB
Columbia Consultants (Chudnovski, Uy)	PDF - 77.81KB
FDOT Responses to Comments from Chudnovsky and Uy	PDF - 17.69KB
Hardie Pipe	PDF - 54.92KB
Beakley	PDF - 131.27KB

6.2 Appendix B

Background Information on Section 3.1

Material from Part II [B1]

The material specification for corrugated HDPE pipes used in transportation applications is based on AASHTO M294 “Standard Specification for Corrugated Polyethylene Pipes”. In the year 2002, the specification adopted the NCLS test which is now ASTM F 2136 “Standard Test Method for Notched Constant Ligament Stress (NCLS) Test to Determine Slow Crack Growth Resistance of HDPE Resins or HDPE Corrugated Pipe”. The modification enhances the SCR of HDPE resins used for corrugated pipes. The NCLS test is a constant stress test in which stress relaxation does not develop, thereby presenting a greater challenge to SCR of the test specimens in comparison to constant strain test (i.e., ASTM D 1693) which was required by the specification until 1999. The minimum cell classes defined in AASHTO M294 are shown in Table 2.2 together with the specified property ranges within each of the cell classes.

In the current M294 specification, environmental stress crack resistance (ESCR) and hydrostatic design basis (HDB) tests, are not specified; instead the NCLS test is required in the specification. Resin samples are made from plaques according to ASTM D 4703. The conditions of the NCLS test are defined to be at 50°C in a 10% Igepal® solution under an applied stress of 600 psi. The average failure time of five test specimens must be greater than 24 hours and no single specimen failure shall be less than 17 hours.

Table 2.2 – Cell Class Properties for Corrugated HDPE Pipes [B1]

Properties	Cell Class	Value
Density	3	< 0.945 – 0.955 g/cc
Melt Index	3	< 0.4 – 0.15 g/10 min
Flexural modulus	5	110,000 to <160,000 psi
Tensile Strength	4	3,000 - <3,500 psi
ESCR*	0	Unspecified
HDB+	0	Unspecified
UV stabilizer	C	2% minimum carbon black

* ESCR – Environmental stress crack resistance

+ HDB – Hydrostatic design basis.

For finished pipe test, the M-294 specification retained the 90^{o} pipe bending test for the evaluation of SCR on the finished pipes. This bending test is based on the same stress condition as ASTM D 1693. The pipe section is under a constant strain condition, thereby allowing stress relaxation to take place during the testing. This finished pipe test does not appropriately challenge the SCR properties of the pipe. Furthermore, the test is impractical for large diameter pipes. Most important, however, the test does not challenge the specific locations that are sensitive to stress cracking, such as junctions, longitudinal profiles, and processing defects. Alternative SCR tests on the finished pipe were developed in this study and are incorporated into the test protocol for predicting long-term stress crack resistance of the finished pipe. The new SCR tests are applied to both short and long-term evaluations. The short-term tests are used for quality assurance and quality control (QA/QC) purposes to confirm the properties of pipes that have previously demonstrated 100-year crack free performance by manufacturers or users. The long-term evaluation employed tests that are performed under a range of different environmental conditions for long-term prediction purpose.

Background

As shown in Table 2.1, the current AASHTO M294 specification does not require the evaluation of antioxidants in HDPE corrugated pipes except for the cell class defined in ASTM D 3350. In the NCHRP Report 429, a large variation was found in the antioxidants of 14 evaluated commercially new pipes. The data is as shown in Fig. 2.18. The amount of antioxidants in the pipe is expressed by the OIT value which ranges from few minutes to over 40 minutes. This large scatter in the data indicates that there is little consistency in the manufacture of different HDPE corrugated pipes and is a major issue of concern.

The function of antioxidants in the corrugated pipe is to protect the polyethylene resin from oxidative degradation. The mechanical properties (including SCR) can only be preserved by properly formulated antioxidants. Thus, the lifetime of antioxidants plays an essential role in the overall service life of the pipe.

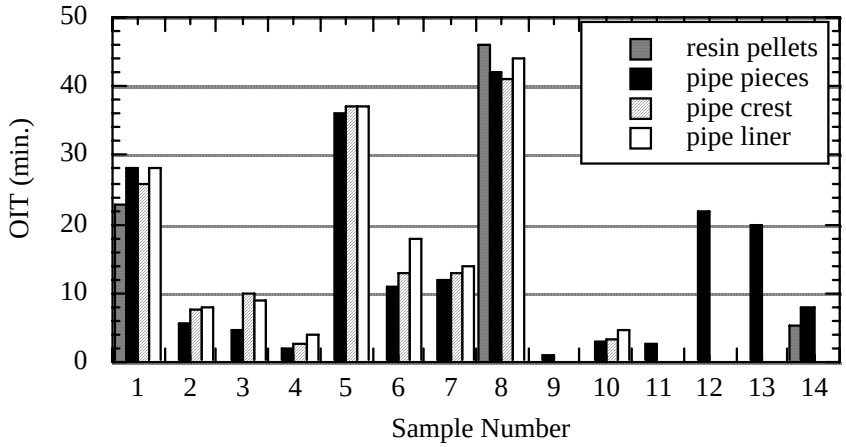


Fig. 2.18 - OIT data of fourteen commercially new pipe samples [B1]

The overall oxidation mechanisms can be divided into three conceptual stages, as shown in Fig. 2.19. These mechanisms are well established in the HDPE pressured pipe industries.

- Stage A represents time to consume all of the antioxidants in the pipe. The duration of this stage depends on both type and amount of antioxidants as well as the site ambient environment or simulated laboratory testing conditions.
- Stage B is the induction time which is the inherent property of the unstabilized polymer. In this stage the polymer reacts with oxygen and generates free radicals and hydroperoxide (ROOH), as expressed in Eqs. 2.5 and 2.6. The duration of this stage is governed by the concentration of hydroperoxide.

$$RH \xrightarrow{Energy} R\bullet + H\bullet \qquad \text{Eq. 2.5}$$

$$R\bullet + O_2 \rightarrow ROOH + R\bullet \qquad \text{Eq. 2.6}$$

- Stage C is the autocatalytic stage of the oxidation in which the formation of free radicals accelerates due to decomposition of ROOH, as indicated in Eqs. 2.7 to 2.9. The onset of the Stage C is when the hydroperoxide in the polymer increases to a critical concentration. The series of free radical reactions that take place in Stage C result in breaking polymer chains which leads to degradation in mechanical properties of the materials.

$$ROOH \xrightarrow{Energy} RO\bullet + OH\bullet \qquad \text{Eq. 2.7}$$

$$RO\bullet + RH \rightarrow ROH + R\bullet \qquad \text{Eq. 2.8}$$

$$OH\bullet + RH \rightarrow H_2O + R\bullet \qquad \text{Eq. 2.9}$$

Note: In Eqs 2.5 to 2.9, *RH* represents the polymer chain and compounds with the symbol (•) are free radicals.

Gedde's group has published a series of papers on the oxidation of HDPE hot water pressure pipes. Their findings are summarized in a review paper [B3]. In their study, the long-term performance of pressurized pipe was evaluated using method similar to ASTM D 2837. The test pipes were subjected to a series of internal pressures using either air or water, and were incubated in both water and air environments at temperatures from 60 to 105°C. The failure modes of the pipe are illustrated in Fig. 2.20. In Stage I, pipes fail by ductile mode. In Stage II, pipes fail in brittle mode via stress crack growth. In Stage III, the effect of mechanical loading becomes insignificant due to extremely low applied stresses, so that the pipes fail in brittle mode by oxidation degradation of the polymer. The transition between Stages II and III may sometimes be difficult to clearly define. Karlsson [B4] found that the formation of carbonyl groups which resulted from the oxidation degradation of polyethylene took place much earlier than the onset of Stage III. However, due to the low applied stress, it took a longer time for the pipe to fail than at a high applied stress.

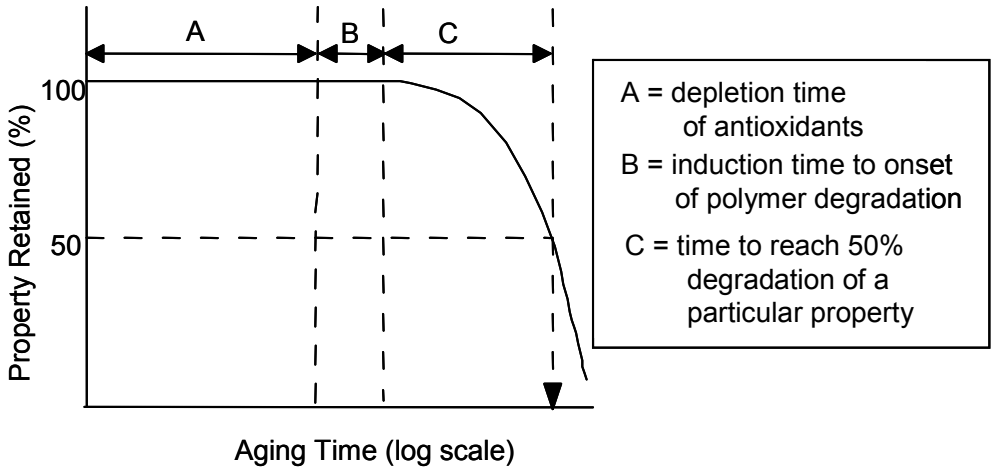


Fig. 2.19 – Three conceptual oxidation stages of HDPE [B1]

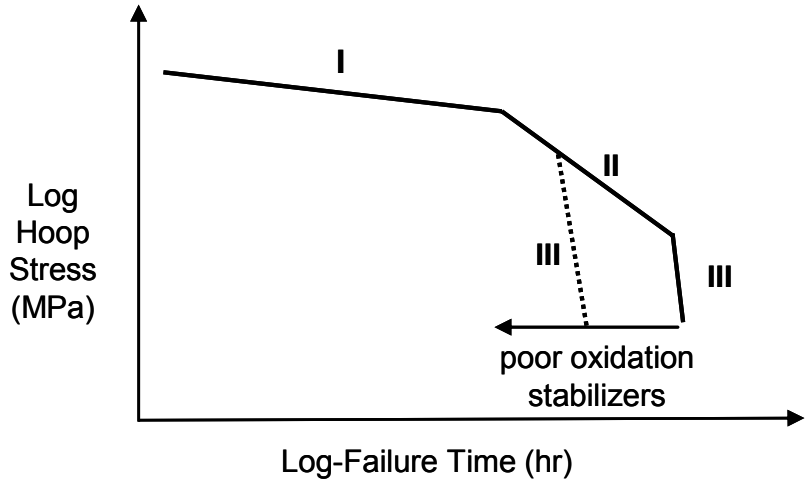


Fig. 2.20 – Three potential failure stages in pressure testing of smooth wall pipes [B1]

By comparing Figs. 2.19 and 2.20, the onset of the Stage III is within Stage C, while the exact position is dependent on the applied stress. Importantly, the onset of the Stage III must be well beyond the design life of the application under consideration. Gedde's data show that for gas pipe with appropriate antioxidants and good stress crack resistance properties, the onset of Stage III can be predicted to 1000 years at 20°C in water and/or air environments. However, without antioxidants, the onset of Stage III shortens to 11 years [B5]. Janson [B6] extrapolated the onset of Stage III using test data that were presented by Gaube's group [B7] and predicted 500 years at 20°C; however, the types of antioxidants in the tested pipes were not presented.

REFERENCES FOR APPENDIX B

[B1] Hsuan, Y.G., and McGrath, T.J., Protocol for Predicting Long-term Service of Corrugated High Density Polyethylene Pipes, Final Report to Florida Department of Transportation, July 29, 2005.

[B2] Hsuan, Y.G., and Koerner, R.M., "Antioxidant Depletion Lifetime in High Density Polyethylene Geomembranes", Journal of Geotechnical and Geo-environmental Engineering, ASCE, Vol. 124 (1999), No. 6, pp. 532-541.

[B3] Gedde, U.W., Viebke, J., Leijstrom, H. and Ifwarson, M., "Long-Term Properties of Hot-Water Polyolefin Pipes – A Review", Polymer Engineering and Science, Vol. 34 (1994), No. 24, pp.1773-1787.

[B4] Karlsson, K.. Smith, G.D., and Gedde, U.W., “Molecular Structure, Morphology, and Antioxidant Consumption in Medium Density Polyethylene Pipes in Hot-Water Applications”, Polymer Engineering and Science, Vol. 32 (1992), No. 10, pp. 649-657.

[B5] Viebke, J., Elble, E., Ifwarson, M., and Gedde, U.W., “Degradation of Unstabilized Medium-Density Polyethylene Pipes in Hot-Water Applications”, Polymer Engineering and Science, Vol. 34 (1994), No. 17, pp. 1354-1361.

[B6] Janson, L-E., Plastics Pipes for Water Supply and Sewage Disposal, Published by Borealis, (1995), 290 pgs.

[B7] Gaube, E., Gebler, H., Muller, W. and Gondro, C., “Zeitstandfestigkeit und Alterung von Rohren aus HDPE”, Kunststoffe 75 (1985), pp. 412-415.

6.3 Appendix C – COMMENTS BY TRG MEMBERS

Comments are numbered consecutively for reference.

6.3.1 Comments by C. Ormsby – Response by R. Granata

C. Ormsby	R. Granata
From: Ormsby, Clay [Clay.Ormsby@fhwa.dot.gov] Sent: Friday, March 16, 2007 8:16 AM To: Richard Granata Subject: RE: Shared comments	
1. I have reviewed all of the comments which you provided. I feel that conclusions of the report are still valid and do not need to be qualified.	Concur.
2. I agree with Steve that you did a good job in putting the report together.	
3. I hope that the report will be processed expeditiously.	No response required.

6.3.2 Comments by S. Boros – Response by R. Granata

S. Boros	R. Granata
March 17, 2007 Dr. Rich Granata Florida Atlantic University Dania Beach, FL Re: Additional Response to Other TRG Member Comments	
Rich,	
1. I am completely opposed to any further revision of this report. The comments from Drs. Stivala and Chudnovsky were thoroughly discussed at the three meetings of the TRG. I believe they are confusing the non-acceptance of their arguments with the TRG not listening. If all we are going to do is have everyone give their opinion, then there is no need for meetings.	Concur.
2. Drs. Chudnovsky and Stivala proposed many questions to which they believe inadequate responses given. Responses were given and the issues were discussed at length. It became obvious there was no answer that would suffice. That is why it is appropriate to move these questions into the "additional research" area.	Concur.
3. While there were questions proposed as to the adequacy of the specification, there were no proposals for	Concur. Re: “no proposals”. Some

S. Boros	R. Granata
improvements to the specification, only further statements that the science used was "questionable" and that further research is needed to develop the science needed to write such a specification. As I stated previously, this specification is very rigorous. Standards and Specifications must be based on the most currently accepted technology available. There is certainly always more knowledge to be gained from an academic standpoint, but from a practical point of view the most current science available must be used to establish appropriate protocols for construction materials. ASTM standard methods are commonly used for these purposes. Dr. Hsuan spent nearly three years reviewing the current science to develop the FDOT specification.	suggestions were made that required additional research to implement or that may have been alternatives to portions of the specification, but provided no clear and immediate benefit.
4. A good example is the questioning of Dr. McGrath, whose findings were questioned as adequate. Dr. McGrath is a noted and respected structural engineer who used currently accepted practices to evaluate the stresses on a corrugated HDPE pipe in service. If Dr. Chudnovsky has some questions about these practices, then some future research by him may be of interest for future application.	Concur.
5. It is stated clearly in the report that OIT is not intended to be used to establish service life. The specification utilizes a multi-faceted approach to establish oxidation degradation. It was never the intent to establish the onset of surface oxidation, only to establish when the oxidation progressed to the point where physical properties would be affected. Dr. Hsuan performed physical property testing which is the accepted practice to evaluate when oxidation has progressed to the point that the material properties are adversely affected. Testing showed that after incubation, oxidation did not progress to the point where physical properties would be adversely affected. In addition, Dr. Cheng, a Research Scientist with Exxon Mobile, performed FTIR testing on incubated samples to look for the presence of oxidation on microtome samples from the specimens. None was found. These tests alone do not provide for the 100 year forecast, but both of these tests together support that the multi-faceted approach taken by the FDOT specification is appropriate.	Concur.
6. The idea that PB failures, in a artificially chlorinated environment mostly at elevated temperatures as high as 140°F, are related to corrugated HDPE pipe in drainage applications in the State of Florida is simply not true. This is a different material in a completely different environment. Free chlorine is not present in storm water drainage systems and free chlorine is a known strong oxidizer of polyolefin materials. There is no known correlation that supports similar oxidation rates will take place in the proposed FDOT application. Dr. Chudnovsky's	Concur.

S. Boros	R. Granata
own paper showed that PE was an order of magnitude more resistant to oxidation than PB. These arguments are not relative (sic) to this specification. Also, these PB materials were NEVER subjected to the same rigorous test requirements as being proposed by this specification.	"relevant"
7. Rich, these comments are simply a repeat of the three meetings held by the task group. As I said in my previous letter, the questions raised were just that – questions. I do not agree that answers were not given, but did agree to put these into the area of "additional research". The report is accurate as written and no further revision is necessary.	Concur.
Respectfully, Stephen Boros Technical Director - Plastics Pipe Institute 105 Decker Court, Suite 825, Irving, TX 75062 P: 469-499-1044 F: 469-499-1063	

6.3.3 Comments by G. Hsuan – Response by R. Granata

G. Hsuan	R. Granata
On Final Report.	
8. It is my opinion that the background in selecting of the TRG members should be further clarified. In page 1, "*TRG members were selected strictly on the basis of their technical capabilities as representing the contrasting views on the topic*" is not sufficient to explain the complexity of the situation and the actual selection process. The members were recommended by different parties, including FDOT, ACPA, and CPPA.	Concur and done.
9. May be we can rephrase the statement "*the findings of the TRG were that the current specifications were adequate*" by the "*The current specifications have incorporated the state-of-practice test methods to evaluate the long-term properties of corrugated HDPE pipes. However, different opinions were expressed by members of TRG regarding the extrapolation methods and the sensitivity of the test methods*."	Done with some additional rephrasing.
10. Emphasizing the interim specification should adopt state-of-practice which the industry (or close related industries) has experience and test data on those tests.	Done with some rephrasing.
11. I would prefer to reference "Drexel Research Report" as "Drexel Study". The project was not a research project and was not intend to be a research project. By referring it as research report, it would mislead the readers that a thoroughly planned research was carried out. We performed limited tests to verify the proposed	Done.

G. Hsuan	R. Granata
specification. 12. There are few small changes and one question on the report. See the attached tracked document.	Minor changes have been made.
Response to Comments from Dr. Stivala and Dr. Chudnovsky based on the Draft Final Report	
13. The comments from Dr. Stivala have largely been incorporated into the Draft Final Report and have been addressed. Maybe by quoting his question in the report it would clarify his points of view.	Adequate portions of Dr. Stivala's comments have been incorporated in the main body of the report. Further details are contained within his comments. Boros and Ormsby oppose changes to report.
14. Although I have not read the GRI report by Dr. Broutman on oxidation of pressure pipes, his study probably on gas pipes that were subjected to high internal pressure and elevated temperatures to accelerate the oxidation. What would be the predicted performance of the aged pipes at ambient condition? Unless we have a copy of the report to identify the test conditions, it would be difficult to understand the impact of the 0.005 inch of the brittle surface on the service life of the pipe at ambient condition.	Although I had asked that copies of all pertinent references be supplied to me, I did not receive the report from Drs. Stivala or Chudnovsky. I recently obtained a copy of GRI-81-0030. The report did not address pipes but it did provide information supporting tensile tests for determining if brittle material is present in PE due to oxidation.
15. In the Draft Report, it has already stated that surface oxidation is likely to take place in any plastic product after certain duration of service. However, the key issue is whether the surface oxidation will yield a mechanical degradation. In the Drexel Report, tensile test data indicated no mechanical degradation in water immersed samples after 300-day at 85°C. In addition, published papers have indicated that the tensile elongation does not decrease as long as antioxidant presence in the polymer. Although FTIR was not carried out on the aged samples, it should not ignore the physical (MI test) and mechanical test data. The surface cracking test proposed by Dr. Chudnovsky would have direct impact on the breaking elongation of the aged specimen. The linkage between microstructure and macrostructure must be directly correlated. For engineering applications, the mechanical performance should be the key measuring parameter in the long-term performance of the pipes.	The GRI-81-0030 Report supports results of the Drexel Report in that the absence of mechanical degradation indicates no significant effect of surface oxidation. FTIR or other testing for surface oxidation is not necessary.
16. This Draft Report also pointed out that the interim specification should not require the manufacturer to determine the onset of the oxidation, which could be beyond 100 years. Furthermore, it was also not the scope	Concur. These points were discussed in detail during the TRG meetings.

G. Hsuan	R. Granata
of the Drexel study. That was the reason why the incubation of the test sample terminated after 300-day which was predicted to have oxidation resistance beyond 100 years. Dr. Stivala's opinions would only valid if the scope of the Drexel study was to determine the lifetime of the corrugated HDPE pipes.	
17. 2. Regarding the five points stated in Dr. Stivala's comments:	
18. i) No characterization of thin surface layers of HDPE CP	See "Responses" below.
19. ii) No correlation of the extent of oxidation with depletion of AO	
20. iii) OIT is fine for assessing levels of AO depletion, but not valid in predicting long-term service life.	
21. iv) Conditions that have been chosen for resting do not provide sufficient acceleration of oxidation	
22. v) The extrapolation from 300 days tests to 100 years is not realistic.	
23. Responses	
24. i) Although surface oxidation layer was not analyzed on all of the aged samples, FTIR did perform on the surface of an aged samples by Exxon-Mobil. This has been explained in the report.	Concur. Follow-on work may be considered as research synthesis and direction.
25. ii) Again, the report (Page 9) referenced the test data that were presented in Drexel report. The MI (melt index), tensile properties and AO depletion were evaluated on the 85°C aged samples throughout 300 days.	Concur. Tensile properties indicated no embrittlement or surface oxidation at end of test.
26. iii) The report (page 8) clearly stated that OIT was not used for predicting the service life, but a tool to monitoring the same AO amount in the pipe.	Concur. To monitor adequate amount of AO remaining in pipe.
27. iv) The incubation condition by fully immersing test samples in the water was extremely challenge to the AO. For polyolefins, the lifetime of AO probably contributes more than half of the total lifetime of the product. Therefore, we should focus on the quality of AOs used in the corrugated HDPE rather than the onset of oxidation in the polymer.	Concur. The immersion extracts AO faster than it could be consumed by oxidation.
28. I do agree that the onset of oxidation in water would be slower than in air due to the limited oxygen presence in the water. However, the focus of the study was on the depletion AO. If we are going to determine the onset of oxidation, then an alternative test environment should be considered.	The depletion of AO must not be confused with the onset of oxidation. Water can rapidly extract AO compared to air. Depletion of AO is an important factor.
29. I don't see the control humidity has anything to do with oxidation.	Humidity effects may be considered in future work.
30. v) I do not see the problem of extrapolation as long as Arrhenius model is valid from 85°C to 23°C.	Concur.
31. 3. For the comments from Dr. Chudnovsky, he referred many times to his report which is not included the	Dr. Chudnovsky's report is included herein as Appendix D.

G. Hsuan	R. Granata
document package. He stated that he questions the scientific basis of testing and data processing proposed in the specification of HDPE CP 100-year service life. On the second page of his comments, he indicated that he has formulated specific questions during our meetings. However, I have difficulty to pinpoint his questions. He agreed the adaptation of RPM to extrapolate the stress cracking data.	The report summarizes the same positions discussed in the TRG meetings. He has challenged most every component of the specifications starting from an academic perspective and a desire to know and understand all possible factors. This approach prevents development of an adequate specification unless all information is known about the materials, processes and eventualities for the application. Such an approach impedes practical engineering progress. It was apparent that Dr. Chudnovsky would not yield to practical solutions for this engineering challenge. The TRG was to determine what known issues should be used to produce a rigorous specification. Dr. Chunovsky's contributions were better suited to research synthesis and direction.
32. 4. He stated in his comments that NCHRP Report 429 is lack of information. What information did he refer? The report does not address 100-years prediction. The NCHRP report identified the index test to evaluate stress crack resistance of the pipe resin as clearly stated in this Draft Report.	Dr. Chudnovsky is referring to both the NCHRP report and the Draft TRG report. The example regarding Dr. McGrath's limitations for finite element analyses emphasizes Dr. Chudnovsky's focus on detail rather than a practical engineering specification. Again, this focus is appropriate for research synthesis and direction.
33. 5. For the stress mode issue, it is not the state-of-practice to evaluate shear stress in all types of corrugated pipes. Until that becomes a common engineering design problem, the specification should not consider such property.	Concur.
34. 6. For the oxidation resistance issues, I have responded earlier in this document.	Concur.

6.3.4 Comments by S. Stivala – Response by R. Granata

S. Stivala	R. Granata
Final Report Draft (3/13/07)	
From: SALVATORE STIVALA	
35. I refer you to the Abstract, 1st paragraph. i.e., "... the findings of the TRG were that the current specifications were adequate,..." I completely disagree. My reasons are clearly discussed in my earlier report to you. In this regard I request that my report be appended to the Final Report*.	Done. The findings are not unanimous. There was no intent to claim the consensus on all points.
36. Further, the Abstract should spell out the names of those who agree and those who disagree with the adequacy of the specification. In this context you should also append the reports to you of other TRG members.	Done.
37. It is important that the officers of FDOT, who are concerned with the proposed specification, have a clear understanding of the basis of the TRG members opposing views, which are not adequately reflected in the draft of Final Report.	Concur. However, it is my opinion that FDOT has documented a clear understanding of the opposing views.
38. Finally, I strongly recommend that the TRG meet with FDOT representatives who may have questions, especially those who would have read the Final Report.	Recommendation noted. The proposed meeting does not appear warranted at this time.
39. P.S.: The PB discussion In the Final Report — Draft (page 10) omits the fundamental observation that oxidation brings about the formation of microcracks within the surface layer, which is the initiation of crack growth in the pipe. HDPE is a polyolefin as is PB. Therefore, this mechanism is also present in HDPE. Three members of TRG, who found the current specification adequate, failed to recognize the importance of this mechanism of polyolefin premature failure, as it has been observed in PB.	PB has tertiary hydrogen which is more susceptible to oxidation than the secondary or primary hydrogen in PE. The rate of oxidation is lower for PE versus PB. The oxidizing agent, chlorine, is not present in this application. The service temperature is 23°C for PE versus 140°C for the PB application. There is disagreement on the importance of this mechanism.
40. *CRITIQUE ON THE LONG-TERM SERVICE OF HIGH DENSITY POLYETHYLENE CORRUGATED PIPES by SALVATORE STIVALA, Ph.D.	
41. 1. INTRODUCTORY BACKGROUND	
42. This critique is based on my review of the final report, by Drs. Y. Grace Hsuan and Timothy McGrath, entitled Protocol for Predicting Long Term Service of Corrugated High Density Polyethylene Pipe, July 24, 2005, herein referred to as the Hsuan Report, and on my participation in discussions at four meetings, which were chaired by Prof. Granata, in the spring of 2006 in Florida.	Three meetings were held.
43. It has been my contention that to predict long-term service life of any product the criterion of failure must be established. Though this was discussed at the Florida	Discussion of criteria included all items closely or remotely related to potential failure. The only items

S. Stivala	R. Granata
meetings, this topic was not resolved, to the best of my recollection.	that were not resolved were those championed by S. Stivala and A. Chudnovsky.
44. The oxidation of polyolefins, such as polyethylene, is a surface phenomenon, the surface of which can greatly effect, or influence, the mechanical properties of the bulk polymer. As in any polyolefin, such as the high-density polyethylene (HDPE) in corrugated pipes (CP), the oxidized surface is a small percentage (about 5%, i.e., about 5-10 mils in depth) of the entire wall of the corrugated pipe. The physical, morphology, and chemical composition of the polymer surface are entirely different from the unoxidized bulk. For example, the unoxidized bulk polyolefin is a semi-crystalline, high molecular weight (MW), high density ductile polyhydrocarbon (hydrogen and carbon) whereas the oxidized surface is a brittle lower MW oxygen-containing polyethylene, (carbon, hydrogen, oxygen), and of higher density and crystallinity. The effect of the thin oxidized polyethylene layer, as stated earlier, will influence the properties of the unoxidized bulk. This effect can be noted from the work of Dr. L. Broutman, performed for the Gas Research Institute.	First sentence – Concur. Second sentence – No, HDPE CP for drainage pipe service has been documented to have failed by oxidation of approximately 5% of its wall thickness. The statement is misleading. It misleads by associating HDPE CP with tests performed for a different application, gas transmission pipe, using material that was fabricated differently from HDPE CP and intentionally oxidized with UV to demonstrate a possible failure mechanism. The same work cited (Broutman) showed that brittle failure occurs without oxidation.
45. Dr. Broutman showed and reported that in polyethylene, a brittle surface, having a depth of approximately 0.005 inch can bring about a brittle fracture in an otherwise ductile polymer. (Dr. Lawrence T. Broutman, Surface Embrittlement of Polyethylene Pipe Grade Resins, final report September 1979 to September 1981, Gas Research Institute.	This information is not important unless oxidation to sufficient depth can be demonstrated for HDPE CP.
46. An oxidized HDPE surface can be detected by Fourier Transform Infrared (FT-IR) spectroscopy, by observing some of the oxidation products, such as the carbonyl compounds, e.g., acids, aldehydes, esters, ketones that exhibit absorbance values of 1705, 1730, 1740, 1715 per centimeters, respectively.	The penetrating depth of ATR FTIR measurement is approximately 0.0002 inches (0.004 cm) which allows good surface sensitivity for the measurement. Observation of these compounds by ATR FTIR can over estimate their significance.
47. Oxidation induction time, OIT is a valuable, and an effective tool for studying anti-oxidants (agents that slow the rate of oxidation). However, OIT is not applicable in predicting long-term service life. OIT is useful for following the depletion of anti-oxidants under various exposure conditions over time.	The negative comment is misleading. OIT is NOT being used to predict long-term service life. It is being used precisely for studying anti-oxidants, observing depletion under specific conditions over time.
48. Accelerated oxidation tests in air and in water of 85°C, were performed on samples of HDPE-CP (the Hsuan Report). Depletion of anti-oxidants, assessed	The OIT measurements were intended to establish continued presence of antioxidants and did

S. Stivala	R. Granata
from OIT measurements, was followed over time. It was shown that the mechanical properties (strength, elongation) and melt index do not change in air and in water, for periods up to 300 days, while the depletion decreases, approaching constancy at about 10-20% depletion of AO in water. Measurements of OIT were made from samples taken across the entire wall thickness rather than the surface, e.g. a layer of approximately 3-5 mils.	so. The absence of mechanical properties changes establishes the absence of significant surface property effects (L. Broutman, GRI-81-0030). Again, this is misleading – There is no requirement that the OIT measurement be made on surface layers.
49. Based on depletion of AO in water, as well as in air, at 85°C and from the application of the Arrhenius Equation, it is predicted that HDPE CP will survive at least 100 years, (the Hsuan Report).	These were criteria selected for practical predictions.
50. II CRITIQUE	
51. The act of AO depletion of itself is not oxidation, but rather, the loss of AO due to: (a) diffusion and/or leaching out (b) the AO working as intended, and (c) interaction with other agents, e.g., water (hydrolysis). The oxidation starts before complete depletion of AO. The extent of oxidation can be assessed from obtaining the carbonyl index (CI) from FT-IR. No measurements of the oxidation were made on the HDPE CP samples that were tested in water and in air, both at 85°C, with the exception of one case that is addressed below.	Oxidation at the surface of HDPE CP is only an indicator if it correlates to changes in mechanical properties. The absence of mechanical properties changes establishes the absence of significant surface property effects (L. Broutman, GRI-81-0030). No need to measure surface oxidation is indicated.
52. Using samples (HDPE CP) cut from across the wall thickness, the contribution of any surface oxidized layer that may have occurred is diluted by the large amount of bulk polymer. Therefore, the oxidation may not be detected. The Hsuan Report mentions "concern has been raised regarding the oxidation status on the surface specimen". Thus, FTIR coupled with ATR (attenuated total reflectance) was used to detect oxidation on the surface of test specimens (thin films of 0.5 and 1.0 mm taken from 2.5 mm thickness wall). FTIR-ATR showed no oxidation after 2341 hours of exposure of these films in water at 85C. Thus, it was concluded, "that the issue of surface degradation does not appear to be a concern".	Ditto (comment immediately above).
53. The above was brought up at the panel meetings in Florida (spring of 2006) by me as well as Dr. Chudnovsky. Dr. Hsuan agreed that the time of exposure of the thin films was too short, as well as oxygen concentration in the water was very low.	This test was performed as a “spot check” and is not critical to the adequacy of the interim specification. There was no significant oxidation observed. Regarding the oxygen concentration in 85°C, the concentration is actually much higher than was assumed during the meetings. It is approximately 2.7 ppm at 85°C.

S. Stivala	R. Granata
54. It has been established, from the literature that the layer of oxidized polyolefin surface can bring about a brittle failure in an otherwise ductile polymer. Though this was discussed a number of times at the panel meetings, members of the panel, with the exception of Drs. Stivala and Chudnovsky, decided that such a thin oxidized layer would not be a concern. It is my opinion that the surface of exposed samples of HDPE CP should have been characterized by FTIR, and optical microscopy to assess the effect of a thin oxidized layer, if not anything else, to at least rule out the effect of surface oxidation.	Disagree. Drs. Stivala and Chudnovsky intently focused on ANY oxidation being critical to the interim specification and gradually transitioning to quantifying 5% thickness being critical. A great deal of time was consumed arguing the importance of oxidation starting with a monolayer (1 nanometer) thickness up to 5% thickness (127 micrometers = 0.005 inches). The mechanical testing rules out the effect of surface oxidation.
55. The Hsuan Report falls short in "evaluating oxidation resistance" of HDPE CP. The factors that support my opinion can be summarized as follows:	Disagree. See next 5 comments.
56. No characterization of thin surface layers of HDPE CP.	Disagree – Not necessary. See comment 51.
57. No correlation of the extent of oxidation with depletion of AO.	Disagree – Not necessary. See comment 51.
58. OIT is fine for assessing levels of AO depletion, but not valid in predicting long-term service life.	Disagree – AO is not used for prediction; it is used for predicting that an effective amount of AO will be present after 100 years..
59. Conditions that have been chosen for testing do not provide sufficient acceleration of oxidation.	The conditions chosen were the closest to field conditions. Other conditions could be established with research synthesis and direction.
60. The extrapolation from 300 days tests to 100 years is not realistic.	Based upon Arrhenius theory and using conservative assumptions, the extrapolation appears useful and reasonable, even though it may not follow the guidelines of a specific extrapolation method.
61. Regarding item 4 (comment 59) among the factors cited above, the Hsuan Report correctly recognizes that in depletion studies "oxidation is suppressed due to the limited oxygen content in the water". It is known that rates of reaction (e.g. oxidation) increase with increasing temperature increase with concentration of a reactant (e.g., oxygen) and stress. The use of 100% oxygen instead of air (21% oxygen) plus temperatures above 85°C or the use of UV light at lower temperatures (below 85°C) would greatly accelerate the reaction. In the case of water containing very low concentration of oxygen, the rate of oxidation at 85°C would increase by adding to the water oxidizing agents, e.g. sodium hypochlorite. The	Disagree – Particularly with respect to foreign oxidizing agents such as hypochlorite or UV which may change the oxidation mechanism. The objective is not to accelerate oxidation of the HDPE CP. The objective is to accelerate the entire process which includes transport of the AO to the surface followed by reaction with the 21% oxygen in air. Research synthesis and direction is the appropriate venue for

S. Stivala	R. Granata
objective of acceleration is to bring about oxidation within a realistic short time compared to the projected lifetime of any product e.g. HDPE CP.	modification of the oxidizing conditions. In service, HDPE CP is not exposed to hypochlorite or UV radiation.
62. In the case of HDPE CP, water is transported through the bottom of the pipe, where loss of AO by diffusion occurs over time. The interior upper portion of the pipe is exposed to atmospheric oxygen of high concentration (21 %). Further, in most instances moisture is present in the upper interior part of the pipe where AO diffusion takes place. The presence of stress from the soil overhead may play an important role in accelerating oxidation. The Hsuan Report, in conducting accelerated testing of HDPE CP in air at 85°C should have been conducted in a chamber of controlled humidity.	Disagree. The impacts of stress and humidity on the rates of consumption of AO are not clear. Research synthesis and direction is the appropriate venue for these considerations.
63. In the ultimate analysis, an oxidized brittle surface layer approximately 0.005 inch or less can bring about a brittle fracture in an otherwise ductile polymer such as HDPE CP. The brittle surface resulting from the oxidation process leads to the formation of micro-cracks, which is the precursor to the crack growth.	Disagree. No instance of this type behavior in HDPE CP has been established. All mechanical tests on HDPE CP have shown no evidence of brittle fracture following the interim specification.
64. In conclusion, the oxidative resistance studies in the Hsuan Report failed to establish the scientific basis to support 100 years lifetime specification for HDPE CP.	Disagree. The methods employed are based upon accepted practices for accelerated aging and measurement of AO content. The interim specification was not formulated to disprove scientific speculation regarding a long lifetime. The interim specification provides a useful, practical and technically rigorous approach to moving forward with HDPE CP for long term applications.

6.3.5 Comments by A. Chudnovsky – Response by R. Granata

A. Chudnovsky	R. Granata
To: Richard D. Granata, Ph.D. Florida Atlantic University Department of Ocean Engineering 101 North Beach Road Dania Beach, FL 33004 Response to the Final Report - Draft.	
65. I have a number of disagreements with the Final	Agree.

A. Chudnovsky	R. Granata
Report - Draft. I hope the opinion expressed below will be taken into consideration in the Final Report.	
66. First of all, at the very beginning of the report, in the Abstract one reads: “Upon consideration of the challenges and broader concerns, the findings of the TRG were that the current specifications were adequate...” The Abstract is concluded as: “These proposed research areas have the potential to advance, in the sense of continuous improvement, the current specifications that represent current state-of-the-practice and are considered adequate.”	The majority of the TRG members hold this view. The facilitator has determined that there not likely be a consensus view obtainable through continued meetings and deliberations. The majority and minority views are contained in this report.
67. I found the specification INADEQUATE and express it during our meetings and in the written report I have sent to you. It is not a question of “additional areas… for research” or “continuous improvement” mentioned in the Abstract. I have questioned the scientific basis of testing and data processing proposed in the specification for HDPE CP 100-year Service Life, and did not obtain satisfactory answers. To my knowledge, Dr. Stivala has expressed similar opinion. Thus, the highlighted above parts of the Abstract “the findings of the TRG” are incorrect.	Your views have been noted. Scientific questions are welcomed as technology moves into new areas. The intent of the TRG was clearly stated to focus on identification of technical flaws in the interim specification and related test protocols. There was no agreement by the TRG that a flaw existed. Additional research and improvements could be considered.
68. Since it is a formal report, the list of TRG members should be formally presented with indication of position and affiliation. For example: 69. Salvatore Stivala, Rene Wasserman Professor Emeritus, Department of Chemistry and Chemical Biology, Stevens Institute of Technology.	Agree and done.
70. According to the statement on page 3 the TRG facilitator selected Mr. Stephen Boros to write an account for the committee’s report of the technical background and synopsis of the Section 948 specification based on the Drexel research. In Technical Review Mr. Boros, claims (page 5, Liner Tests): 71. “NCLS test has proven to be a valid index test to quantify the SCR property of pipe resins and pipes (NCHRP 429 and TRB paper). This interim specification adequately addresses the initial qualification of the corrugated PE pipe for a Class II or 100 year service life.” 72. I have formulated specific questions during our meetings and in my report to you “An Assessment of HDPE CP Specification for 100-years Service Life” regarding the test as well as extrapolation of the test results to 100-year service life. I did not (receive) answers to my questions.	This comment applicable to items 70, 71 & 72: I have attached your report-questions as Appendix 6.4. The issues and questions in your report have been addressed or taken into consideration during the TRG meetings and written exchanges.

A. Chudnovsky	R. Granata
73. Then, in the section Junction Tests starting from page 5 there are references to "extensive field failure investigation by Hsuan and McGrath (NCHRP report 429) [4]", and "report from Dr. McGrath [1]". I have posed specific questions during our meetings and in my report to you "An Assessment of HDPE CP Specification for 100-years Service Life" regarding lack of information in the report [4] for the test design as well as extrapolation of the test results to 100-year Service Life. I found a few indirect mentioning of the topics of my questions on page 6 and 7, but no satisfactory answers. For example, on page 7 one reads: 74. "Some discussion suggested that there may be stresses other than tensile stress in the corrugated PE pipe – such as shear stress or compressive stress. The finite element analysis performed by Dr. McGrath did not propose these other stresses and there is currently no accepted design methodology that includes shear or compressive stress as a major or limiting stress in corrugated PE pipe. However, this may be an area where further research could be considered". 75. The problem is that the answer to that question is the basis of the relevancy of the proposed test to the Service Life of HDPE CP. One should not claim the specification "adequate" without resolving this question. The absence of "other stresses" in Dr. McGrath analysis results from assumptions in the formulation of the problem.	This comment applicable to items 73, 74 and 75: In the comment sections above by S. Boros and G. Hsuan, this issue has been answered. Dr. McGrath has used state-of-the-practice methods and additional work is welcomed in research synthesis and direction.

A. Chudnovsky	R. Granata
76. The topic addressed in the section "Oxidation resistance of Pipes" has been heavily discussed during our meetings, as well as in the reports. 77. There is no point to repeat the arguments here. I am confident, The Oxidation Resistance of Pipes test (see page 4, Table 1, continued) is inadequate for detection of surface oxidation, which is a major mode of premature failure of plastic components intended for long service life (50 ~ 100 years). In response to this of argument, Mr. Boros writes: 78. "There was some question on whether using the OIT on the "bulk" pipe specimen is appropriate, or whether the OIT test should be performed on the surface of the specimen. Work performed by Dr. Cheng at Exxon Mobile examined pipe specimens that had been incubated according to the interim specification requirements. Using FTIR, the specimens were analyzed for the presence of the carbonyl functional group which is a well known and accepted indication of PE degradation. Testing of the 2.5 mm thick specimens was done on the surface, 0.5 mm removed, and 1 mm removed. None of the specimens displayed any presence of the carbonyl groups, indicating that degradation did not occur in these specimens – even on the surface. Further discussion on the FTIR method suggested that the specific technique used may not be sensitive enough to detect very small levels of oxidation on the surface of the exposed specimens". 79. Dr Stivala, the world recognized authority on polymers oxidation, gave the detailed explanation why Dr. Cheng's test does not resolve the controversy.	This comment is applicable to items 76, 77, 78 and 79: A. Chudnovsky raises the same point that S. Stivala raises and defers the answer to S. Stivala. I defer my response to that provided in the comment section of S. Stivala (above Comment #24). There may be some confusion on the part of A. Chudnovsky regarding the discussion of the FTIR method sensitivity for surface oxidation of HDPE. The method detects oxidation products in thicknesses much less that the 2 or 5 mils thickness cited for surface cracking. The point is moot because the mechanical testing performed within the interim specification eliminated the possibility that surface oxidation had occurred to an extent capable of causing cracking.
80. There is a statement in Conclusion (page 10) "The FDOT Interim Specification on Corrugated PE Pipe appears to adequately cover the known failure concerns – SCR of PE resin, SCR of corrugated PE pipe junction and profile, oxidation resistance including potential for extraction of AO in a water environment. " 81. I disagree with that statement. In my view, the FDOT Interim Specification on Corrugated PE Pipe appears inadequate for the reasons presented in my report to you "An Assessment of HDPE CP Specification for 100-years Service Life".	This comment applicable to items 80 & 81: In the comment sections above by S. Boros and G. Hsuan, the issues have been answered. Additional work is welcomed in research synthesis and direction.

A. Chudnovsky	R. Granata
82. In the section 3.2. Account for Research Synthesis and Direction, on page 11 one reads: "Chudnovsky concurs with determining details of surface oxidation to the extent that surface oxidation greater that 5 mils (127 micrometers) can result in brittle surfaces capable of forming microcracks which can initiate larger scale damage. References were provided that indicated FTIR was adequate to determine the presence of potentially brittle surfaces and included alternate techniques capable of providing additional information on brittle layers [7,8]." 83. Indeed, I have provided the references for alternative to FTIR, less expansive (sic) and sometimes more sensitive than FTIR techniques for surface oxidation detection. 84. In view of inadequacy of traditional OIT test for detection of surface oxidation, I am surprised that the above simple and practical suggestion has been ignored.	This comment is relative to items 82, 83 and 84: In the comment sections above by S. Boros and G. Hsuan, the issues have been answered. Again, detection of surface oxidation is accommodated in the interim specification as a mechanical test. The suggestion was not ignored. A specific surface oxidation test is not necessary. Additional work is welcomed in research synthesis and direction.
85. There is also a minor point made during our discussion that there are evidences of surface oxidation less than 5 mils, sometime ~2 mils leading to brittle fracture.	This point is included in the above comments and responses.
86. It is written in Conclusion (page 11) "A majority believe that interim specification is adequate". 87. It is well known that the scientific truth is established by scientific observations and facts, rather than by the majority. The "majority" in this case is 3 (including the author of specification) out of 5 members,.	This comment is relative to items 86 and 87: No suitable scientific observations or facts have been offered that could be practically applied to the interim specification, at least in the opinion of 3 of 5.
88. I would like to ask for clarification of the following statement in the section 4. Conclusion (page 11): "The TRG members confirming the adequacy of the specification are those with the greatest experience specifically with HDPE CP."	It is my intent that the statement emphases CP rather than HDPE or PB. It is an opinion based upon observations during the TRG activities. This statement may be subject to disagreement.

A. Chudnovsky	R. Granata
89. (Preceeding comment text and relevant text in the body of this report was deleted as directed by FDOT Project Manager 04/04/07) Both of us, Dr. Stivala and myself, have raised questions of scientific validity of testing programs intended for extrapolation of relatively short test data into 100-year service life, since we are well familiar with a dramatic failure of the same methodology applied to PB pipe 50-year Service Life prediction. That failure cost the industry billions of dollars.	The literal claim was made that <u>any</u> oxidation is unacceptable.
Alexander Chudnovsky Professor of Mechanics and Materials UIC Distinguished Professor Director of Fracture Mechanics & Materials Durability Lab. Department of Civil and Materials Engineering, UIC (MC 246) 2095 Engineering Research Facility 842 West Taylor Street, Chicago, IL 60607 – 7023	

6.4 APPENDIX D – A. Chudnovsky input, 12-04-2006,

"An Assessment of HDPE CP Specification for 100-years Service Life"

From: Chudnovsky, Alexander [achudnov@uic.edu]
Sent: Monday, December 04, 2006 4:27 PM
To: Richard Granata
Subject: Re: Status

Attachments: Granata.doc

Dr. Granata,
I am using different e-mail system, which is very unfriendly.
Attached is my assessment of our work.
Regards,

AC

An Assessment of HDPE CP Specification for 100-years Service Life

I. Introduction

It is my understanding that one of the important objectives of Specification for 100-years service life of HDPE CP is the designing of accelerated tests that provide a sufficient basis for the scientific assessment of HDPE CP reliability. Reliability in this case implies the probability (with certain confidence level) of "NO Failure" within first 100 years by either (a) one of the already observed mechanisms of HDPE CP field failure, or (b) an unobserved, but anticipated failure mechanism that may manifest itself in a longer period of service time, than the existing record of HDPE CP performance. "Anticipation" of failure mechanism may be based either on fundamental science, or on experience with similar product failures, or both.

Accelerated test conditions should be selected based on existing practice, with application of a very important criterion: to be relevant, the accelerated test must reproduce the observed or anticipated failure mechanisms.

Thus, to address the challenge to design HDPE CP 100-years service life specification, one starts with

1) observation, classification, and characterization of HDPE CP field failure, and
2) an analysis of potential modes of failure operating over a long period of time (20, 30, 50 years and more) that may not reveal itself in a shorter service time.

NCHRP Report 429, as well as KY, and OH task group evaluations of HDPE CP long-term performance partially address the task 1) above. A large amount of work is summarized in the referred above reports, which includes observation of HDPE CP field performance, including failures mechanisms, and some (limited) statistics of failures.

Large amount of work done on accelerated testing of MDPE and HDPE pipes for water distribution application, as well as premature field failure of PB tubing in water distribution systems suggest at least one potential mechanism of premature (with respect to 100 years) HDPE CP failure: stress corrosion cracking (SCC), also known as environmental stress cracking (ESC).

II. Background.

NCHRP Report 429, KY DOT, and OH DOT reports are essentially in agreement documenting various mechanisms of HDPE CP failure: circumferential and longitudinal cracking, tearing, sagging, buckling.

Thus, stress cracking resistance, and oxidation resistance, directly related to stress corrosion cracking is the concern of the specification.

Observations of circumferential and longitudinal cracking are reported in the above three reports. Some circumferential cracks are reported to propagate from outer liner surface inward, others grew outward; some cracks appear together with buckling and/or large deflection, others are formed with no noticeable buckling and/or deflection.

Apparently, the cracking is time dependent phenomenon.

An increase by 4 to 7 times in number of cracks have been reported in 2005 OH sites inspections in comparison to 2001 inspection of the same sites.

It is primarily important in designing an accelerated stress crack resistance (SCR) test to understand a critical combination(s) of loading, environment, and material parameters that leads to crack initiation and growth. The loading (that include pipe-soil interaction) should be translated into local stress state at corrugation-liner junction.

Among other parameters, material characterization should also include characterization of defects population that often is responsible for crack initiation.

Unfortunately, the question “what factor or a combination of factors plays the leading role in premature cracking” is not addressed in any of the reports referred above.

III. Shortcomings of FDOT Stress Crack Resistance Test.

FDOT SCR prediction for 100 years follows and refers to ASTM D 2837 Method. However, it significantly departs from ASTM D2837.

The limitations of FDOT SCR Test Procedure.

a) It employs a small size NCLS specimen (ASTM F 2136), in contrast with an actual pipe section testing according to ASTM D2837. Thus, FDOT protocol overestimate SCR, since it does not take into account a well-known scale effect: a small specimen exhibits higher short- and long-term strength than a large sample.

b) SCR test is conducted in simple tension. However, there is a complex stress state in various domains, where cracking has been reported, e.g., at corrugation-liner junction. Complex stress states, i.e., a combination of tension and shear, as well as intermittent stresses, are known to have a noticeable effect on crack initiation and growth (mix mode fracture, fatigue).

c) The specification employs too large extrapolation factor for failure time (extrapolation of less then a year test results into 100 years service time for material, which has been in service only 50 ~ 60 years). Taking in consideration the listed above limitations, the proposed extrapolation factor has no justification, and is highly speculative.

IV. Shortcomings of FDOT Oxidation Resistance (OR) Test.

OR test is a new protocol based on standard OIT test. It is proposed to address stress corrosion (or environmental stress) cracking. However, it does not take into account the fact that SCC (or ESC) is a surface phenomenon. Depletion of antioxidants (AO) from a thin surface layer (in order of 5% of wall thickness) results in surface layer degradation that leads to crack initiation and farther growth.

a) The standard OIT test measures AO content averaged over the pipe wall thickness. Surface depletion of AO is undetectable by conventional OIT. Thus, the proposed test is useless for SCC Resistance evaluation. Moreover, it may be misleading, if performed and reported.

b) The proposed test fails to differentiate HDPE Resin with respect to Resistance to AO Extraction (Leaching), which is an important factor in SCC resistance.

c) The specification proposes a highly speculative extrapolation of OR test results to 100-year Lifetime with insufficient experimental data and no fundamental science to support it.

V. Conclusion.

1. FDOT SCR testing is inadequate. More work should be done in the analysis of loading, load variations, stress state and environmental conditions that lead to cracking observed in the field prior to designing an accelerated test for SCR and it extrapolation technique.

2. FDOT OR Test Specification does not address SCC Problem. The standard OIT test is good for QC and basic material characterization only. Very different type of testing is required to determine a particular HDPE resin resistance to AO Extraction, Surface Degradation and Embrittlement as SCC precursors.

I suggest employing a simple technique of surface degradation detection, discussed in two references I have forwarded to Dr. Granata.

3. Overall, I am very disappointed with unproductive panel discussions. According to my understanding, the objective of the panel was to consider and sort out the concerns and objections expressed with respect to the specification. However, the panel failed this task. The members of the panel, except Dr. Stivala, would not even reconsider an obviously erroneous OR test, not to mention more technical objections to SCR testing.

Alexander Chudnovsky
Professor of Mechanics and Materials
UIC Distinguished Professor
Director of Fracture Mech. & Materials Durability Lab.
CME Department, UIC (MC 246)
2095 ERF, 842 W. Taylor Str., Chicago, IL 60607
Phone: (312) 996-8258; Fax: (312) 996-2426
E-Mail: achudnov@uic.edu