

# CHARACTERIZING LONG-TERM PERFORMANCE OF PLASTIC PIPING MATERIALS IN POTABLE WATER APPLICATIONS

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## ABSTRACT

Plastic piping materials confer many advantages for applications in Potable Water transportation. Considerable research has been conducted to develop methodologies for ensuring performance in these applications for service lifetimes that target an excess of 100 years. A key component of these accelerated methodologies is ensuring that the mechanisms observed in accelerated testing are the same as those potentially occurring in the field. In this paper, field exposed plastic piping materials are examined to determine the mechanisms of aging observed in actual end-use environments and compare those to accelerated validation methodologies employed in the laboratory. Based on these observations, strategies for performance validation are discussed.

## INTRODUCTION

Plastic piping materials have enjoyed a long and very successful history in potable water applications. With the significant evolutions in material performance that have occurred, current generation materials are expected to add significantly to this successful usage history.

As the different types of plastic piping materials and the number of applications for plastic piping materials increases, several test methodologies have been developed to forecast the long-term performance of plastic piping materials. In potable water applications, there are test methods such as ASTM F2023, *Standard Test Method for Evaluating the Oxidative Resistance of Crosslinked Polyethylene (PEX) Tubing and Systems to Hot Chlorinated Water* (1) and ASTM

*F2263, Standard Test Method for Evaluating the Oxidative Resistance of Polyethylene (PE) Pipe to Chlorinated Water (2).*

The test methodology for PEX pipe is generally applicable based on years of testing and comparisons to field performance (3, 4). For other polyolefin piping materials, validation of accelerated test methodologies is still in progress. It has been shown that field aging mechanisms in potable water applications are consistent with accelerated laboratory failures based on ASTM F2263 testing and that the method appears to be valid for examining the impact of disinfectants on lifetime performance (5).

In this paper, a deeper examination of field aging mechanisms is performed to identify the different mechanisms observed in the field as well as a review of the test method and its ability to evaluate lifetime performance of polyolefin materials in potable water applications.

As newer generation PE materials in traditional applications have target service lifetimes in excess of 100 years, obtaining field exposed specimens with sufficient levels of field exposure for direct assessment of the mechanisms of aging in the field is not possible. It is, therefore, difficult for these high performing materials to assess and ensure that the mechanisms observed in the field are comparable to those observed in the laboratory (and, thereby, validate the laboratory validation approach). It is still desired, however, to confirm that these mechanisms are the same so that the validation methodologies can be developed to assess the performance of these materials and determine their service lifetimes. In order to obtain field specimens for analysis, therefore, specimens that had been exposed to chlorine and chlorine dioxide disinfected water environments were exhumed for analysis.

## **EXPERIMENTAL PROCEDURE**

Polyolefin pipe samples from the field were examined. The specimens were exhumed from water service lines disinfected by chlorine or chlorine dioxide. Samples were examined using the following methods:

1. Visual examination through Optical Microscopy of the inner and fracture surfaces
2. Fourier Transform Infrared Spectroscopy (FTIR) on the fracture surface of selected specimens
3. Oxidation Induction Time (OIT) at 200°C (392°F) in general accordance with ISO 11357-6:2002 (E) (6) at the inner and mid walls

## RESULTS

Samples were visually examined and were observed to fall into two main categories:

- Samples with no apparent external load history
- Samples with an apparent external load history

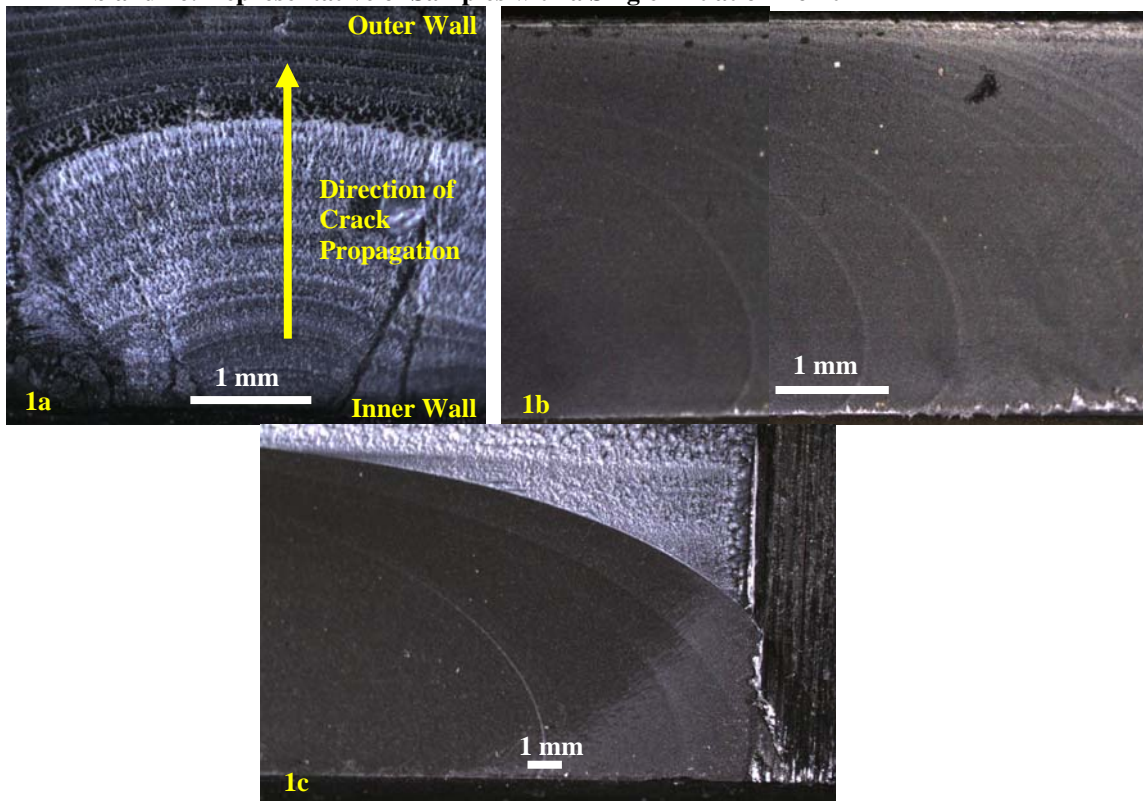
### Slit Mechanism with No Apparent External Load History

For all of the specimens with no apparent external load history, brittle cracks were observed to initiate at the inner wall and propagate from the inner to the outer wall. The majority of the samples were observed to have multiple initiation points with the exception of two samples, which were observed to have single initiation points. As shown in Figure 1, the major features of the fracture surface are the alternating bright/dark growth rings that coincide with the slip/stick (propagating/arresting) features of propagation under plane strain. The propagation direction can be determined from the growth rings as the rings indicate the crack fronts during propagation. All of the samples in this category show propagation from the inner wall to the outer wall and the slow crack growth ring features.

**Figure 1: Microscopy of Slow Crack Growth Rings of Samples without an External Load History**

**1a: Representative of Samples with Multiple Initiation Points**

**1b and 1c: Representative of Samples with a Single Initiation Point**



Differences in the nature of the slow crack growth rings are observed between the samples with multiple initiation points and those with a single initiation point. For the slit failures with multiple initiation points on the inner wall, the characteristic slow crack growth rings are observed to have alternating brittle/ductile growth rings (Figure 1a).

In contrast to these specimens, the specimens with slit cracks with a single initiation point show evidence of more axial propagation along the pipe wall in addition to the normal inner wall to outer wall propagation. As shown in Figures 1b and 1c, the differences between the dark and bright rings are much less defined than for the samples with multiple initiation points. As well, the subtle differences in the rings suggest that crack propagation is more brittle.

Table 1 shows that all of the specimens with through penetrating cracks have low OIT values at the inner wall indicating depletion of the antioxidants. The OIT value from the mid-wall, however, varies significantly between the samples. The variation at the mid-wall may be a result of the different disinfectant types. Samples B, C and E, which were disinfected with chlorine dioxide, have moderately high OIT values at the mid-wall. It has been shown in other studies that chlorine dioxide attacks the inner wall quickly and aggressively without significant depletion of the mid-wall (7). This suggests that the material at the mid-wall still maintains a certain level of ductility whereas the inner wall would appear to be embrittled from the chlorine dioxide exposure.

**Table 1: Samples without Apparent External Load History**

Sample ID	Disinfectant Type	General Appearance	Points of Initiation	Propagation Direction	OIT (minutes)	
					Inner Wall	Mid-Wall
A	Undetermined	Longitudinal Slit	Multiple	Inside-Out	0.2	6.0
B	Chlorine Dioxide	Longitudinal Slit	Multiple	Inside-Out	1.6	25
C	Chlorine Dioxide	Longitudinal Slit	Multiple	Inside-Out	1.0	53
D	Chlorine	Longitudinal Slit	Single	Inside-Out	0.7	4.9
E	Chlorine Dioxide	Longitudinal Slit	Multiple	Inside-Out	0.2	39
F	Chlorine	Longitudinal Slit	Single	Inside-Out	0.7	4.2

## Slit Mechanism with Apparent External Load History

Table 2 summarizes the specimens with evidence of an external load history. These cracks on the outer wall appear as longitudinal or non-longitudinal slits. A slit crack of this category typically has marks on the outer wall in the form of impingement and/or bending. These specimens differ from the specimens with slit cracks without an external load history in the following aspects:

- The sets of crack rings that develop from the initiation points on the inner wall may not be symmetrical around the initiation points as in the case of a slit cracking without an external load history
- A damaged area is often observed on the outer wall

**Table 2: Samples with Apparent History of External Loading**

Sample ID	Disinfectant Type	Crack Type	Loading History	OIT (minutes)	
				Inner Wall	Mid-Wall
G	Chlorine	Longitudinal Slit	Impingement	0.4	5.2
H	Chlorine	Longitudinal Slit	Impingement	0.2	0.2
I	Chlorine Dioxide	Longitudinal Slit	Impingement	0.3	9.2
J	Chlorine	Longitudinal Slit	Impingement	0.5	4.5
K	Chlorine Dioxide	Longitudinal Slit	Impingement	0.2	1.7
L	Chlorine Dioxide	Longitudinal Slit	Impingement	0.2	9.7
M	Chlorine	Longitudinal Slit	Impingement	0.2	0.9
N	Chlorine Dioxide	Non-Longitudinal Slit	Impingement plus bending	0.3	3.0
O	Chlorine	Non-Longitudinal Slit	Bending	0.2	0.5
Q	Chlorine Dioxide	Longitudinal Slit	Impingement	0.4	22
R	Chlorine Dioxide	Non-Longitudinal Slit	Impingement	0.9	24

These slit type cracks typically initiate from two or more locations at the inner wall directly under the external loading marks on the outer wall. Slow crack growth rings are observed for some samples, as illustrated in Figure 2. The contribution of the external load is typically evident based on the appearance of the damage on the outer wall and of the morphology of the fracture surface. The samples with non-longitudinal slits are usually observed to have evidence of bending in addition to the signs of external loading.

**Figure 2: Microscopy of a Sample with an External Load History  
(Slow Crack Growth Rings near the Center of Failure on the Inner Surface)**

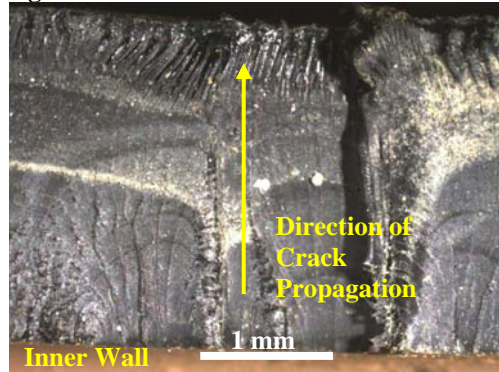


Table 2 shows that, in general, the specimens have low OIT values at the inner wall indicating depletion of the antioxidants. The OIT value of the mid-wall, however, varies significantly between the samples. Similar to the samples without an apparent external load history, samples with chlorine dioxide exposure generally have higher mid-wall OIT values compared to the samples with chlorine exposure.

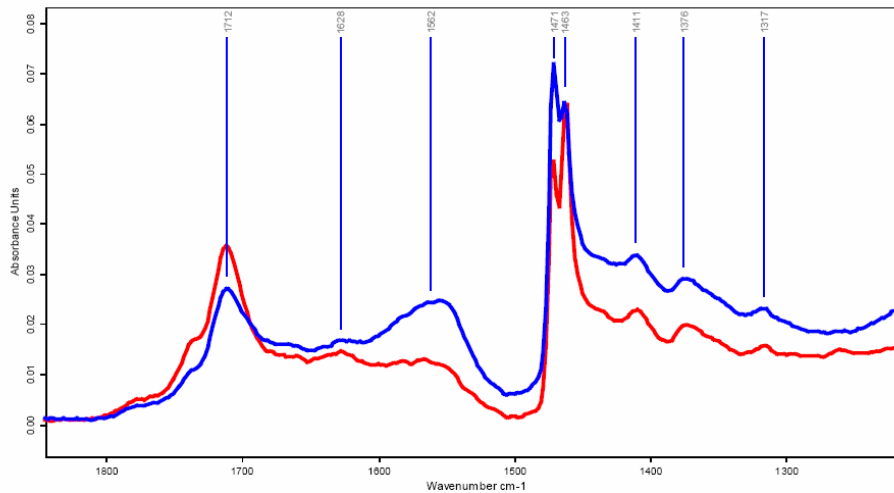
## **DISCUSSION**

### **Slit Mechanism with Multiple Initiation Points - No Apparent External Load History**

The specimens with slit cracking at multiple initiation points appear to initiate under a plane strain stress mode at or near the inner wall and propagate from the inner wall to the outer wall. The initiation under plane strain mode confirms that the hoop stress is generally the dominant factor causing fracture. These cracks appear to be typical of slow crack growth under hoop stress.

The OIT values are low on the inner surface for the majority of these samples. The mid-wall OIT values indicate that some antioxidant remains in the mid-wall for some of the samples. To determine if the low OIT value at the inner wall was only an indication of antioxidant depletion or a sign that degradation had initiated, FTIR analysis of a selected number of samples was performed. The presence of the carbonyl peak in the region of  $1812$  to  $1660\text{ cm}^{-1}$ , as shown in Figure 3, indicates significant oxidation on the inner wall. As mentioned above, the samples are observed to have distinctive slow crack growth features of propagation. Together, these results suggest that the inner surface has experienced oxidative degradation which has led to slow crack growth over time. These failures appear to be Mode 3 (Oxidative initiation, mechanical propagation) failures (3).

**Figure 3: FTIR of Sample A Indicating Significant Oxidation of the Inner Wall and on the Fracture Surface**



### **Slit Mechanism with a Single Initiation Point - No Apparent External Load History**

In contrast to the multiple initiation point failures, crack propagation in the longitudinal direction is observed for the two slit failures with single initiation points (Samples D and F as shown in Figures 1b and 1c, respectively). The two samples have axial propagation features on the fracture surface that appear to be more brittle than for the other samples.

Evidence of material degradation is observed for these two samples. The samples have low OIT values at the inner wall and the mid-wall. The low mid-wall OIT values suggest that, if the sample had antioxidants prior to service, a depletion of the antioxidants has not only occurred at the inner wall but also at the mid-wall. As antioxidant depletion starts at the inner surface, this would imply that sufficient time has passed such that the antioxidant is completely consumed from the inner wall allowing oxidative degradation to initiate. The degradation in the bulk wall may have been initiated regardless of the presence of antioxidants in the mid-wall due to limited antioxidant migration to antioxidant depleted areas. Thermal degradation in the bulk wall may have caused a reduction of ductility of the bulk pipe material and made the pipe vulnerable to any impact and resulted in the observed fast, brittle fracture.

Regardless of the precise degradation mechanisms, the low mid-wall OIT values suggest that the observed brittle propagation may have partially been a result from degradation of the pipe material through the entire pipe wall. However, the properties of the original pipe material prior to service are unknown. These failures also appear to be Mode 3 (Oxidative initiation, mechanical propagation) failures.

## **Slit Mechanism Apparent External Load History**

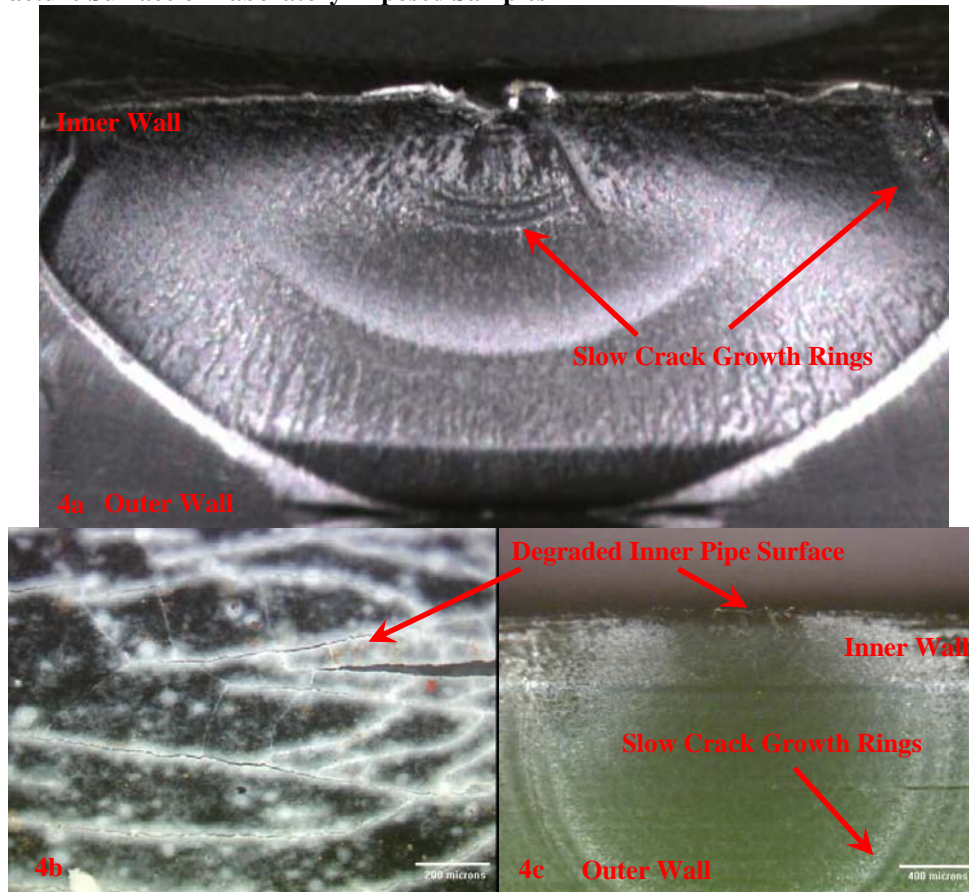
Failures with apparent external load history appear to have resulted from various situations due to additional mechanical loading in the application.

The OIT analysis suggests that stabilizer depletion and inner surface degradation occur as the initial phase of the degradation process. The majority of the samples have low OIT values at the inner wall. In addition to the low OIT values at the inner wall, several of the failures also have low OIT values at the mid-wall which suggests that degradation has initiated on the inner surface of the samples. The samples are also observed to have distinctive slow crack growth features of propagation. Similar to the specimens without apparent external load history, these failures appear to be Mode 3 (Oxidative initiation, mechanical propagation) failures.

## **COMPARISON TO LABORATORY FAILURES**

It has been previously shown that aging mechanism observed in Laboratory testing is stabilizer depletion at the inner surface followed by oxidative degradation of the inner surface followed by slow crack growth (5). Figure 4 provides an example of the fracture face of a laboratory tested sample. Figure 4b shows the degradation on the inner pipe surface. Based on the observed field specimens, it would appear that the same mechanisms are occurring in the field as observed in accelerated testing. The ASTM F2263 test methodology, therefore, would appear to replicate the aging mechanisms observed in the field.

**Figure 4: Fracture Surface of Laboratory Exposed Samples**



## **CONCLUSIONS**

Through examination of field exposed pipe specimens and laboratory specimens exposed to accelerated conditions several modes of aging with common features were identified. The crack mechanisms for both the field and laboratory exposed specimens appear to be Mode 3 (Oxidative initiation, mechanical propagation), suggesting that the ASTM F2263 test methodology is capable of replicating the mechanisms of field aging under accelerated conditions. This methodology, therefore, appears able to provide a basis for the development of validation methodologies for demonstrating the performance properties of potable water piping.

## **ACKNOWLEDGEMENTS**

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