

Public Quarterly Report

Date of Report: 8th Quarterly Report-November 6, 2024

Contract Number: 693JK322RA0001

Prepared for: US Pipeline and Hazardous Materials Safety Administration

Project Title: Rapid Ultraviolet (UV) Cured Adhesive for Gas Main Cured-in-Place-Lining (CIPL)

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For quarterly period ending: October 30, 2024

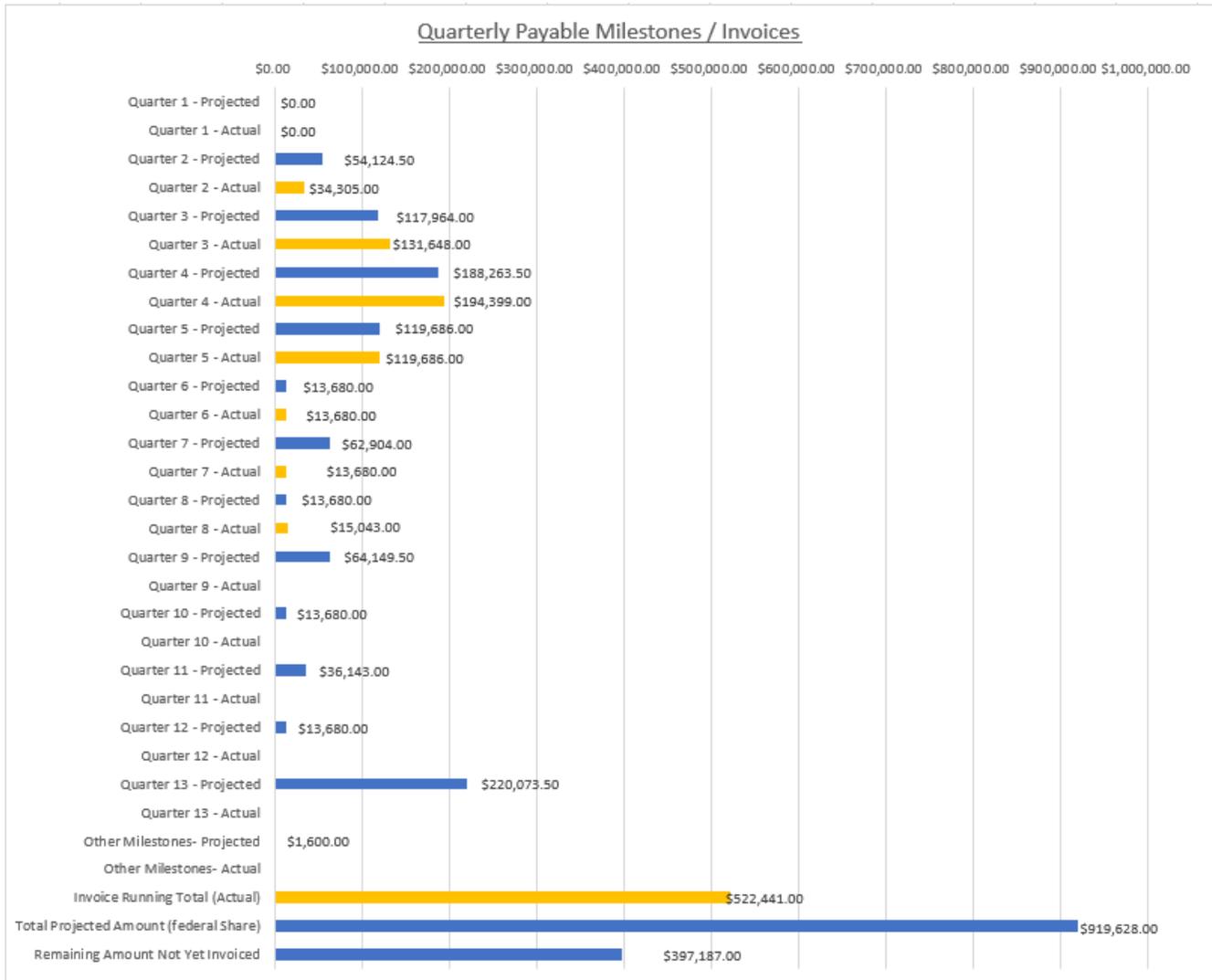
1: Items Completed During this Quarterly Period:

<i>Item #</i>	<i>Task #</i>	<i>Activity/Deliverable</i>	<i>Title</i>	<i>Federal Cost</i>	<i>Cost Share</i>
37	32	Coupon Testing - Lap Shear Strength	<i>Coupon Testing - Lap Shear Strength</i>	\$681.50	\$681.50
38	33	Coupon Testing - Composite Liner Peel Strength	Coupon Testing - Composite Liner Peel Strength	\$681.50	\$681.50
44	7	8th Quartey Status Report & Data Analysis	8th Quartey Status Report	\$13,680.00	\$13,680.00

2: Items Not-Completed During this Quarterly Period:

<i>Item #</i>	<i>Task #</i>	<i>Activity/Deliverable</i>	<i>Title</i>	<i>Federal Cost</i>	<i>Cost Share</i>
39	34	Chemical Resistance [Adhesive System (Post-Installation and Cure)]	Chemical Resistance	\$3,964.00	\$3,964.00
40	35	Chemical Resistance (composite, post-installation and cure)	Chemical Resistance	\$6,606.50	\$6,606.50
42	37	Laboratory Mobilization & Test Plans	Laboratory Mobilization & Test Plans	\$37,290.50	\$37,290.50

3: Project Financial Tracking During this Quarterly Period:



4: Project Technical Status –

Introduction

This report details the findings of the following tests performed at CIEST:

- ASTM D790: “Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials” (2017) (Procedure B – 0.10 mm/mm rate of straining of the outer surface of the test specimen)
- ASTM D638: “Standard Test Method for Tensile Properties of Plastics” (2022) (Rigid and Semirigid classification Type I specimens with speed of testing $5 (0.2) \pm 25 \% \text{ mm/min}$)
- ASTM D695: “Standard Test Method for Compressive Properties of Rigid Plastics” (2023).

- ASTM D543-21: “Standard Practices for Evaluating the Resistance of Plastics to Chemical Reagents” (2021)
- ASTM D3167-10: “Standard Test Method for Floating Roller Peel Resistance of Adhesives” (2017)
- ASTM D5868-95: “Standard Test Method for Lap Shear Adhesion for Fiber Reinforced Plastic (FRP) Bonding” (2024)
- ASTM D2240-15: “Standard Test Method for Rubber Property—Durometer Hardness” (2021) (Type D handheld durometer)
- ASTM F2207-06: “Standard Specification for Cured-in-Place Pipe Lining System for Rehabilitation of Metallic Gas Pipe” (2019)

The test involves different dimensions of samples made of curable plastic adhesives for use in cured-in-place pipe liners (CIPL), as well as coupon metal pipe samples with the liner adhered. Current cured in place pipe liners are available on the market, however these systems take multiple days to cure before the pipe can be deemed safe and usable. Ultraviolet curable plastic adhesives aim to drastically cut down on the curing time of these pipe liners and allow for pipelines to minimize their downtime and potential negative effects of taking a pipeline out of service for an extended amount of time. The procedure focuses on determination of the mechanical properties of curable plastic adhesives, when loaded in compression, flexural, and tensile properties at relatively uniform rates of straining, as well as determining the peel and lap shear strength of the adhesive, and the hardness of the pipe liner. **Table 1.1** contains the summary of the final test results from flexural, compressive and tensile testing of the ultraviolet cured adhesive samples. Results from peel, lap shear and hardness testing of the composite pipe and liner specimens are given in Section 6 through 9.

Table 1.1: Summary of test results

SAMPLE ID	MAXIMUM FLEXURAL FIBER STRESS (kPa) ASTMD 790	FLEXURAL MODULUS OF ELASTICITY (kPa) ASTMD 790	TENSILE STRENGTH @MAX (N) ASTMD 638	TENSILE ELONGATION @MAX (%) ASTMD 638	COMPRESSIVE STRENGTH @MAX (N) ASTMD695
---	115,377	5.61×10^7	1,325	0.916	46,256

Test Specimens

As shown in **Figure 2.1**, All test specimens were made and shipped to the University of Colorado Boulder. In each package, there were five specimens for each standard, totaling 15 specimens overall. One side of the specimens was sticky with sharp edge while the other side was dry with rounded edge. Some of the specimens contained one or two trapped air bubbles. The specimens had a yellowish transparent color. For each standard, the test procedure was repeated five times (following recommendations for standard). The test specimens were numbered in order of #. Calipers with a precision of .02mm were used to measure dimensions. These measurements were performed on each individual testing coupon.



Figure 2.1: Received package of specimens

Flexural Tests (ASTM D790)

Flexural Test Specimens

As shown **Figure 3.1**, a total of 5 samples were sent for flex test. Coupon dimensions were manufactured with a rectangular shape following recommendations of ASTM D790 for “High Strength Reinforced Composites” as:

- *Support span-to-depth ratio of 16:1 is ok if the tensile strength to shear strength is less than 8:1.*
- *If the coupon has low shear strength in the plane of the laminate and high tensile strength parallel to the support span 32:1 or 40:1 is recommended.*
- *For highly anisotropic materials support span-to-depth ratio of 60:1 is to be used.*
- *Coupon should be rectangular, and the depth should not exceed the width of the coupon.*
- *For our testing setup the maximum support span we have that would allow for overhang of the coupon on either side is 200 mm.*

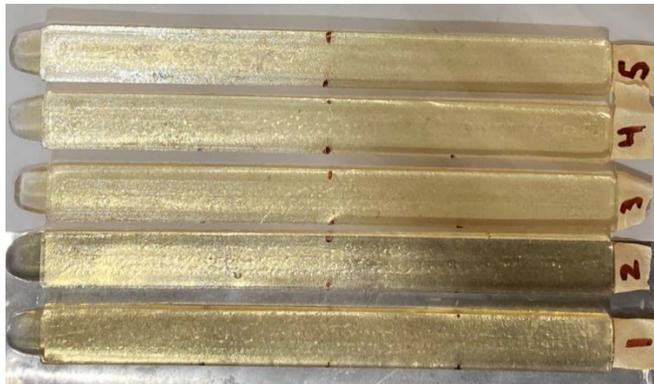


Figure 3.2: Flex test specimens

The measurements of the flex test specimens dimension can be found in

Table 3.1. On average, the width, depth, and span measurements are 12.35mm, 3.54mm, and 56.73mm, respectively.

Table 3.2: Dimensions of the flex test samples

(mm)	S01	S02	S03	S04	S05
Width	12.33	12.36	12.46	12.32	12.3
Depth	3.46	3.45	3.59	3.6	3.63
Span	56.74	56.74	56.74	56.74	56.74

Flexural Test Setup (ASTM D790)

The midpoint of all samples was determined. The loading nose and supports were aligned carefully to ensure accuracy. The specimen was centered in the middle of the supports with its long axis perpendicular to the loading nose and supports. Load-deflection data was recorded while applying the load at a specified rate. **Figure 3.3** shows the loaded sample.

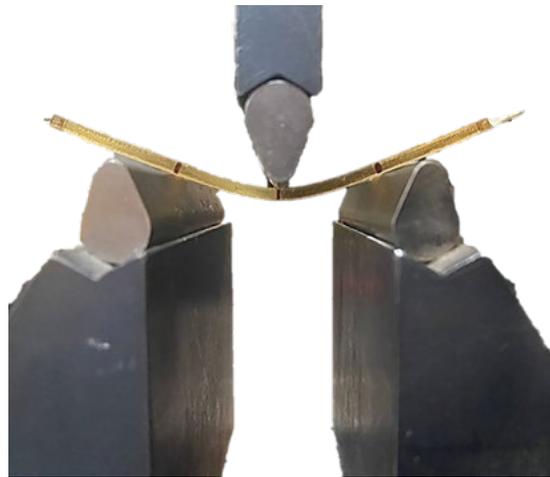


Figure 3.3: Flexural test loading procedure

To compensation was performed to correct for seating. A controlled deformation process was performed on the test specimen with a uniform strain rate of 15.33 mm/min applied to the outer surface. The structural integrity of the specimen was strictly maintained, and the maximum deflection was limited to 7.66 mm. The sample broke in the middle section where the load was applied before reaching maximum deformation. Based on observation, the specimens exhibited brittle behavior. **Figure 3.3** shows the specimens that were tested.

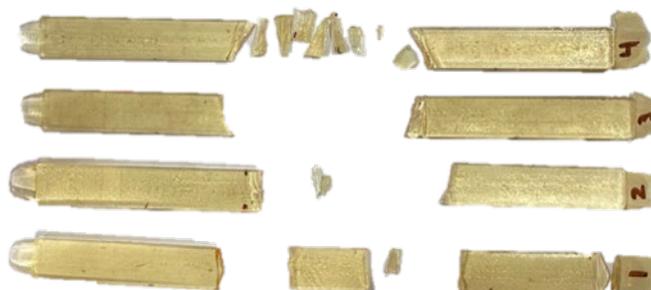
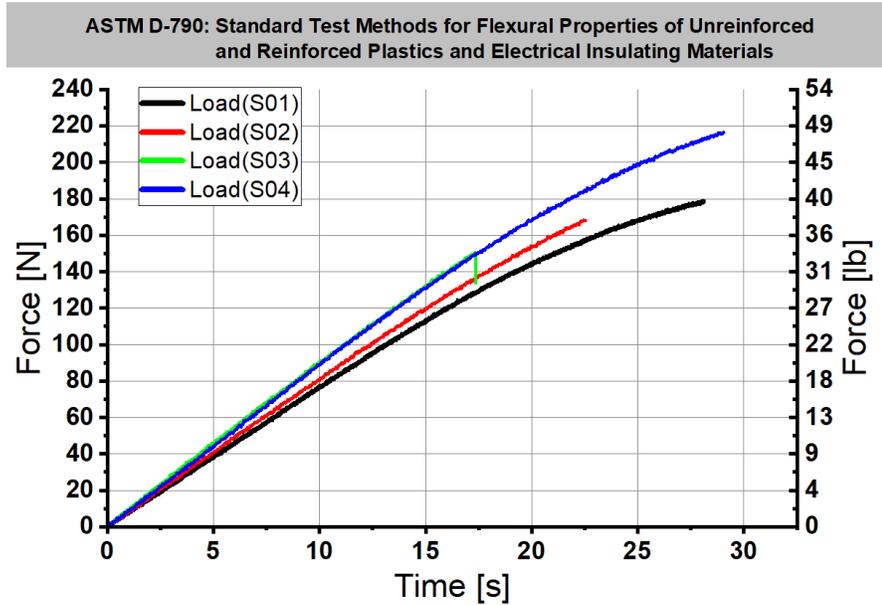


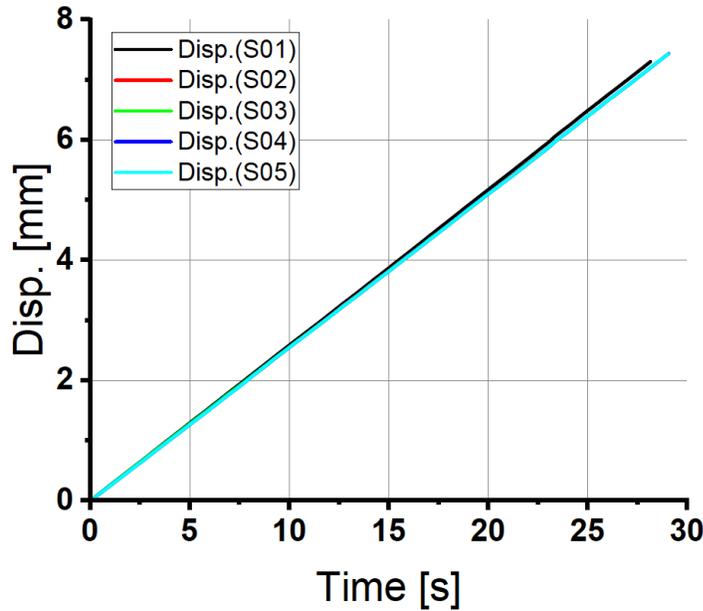
Figure 3.4: Specimens after flexural test

As the loading rate for the first sample was determined through "Procedure A," it did not fail at 5% strain. Therefore, the remaining samples were subjected to testing using the loading rate specified in "Procedure B." The ASTM D790 document makes note that *“For some materials that do not yield or break within the 5 % strain limit when tested by Procedure A, the increased strain rate allowed by Procedure B (see 10.2) may induce the specimen to yield or break, or both, within the required 5 % strain limit.”*

Flexural Test Results



(a)



(b)

Figure 3.5: Flexural test loading procedure including: (a) force vs. time, (b) displacement vs. time

Tensile Tests (ASTM D638)

Tensile Test Specimens

As shown **Figure 4.1**, a total of 5 samples were sent for tension test. Coupon dimensions were manufactured with a dogbone shape following recommendations of ASTM D638 for “Type I specimen” as shown in **Figure 4.2**. The measurements of the tensile test specimens dimension can be found in **Table 4.1**.



Figure 4.6: Tensile test specimens

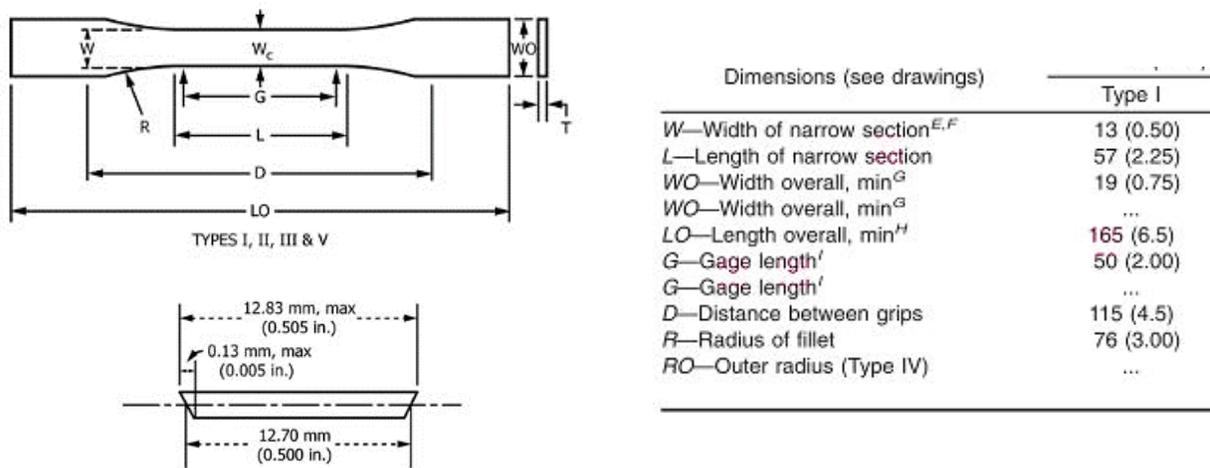


Figure 4.7: Dimensions for samples according to ASTM D638

Table 4.3: Dimensions of the tensile test samples

	(mm)	S01	S02	S03	S04	S05
Center	Width	12.5	11.21	12.47	12.49	11.17
	Thickness	3.31	2.09	3.38	3.32	2.02
Right	Width	12.46	11.24	12.66	12.49	11.27
	Thickness	3.33	2.16	3.43	3.22	2.06
Left	Width	12.48	11.21	12.44	1.73	11.18
	Thickness	3.34	1.89	3.22	3.37	1.85
Average	Width	12.48	11.22	12.52	8.90	11.20
	Thickness	3.33	2.05	3.34	3.30	1.98

Tensile Test Setup (ASTM D638)

As shown in **Figure 4.3**, to prevent specimens from shattering at the end where they are attached to the grips, both ends of the coupons were sanded due to slight sectional curvature. This process was necessary as specimens without it had a tendency to break.

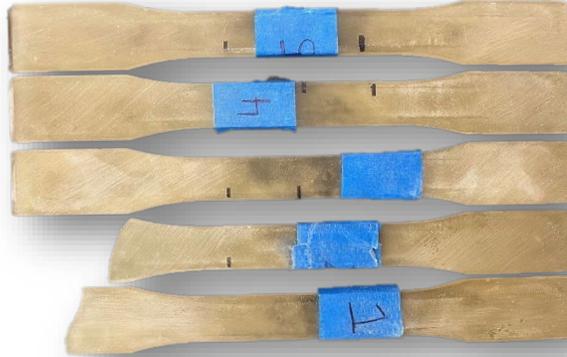


Figure 4.8: Tensile test specimens

The width and thickness of flat specimens were measured at the center of each specimen and within 5 mm of each end of the gage length. As shown in **Figure 4.4**, the specimen was placed in the grips of the testing machine, with attention given to aligning the long axis of the specimen and the grips along a straight line that connected the points of attachment of the grips to the machine.

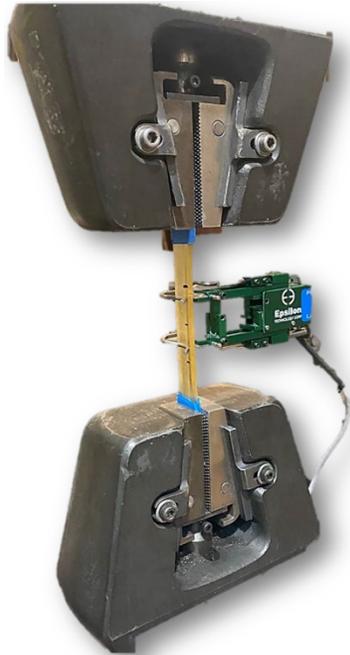


Figure 4.9: Tensile test loading procedure

The distance between the ends of the gripping surfaces was maintained according to the standard procedure. The grips were tightened evenly to the extent required to prevent any slippage of the specimen during the test, while avoiding excessive compression of the specimen. The extensometer was attached in the middle of the specimen.

The speed of testing was set to 5 mm/min, and the machine was initiated. **Figure 4.5** shows the specimens that were tested without sanding at the end points, which is not considered standard practice.



Figure 4.10: Specimens after tensile test

Tensile Test Results (ASTM D638)

Figure 4.6 and Figure 4.7 provide tensile coupon results for two specimens. Figure 4.6 provides various measured values vs. time of test while Figure 4.7 provided the stress vs. strain data up until ultimate coupon capacity.

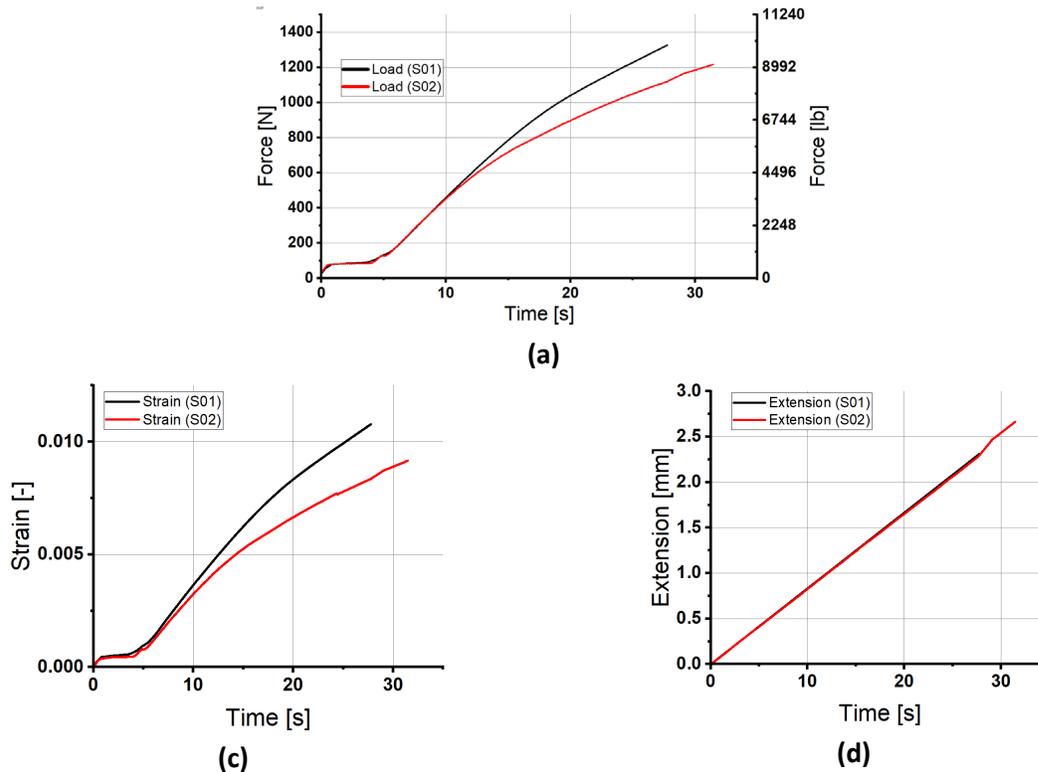


Figure 4.11: Tensile test loading procedure including: (a) force vs. time, (b) strain vs. time, and (c) displacement vs. time

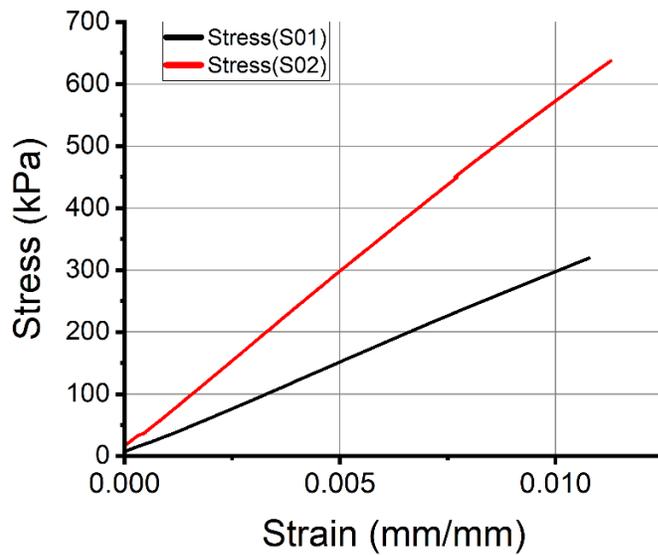


Figure 4.12: Tensile coupon stress-strain data

Compressive Tests (ASTM D695)

Compressive Test Specimens

As shown **Figure 5.1**, a total of 5 samples were sent for compressive test. Coupon dimensions were manufactured with a cylindrical shape following recommendations of ASTM D695 for “Reinforced Plastics” as:

- For materials 3.2 mm to 6.4 mm thick the specimen used for strength measurements shall consist of a prism having a cross section of 12.7 mm by the thickness of the material and a length of 21.7 mm
- For material greater than 6.4 mm in thickness, specimens should be a prism whose length is twice its principal width. Preferred sizes are 12.7 x 12.7 x 25.4 mm.
 - If getting modulus or offset yield measurement the slenderness ratio should be from 11-16:1 which equates to 12.7 x 12.7 x 50.8 mm.



Figure 5.13: Compressive test specimens

The measurements of the compressive test specimens’ dimension can be found in **Table 5.1**.

Table 5.4: Dimensions of the Compressive test samples

(mm)	S01	S02	S03	S04	S05
Width	12.34	12.37	12.4	12.37	12.42
	12.37	12.38	12.34	12.36	12.34
	12.38	12.39	12.4	12.45	12.43

Average Width	12.36	12.38	12.38	12.39	12.39
Depth	12	11.31	12.13	11.61	12.19

Compressive Test Setup ASTM D695

To ensure that the ends of the specimen were parallel with the surface of the compression tool, both end of samples were sanded as shown in **Figure 5.2**.



Figure 5.14: Compressive test specimen after sanding

The width and thickness (or diameter) of the specimen were measured to the nearest 0.025 mm (0.001 in.) at multiple points along its length. The minimum value of the cross-sectional area was calculated. The length of the specimen was measured. As shown in **Figure 5.3**, specimen was positioned between the surfaces of the compression tool, ensuring that the center line of its long axis was aligned with the center line of the plunger.

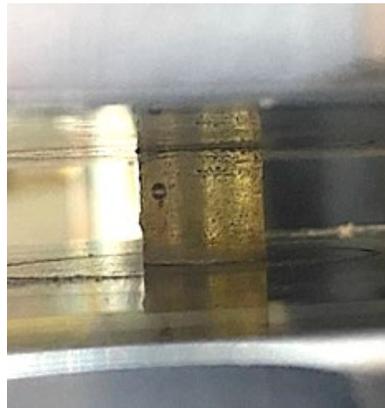
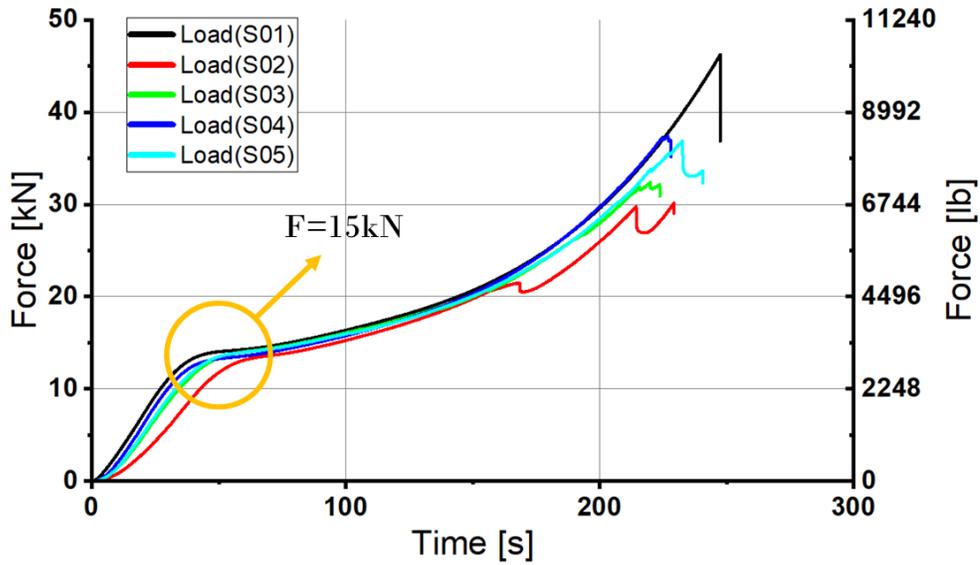


Figure 5.15: Compressive test loading procedure

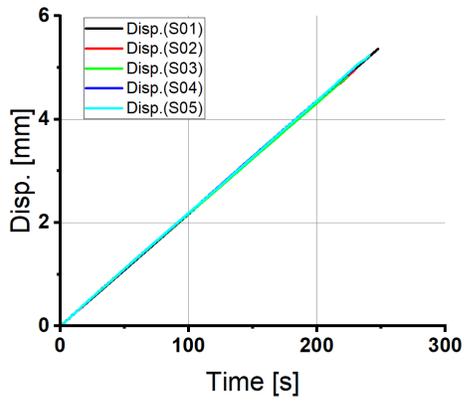
The crosshead of the testing machine was adjusted until the top of the compression tool plunger was barely in contact with it. The nuts or screws on the jig were kept at finger-tightness. The speed control was set to 1.3 mm/min, and the machine was initiated.

1.1 Compressive Test Results (ASTM D695)

This section provides results from the compression tests. **Figure 5.4** shows results from five (5) tests performed on the specimens previously discussed.



(a)



(b)



(c)

Figure 5.16: Compressive test loading including: (a) test results, (b) Compressive displacement vs. time, and (c) Post test (typical)

Peel Tests (ASTM D3167-10) (Task 33)

Peel Test Specimens

The peel test samples are approximately ½-inch thick, 1-inch-wide coupon samples, cut from both cast iron and steel pipelines. Both pipelines are lined with composite internal replacement pipe liner. Each coupon was measured to determine the length and width of the area of metal adhered to the liner. These measurements were taken using calipers with a precision of 0.0005in (0.01mm). The objective of the peel test is to determine the strength required to break the bond between the liner and host pipe through peeling the liner from the host pipe coupon.

Peel Test Setup

These tests were performed on an Instron Universal Test Machine, model 5869, with a load cell capable of measuring up to 1124.04 lbf (5 kilonewtons). A peel fixture was ordered to match the specification in the standard. However, the coupons were larger than the standard specified. As such, the design of the peel fixture was modified such that the coupons could fit inside of it for testing.

The peel fixture was modified so that the rollers would have more distance between them, and the triangle plates would be farther apart. **Figure 7.1** shows the new design of the triangle plates. The center of the holes pictured in the drawing are also the center of the rollers. The distance between the two horizontal holes on the bottom was increased from 1.20 inches to 1.85 inches. The distance between the two vertical holes on the right side was increased from 1.3 inches to 2 inches. These two distances were increased by a factor of ~1.54 to keep the peel angle the same before and after modification. Further modifications included: increasing the distance between the two plates to 1.8625 in. and lengthening the pins to 2.602 in. The original rollers were kept in the design of the new peel fixture, and washers were used as spacers to keep the rollers centered on the larger pins.

During preliminary testing the coupons would sometimes rotate to the side on the rollers, causing rubbing of the coupon on the new triangle side plates and a diagonal peel. To fix the issue, acrylic triangle plates were fabricated. These plates were placed on the inside of the steel plates as pictured in **Figure 7.2**. The acrylic spacers reduced the space between plates to 1.398 in. This ensured a straight, low-friction travel of the specimen on the peel fixture.

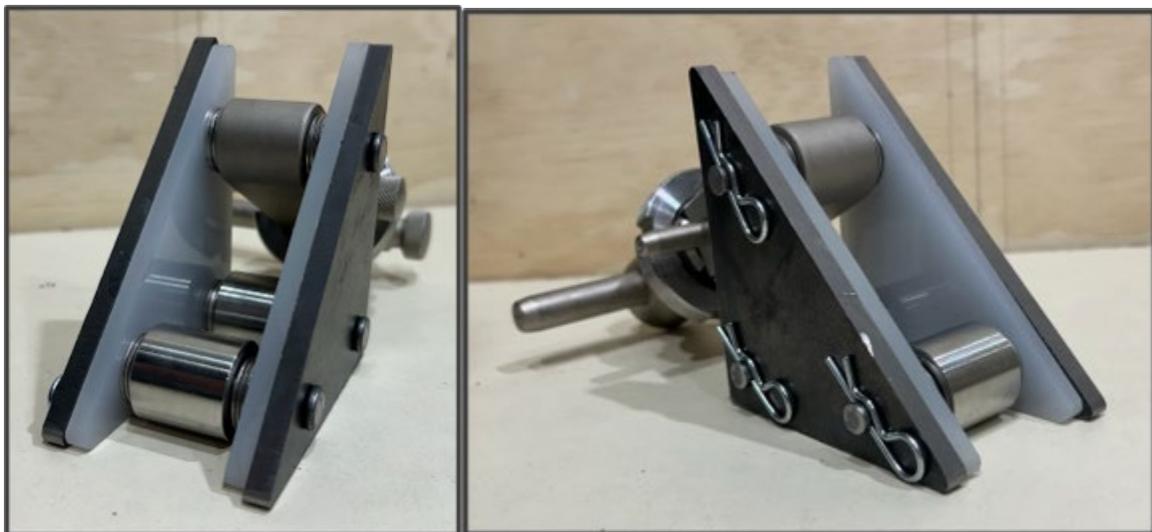


Figure 7.17: The final modified floating roller peel drum.

Due to the modifications of the test fixture, each sample was pre-peeled one inch by hand to prevent it from slipping out of the peel fixture during testing. The liner was gripped using the lower Instron tension grip, and tensile load was applied at a constant speed of 6-in./min (152 mm/min). Each test was conducted until the liner was fully detached from the metal. Two control samples of each material were tested to obtain baseline peel strength values.

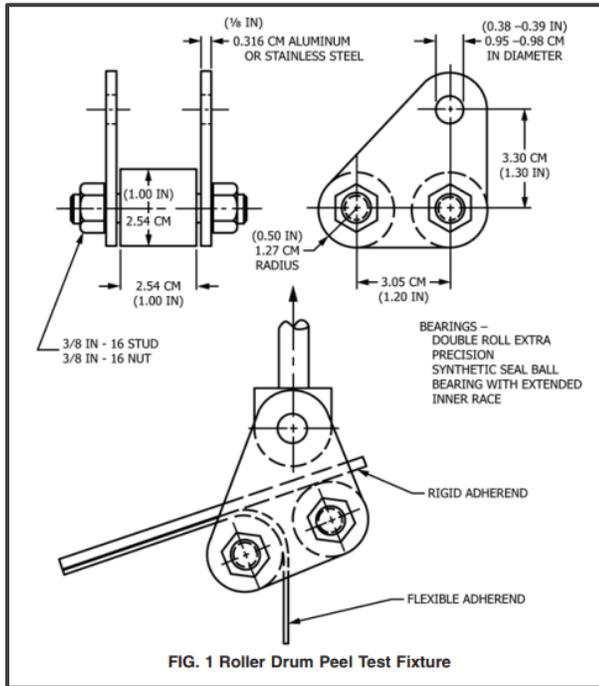


Figure 7.18: Peel Test Setup

Peel Test Results

The average peel strength of the steel control tests was found to be 6.39 lbf/in, and the average peel strength of the cast iron control tests was found to be 8.39 lbf/in. These results meet the minimum peel strength of 6 lbf/in required by the standard. The results of the peel tests are shown in **Figure 7.3**. At least three inches of data is taken from each peel test, disregarding the first one inch of peel.

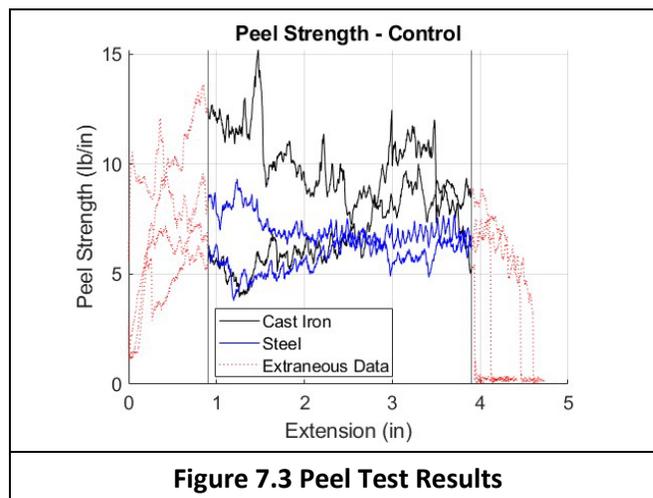


Table 7.5: Peel test results showing the average peel strength (lb/in.) for each test type.

Coupon Treatment	Steel	Cast Iron
Control	6.39	8.39

The tables below give the type of failure for each peel test specimen. Cohesive failure indicates that the adhesive separated from itself, while adhesive failure indicates that the adhesive separated completely from the host metal.

Table 7.6: Type of Peel Failure per Coupon – Cast Iron

Chemical Treatment	Coupon 1	Coupon 2
Control	Cohesive	Cohesive

Table 7.7: Type of Peel Failure per Coupon – Steel

Chemical Treatment	Coupon 1	Coupon 2
Control	Cohesive	Cohesive

Hardness Tests (ASTM D2240)

Hardness Test Specimens

The hardness test samples are approximately ½-inch thick, 1-inch-wide, and 6-inch-long coupon samples, cut from both cast iron and steel pipelines. An 8-inch-long internal pipe replacement liner is adhered to all test samples of both types. One cast iron and one steel coupon were used for control hardness testing.

Hardness Test Setup

Hardness testing was performed with a Rex Handheld Durometer, Model 1600-D (Type D). Each coupon was laid flat on a table with the inner face of the liner facing upwards. The durometer was pressed into the inner face of the liner for one second, and the value shown on the dial was recorded. This process was conducted five separate times on each coupon. Each test was conducted along the center line of the coupon and was at least 0.24in (6.0mm) away from any other hardness test sites on the same coupon.

Hardness Test Results

The average hardness of the control tests was 48.5.

Table 8.8: Hardness test results showing the average hardness value for each test type.

Coupon Treatment	Average Hardness
Control	48.5

Lap Shear Tests (ASTM D5868-95) (Task 32)

Lap Shear Specimens

The lap shear test samples are approximately ½-inch-thick, 1-inch-wide, and 6-inch-long coupon samples, cut from both cast iron and steel pipelines. An 8-inch-long internal pipe replacement liner is adhered to all test samples of both types. Each coupon was measured to determine the length and width of the area of metal adhered to the liner. These measurements were taken using calipers with a precision of 0.0005in (0.01mm).

Lap Shear Setup

These tests were performed on an MTS Exceed Electromechanical Test System, model E43-504, with a load cell capable of measuring up to 11,240.45 lbf (50 kilonewtons). To prepare the specimens for testing, each coupon was peeled one inch from the end of the specimen with no excess liner. The liner was pushed to the side and the exposed one inch of metal was placed in the lower tensile grips of the MTS. About one inch of liner from the other side of the specimen was placed in the top grips. The lap shear test setup can be seen in **Figure 9.1**. All specimens underwent a steady loading rate of 0.5 in/min (13 mm/min).

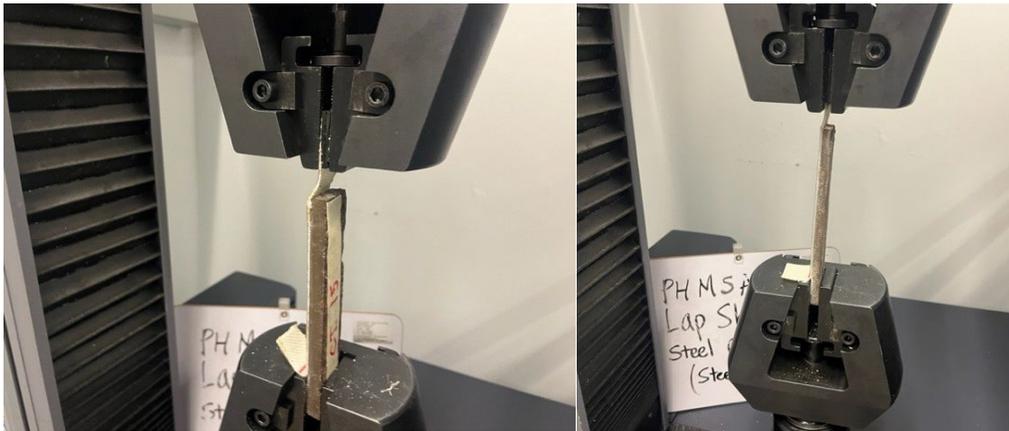


Figure 9.19: Lap Shear Testing Setup

Lap Shear Results

The Lap Shear testing results are plotted below in Figure 9.2.

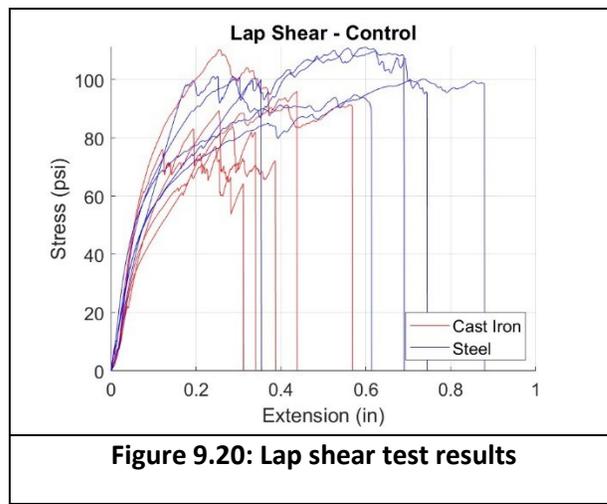


Figure 9.20: Lap shear test results

The peak load attained by each coupon was recorded and stress was calculated using the measured area of the coupon. These peak stresses are given in the tables below.

Table 9.9: Lap shear test results showing the maximum stress value for each Cast Iron coupon test.

Chemical Treatment	Coupon 1	Coupon 2	Coupon 3	Coupon 4	Coupon 5
Control	89.33	77.14	83.86	91.34	110.28

Table 9.10: Lap shear test results showing the maximum stress value for each Steel coupon test.

Chemical Treatment	Coupon 1	Coupon 2	Coupon 3	Coupon 4	Coupon 5
Control	100.15	94.83	101.15	111.07	110.75

The tables below give the maximum, minimum, and average peak stresses for the Lap Shear tests.

Table 9.11: Lap shear test results showing the maximum, minimum, and average stress for each Cast Iron test type.

Chemical Treatment	Maximum Stress (psi)	Minimum Stress (psi)	Average Stress (psi)
Control	110.28	77.14	90.39

Table 9.12: Lap shear test results showing the maximum, minimum, and average stress for each Steel test type.

Chemical Treatment	Maximum Stress (psi)	Minimum Stress (psi)	Average Stress (psi)
Control	111.07	94.83	103.59

The type of failure experienced by each coupon sample is given in the tables below. Most specimens experienced cohesive failure (separation of the adhesive from itself, or separation of the liner from the adhesive) rather than adhesive failure (complete separation of the liner and adhesive from the host metal).

Table 9.13: The type of failure observed for every Cast Iron lap shear test performed.

Chemical Treatment	Coupon 1	Coupon 2	Coupon 3	Coupon 4	Coupon 5
Control	Cohesive (epoxy still on the sample)	Adhesive (epoxy remains on the liner)	Cohesive	Cohesive	Cohesive

Table 9.14: The type of failure observed for every Steel lap shear test performed.

Chemical Treatment	Coupon 1	Coupon 2	Coupon 3	Coupon 4	Coupon 5
Control	Cohesive	Cohesive	Cohesive	Cohesive	Cohesive

References

ASTM D638-22. (2022). Standard Test Method for Tensile Properties of Plastics. ASTM Book of Standards, May, 1–16. <https://doi.org/10.1520/D0638-22>

ASTM D695-23. (2023). Standard Test Method for Compressive Properties of Rigid Plastics. ASTM International, i(April 2003), 1–8. <https://doi.org/10.1520/D0695-23>

ASTM D790-17. (2017). Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. Annual Book of ASTM Standards, 1–12. <https://doi.org/10.1520/D0790-17>

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ASTM International. (2023). *Standard Test Method for Lap Shear Adhesion for Fiber Reinforced Plastic (FRP) Bonding* (ASTM D5868-01 (2023)). ASTM International. <https://doi.org/10.1520/D5868-01R23>

ASTM International. (2019). *Standard Specification for Cured-in-Place Pipe Lining System for Rehabilitation of Metallic Gas Pipe* (ASTM F2207). ASTM International. <https://doi.org/10.1520/F2207>

5: Project Schedule –

- Items not complete in Q8, possibly to be included in the Q9 report are as follows:

Item #	Task #	Activity/Deliverable	Title
39	34	Chemical Resistance [Adhesive System (Post-Installation and Cure)]	Chemical Resistance [Adhesive System (Post-Installation and Cure)]
40	35	Chemical Resistance (composite, post-installation and cure)	Chemical Resistance (composite, post-installation and cure)
46	39	Traffic Loading/fatigue & Parallel Excavation	Traffic Loading/fatigue & Parallel Excavation

Note:

We are experiencing an equipment malfunction while fabricating the test samples. We are looking into a couple of ways to address it. Unfortunately, this challenge will throw us off schedule. As discussed during October's Status meeting with PHMSA, I have contacted PHMSA to request an extension.