## EXPERIMENTAL STUDY OF ELEVATED TEMPERATURE COMPOSITE REPAIR MATERIALS TO GUIDE INTEGRITY DECISIONS

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## ABSTRACT

Over the past two decades, a significant amount of research has been conducted on the use of composite materials for the repair and reinforcement of pipelines. This has led to vast improvements in the quality of composite systems used for pipeline repair and has increased the range of applications for which they are viable solutions (including corrosion and mechanical damage). By using composite repair systems, pipeline operators are often able to restore the structural integrity of damaged pipelines to levels equal to or even in excess of the original undamaged pipe. Although this research has led to substantial advancements in the quality of these repair systems, there are still specific applications where questions remain regarding the strength, durability, and effectiveness of composite repair systems, especially in elevated temperature, harsh environment conditions.

This program initially involved composite repair systems from six manufacturers. The test group included both carbon and E-glass based systems. Performance based qualifications were used to reduce the size of the test group from the initial six systems down to three. The experimental study consisted of small-scale testing efforts that ranged from tensile tests performed over a range of temperatures to 10,000-hour material coupon tests at elevated temperatures. The elevated temperatures used for testing were intentionally selected by the operator to reflect the 248 °F design temperature of the target pipeline.

Using small-scale qualification testing outlined in ASME PCC-2 - Repair of Pressure Equipment and Piping standard (Article 4.1, Nonmetallic Composite Repair Systems: High-Risk Applications) as a foundation, the test program described in this paper was able to demonstrate that, when properly designed, and installed, some composite materials are able to maintain their effectiveness at high temperatures. This study combined short-term and long-term testing of composite systems and demonstrated the advantages of a 10,000 hour test when aging properties are unknown. Finally, the study showed that, although high-temperature reinforcement using composite repair systems is feasible and commercially available, this capability is not standard practice across the composite repair industry. Proper analysis and verification using experimental methods, including full scale testing should be conducted prior to installation of a composite repair system in these types of harsh conditions.

## INTRODUCTION

The tasks described in this test program attempt to address questions regarding the use of six prospective composite repair systems in an elevated temperature, harsh environment condition. While industry used de-rating factors are currently available for composite reinforcement systems, the critical nature of the application warranted a more rigorous qualification of capable repair systems. It is yet to be determined if industry de-rating factors are appropriately conservative for composite repair systems with performance testing conducted at temperatures below the final operating temperature. In order to qualify potential composite repair systems for such an application, a series of elevated temperature material tests were performed. These tests included tensile performance and 10,000-hour coupon tests at elevated temperatures.

The six initial composite repair systems considered in this study included both carbon fiber and E-glass systems. The distribution of carbon and E-glass systems are listed below according to manufacturer:

- SystemA E-glass
- System B E-glass
- System C E-glass
- System D Carbon
- System E Carbon
- System F E-glass

The intent from the outset of the test program was to use performance based criteria to eliminate underperforming systems prior to beginning more costly aspects of testing. Later stages of the assessment included full-scale, elevated temperature tests which allowed for a more in-depth analysis of the performance of selected repairs. [1] The methodology and benchmarks used in analyzing these systems is outlined in subsequent sections.

## **TEST METHODS**

Three primary small-scale tests were conducted as part of the test program beginning with short-term tensile tests at elevated temperature. 10,000-hour coupon tests and load transfer material compression testing followed tensile testing and only considered three of the original six systems. Glass transition temperatures (Tg) were also determined for the three selected systems.

#### **Tensile Coupons**

Sub-scale, composite panel coupon tensile testing was completed on six candidate pipe-repair systems. The test panels were provided by each of the composite repair manufacturers using composite materials for their respective systems. This testing was completed at six temperatures ranging from 80.6 °F to 284 °F. The results were used to determine modulus and ultimate tensile strength for each respective system.

The test systems were delivered as flat plates produced by each manufacturer, approximately 12 inches by 12 inches in size. Thicknesses ranged from approximately 0.06 inch to 0.20 inch. Tabs were applied to each side of the sample plate to increase the thickness of the grip section of the samples. Both the tabbing material and the sample material were lightly sandblasted before epoxy was applied to roughen the surface and improve adhesion. The gauge section of the samples was masked from the sandblasting. Epoxy was used to fillet the transition from the tab to the gauge length.

The tabbed samples were assembled using a mold to position the glass-fiber tabs, set the epoxy thickness, and mold the fillet transition into the epoxy. The mold was clamped shut and cured at 284 °F for 4 hours. The molds were then separated, and the tabbed sample plate was post-cured at 284 °F for an additional 20 hours.

After curing, the sample plate was trimmed and then cut into 1-inch wide test specimens using a fence and table saw with a diamond grit blade. Sample dimensions were checked to ensure that the sample sides and tabs were straight and parallel. All samples were conditioned for at least 2 hours at controlled temperature and humidity.

All tests were conducted on an electro-mechanical load frame with a maximum load capacity of 22,000 pounds. An environmental chamber was placed in the load frame that completely enclosed the sample and grips. For the higher strength materials, a localized heater was used. Sample elongation was measured using an extensometer with a 2-inch gauge length. Samples were tested at a constant crosshead rate of 0.1 inch/min or 0.2 inch/min in order to achieve an average strain rate of 0.01-0.02 inch/inch/min.

## 10,000-hour Creep-rupture Testing

Three candidate composite repair systems were subjected to 10,000-hour creep-rupture testing at 248 °F. The test applied a constant load generated by a level-multiplied dead weight until the sample failed or was shut down and deemed a run-out.

The creep test samples were tabbed in a fashion similar to the short-term tensile test samples. Variations from that procedure included:

- 1. The sample plates were cut into 0.375-inch width dogbone style test specimens using a water-jet cutting process. The samples were tabbed after cutting to ensure the samples were aligned properly. The same material from the short-term tensile testing was used.
- 2. A single hole was drilled into each tabbed end and was used for one of the three bolts used to clamp the grip at each end for loading into the creep frame. Another hole in each clamping plate was attached with a pin to rod ends on the creep frame.
- 3. Steel shims were placed on either side of the tabbed ends to create enough space to attach the sample to the rod ends on the creep frames. These shims were also extended past the edge of the glass-fiber tab, such that the step prevented the tab from sliding relative to the shim.
- 4. Localized heater plates were attached to each side of the gauge length. A small groove was cut into each aluminum plate on the sample-side face for a

thermocouple to be placed against the composite sample surface. The heater plates were held against the sample surface by small springs to maintain contact.

All tests were conducted using Satec M3 lever-arm load frames with a maximum load capacity of 6,000 lbs on each sample (Figure 1). The load is generated by hanging steel weights from a lever arm with a 16:1 multiplication factor. The maximum load limit determined the gauge width required for the samples, as full 1-inch specimens of System D and System E would have required loads far exceeding the machine's capabilities.

Standard tensile tests were run on the dogbone-style specimens to ensure that the reduced-width gauge length did not adversely affect the observed failure characteristics or ultimate stress. No effect was observed for three tested systems.

Each sample was assembled with the required shims and clamping plates, then loaded into the creep frame. The sample was preheated to 248 °F for 15 minutes before being loaded.

#### Load Transfer Material Compression Testing

The load transfer material compression testing was performed using the same three candidate systems that were used in 10,000-hour creep-rupture testing. The intent of this phase was to identify the strength and modulus of the load transfer materials as a function of temperature.

The load transfer material compression test specimens were prepared by the respective manufacturers at their facilities and then provided to Stress Engineering Services (SES) for testing. Dimensions of the specimens were kept consistent through the use of 1-inch NPS PVC pipe molds that were provided to the manufacturers by SES. Five samples from each manufacturer were prepared and tested.

Nominally, the samples were molded to be 1 inch in diameter and 2 inches in length. Slight variations from these dimensions were noted and were generally attributed to differences in the load transfer materials themselves and the installation methods used by the manufacturers. The consistency between samples varied significantly according to manufacturer.

The samples were heated to a temperature of 266 °F before being subjected to compressive loading. The desired temperature was achieved by placing the samples in an oven at the test temperature for 30 minutes before removing them and placing them in an insulated test box that encapsulated the test region. Resistance heating units were used to keep the temperature of the test box near 266 °F. A photograph of the test set-up including the insulated box and controller used to maintain the test temperature is shown in Figure 2. The compressive load was applied a rate that did not exceed 0.5 inch/minute.

#### RESULTS

Results are presented for the tensile coupons, 10,000-hour creep-rupture testing, and load transfer material compressions testing. Glass transition temperatures for three of the systems were measured from repairs installed on full-scale pipe samples in a separate study. These Tg values are given in Table 1.

## **Tensile Coupons**

Overall, clean gauge-length failures were observed for all systems. Every system tested exhibited a highly linear stress/strain response, though fiber breakage was common during testing. All six systems exhibited some modulus loss at elevated temperatures, ranging from 5% to 25% at 212 °F. The systems exhibited a wide range of ultimate strength reductions over the temperature range, from 0% (no reduction) to nearly 60% at 284 °F. Figure 3A summarizes the results of the tensile testing. It can be seen that Systems D, E, and F exhibited the lowest reduction in ultimate strength over the course of the temperature range. This pattern of performance can also be seen in follow-up full-scale testing that was performed. [1]

## 10,000-hour Creep-rupture Testing

The initial goal of the creep rupture study was to meet the requirements of ASTM D2992 testing as specified in ASME PCC-2 Article 4.1, Section V-2.3. This requires a distribution of failure times as shown in Table 2.

It was discovered as testing began that achieving failure times in the 10 to 1,000 hour range, and to a lesser extent the 1,000 to 6,000 hour range, would require the application of loads approaching the short-term (i.e., instantaneous) ultimate tensile strengths of the System E and System F samples. This led to a large number of samples breaking immediately upon loading, as the required loads were near the measured ultimate strength for these systems. The System D samples exhibited less of this type of behavior, and the ASTM D2992 requirements were met for that particular system.

Discoloration was observed after the long-term creeprupture tests on all three systems. This discoloration is believed to have been caused by the epoxy/resin components of the composites, and was most pronounced in Systems D and F. System F developed a very dark discoloration; System D, and to a lesser extent System E, developed reddish discolorations. It should be noted that the operator accepted the fewer than 18 data points required by ASTM D2292 (as noted in Table 2) for System E and System F, based on their exceptional performance due to the runout conditions that were achieved. A conscious decision was made to accept fewer points; the absence of data should not be interpreted as an incomplete test. System D was the only system that was able to achieve the required failure times to complete the D2992 test requirements. Creep-rupture results and corresponding 95% confidence curves are shown in

Figure 3B. It is noteworthy that System D showed a significantly higher regression slope than the other systems. This is surprising because it is a carbon-fiber system and the industry standard expectation is that carbon-fiber composites will not exhibit significant loss of strength in creep. Figure 3B summarizes the results of the System D creep-rupture testing. A photograph of a post-failure sample is shown in Figure 4.

System E exhibited the highest moduli and ultimate stresses of all the tested systems. The samples exhibited exceptional creep-rupture performance as indicated in Figure 3B. A photograph of a post-failure sample is shown in Figure 5.

System F exhibited good creep-rupture performance as indicated in Figure 3B. Most samples became "run-outs," and were shut down after 10,000 hours. Some poorly performing plates of material were discovered that failed at loads of only 65% of the ultimate tensile strength (as measured by previous tensile testing). It is believed that these plates of material comprised lower resin content than the other sheets, as they exhibited unusual delamination of the entire gauge length face upon failure (Figure 6). Replacement sheets of material were provided by the manufacturer and these unusual failures were not observed with the new samples. A photograph of a postfailure sample is shown in Figure 7.

#### Load Transfer Material Compression Testing

The results from the compression tests are summarized in Table 3. Table 3 shows the modulus for each sample as determined from the compression testing. To determine the modulus of the load transfer materials, raw data from each compression sample was reviewed and two representative points on the stress vs. strain curve were selected. The slope of these two points represents the calculated compressive modulus of the filler materials.

#### CONCLUSIONS

The experimental testing outlined in this paper provides a basis for early-stage decision making when selecting composite repair systems for elevated temperature applications. The small-scale testing methods described are significantly more economical than full-scale testing and allow for a number of systems to be screened and selected based on material performance. This includes long-term (10,000 hour) testing of composite materials, which remains a prudent criterion for assessing their capability for integrity applications. In the case of this particular test program, the measurement and calculation of temperature-time-regression lines provided a fundamental basis for long-term extrapolation of failure stress in the materials tested.

Additional full-scale testing for the specific intended application using the material performance information is a good practice and is described in a related paper.

### DISCUSSION

The small scale testing methodology applied here conducted on various composite wrap repair systems provides a targeted method for low-cost screening of candidate materials for an operator. By comparative ranking of systems in terms of % performance loss at temperature, a large number of repair systems were effectively screened and only a small number advanced to the more costly stages of full-scale or long-term testing.

The long-term creep testing conducted here illustrates the need for long-term tests at actual maximum operating temperature. This requirement is a standard requirement among reinforced (RTP) pipe manufacturers and can be considered by operators as a prudent measure for composite wrap repair materials as well. Two important conclusions were drawn from the long-term test at temperature: (1) one carbon fiber system showed a time-dependent aging mechanism that was unexpected based on literature values and industry consensus, and (2) the long-term elevated temperature properties of each system could not have been accurately predicted using temperature scaling factors calculated from short-term tensile testing at elevated temperature.

Both observations provide good examples of the need for material-specific testing at the intended operating temperature when new systems are being developed. The first observation shows that a totally unexpected degradation mechanism was identified as a result of this testing. This mechanism did not compromise the long-term integrity of the repair, but it did lower the long-term allowable strain applied to the system in question.

The second observation came about because of a change in the slope of the creep-rupture curve at elevated temperature. This change in slope indicates an increase in rate of damage accumulation and not just a decrease in overall strain to failure. Because the increase in damage accumulation rate cannot be predicted by short-term elevated temperature tests, it can be seen that long-term testing is advisable in this case. A lessrigorous approach would have been to conduct short-term elevated temperature testing and apply a temperature derating factor to the long-term allowable strain at ambient. This approach would not have captured the change in damage accumulation rate (degradation slope) and would have underestimated the effect of increased temperature on performance.

Load transfer material testing did not have an immediate bearing on the outcome of the testing program, but proved very useful in later stages of testing. The compressive properties of the load transfer have been useful in follow-up FEA models based on this work, and have also served as benchmark properties when considering changes to load transfer material. For the relatively small characterization cost associated with compressive testing of the load transfer material, this was a worthwhile addition to the testing program.

### REFERENCES

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Figure 1: M3 Creep Frame / Loaded creep-rupture sample

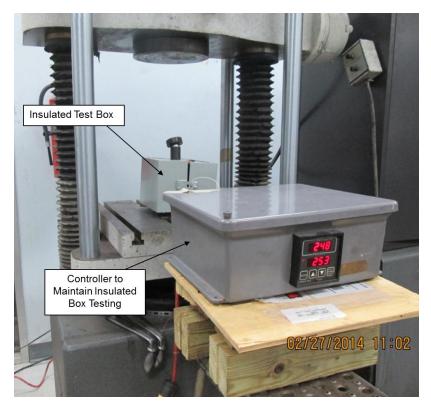


Figure 2: Load transfer material compression test set-up

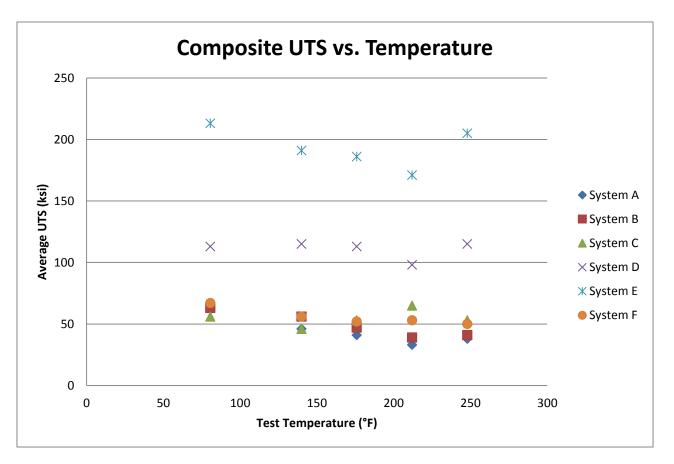
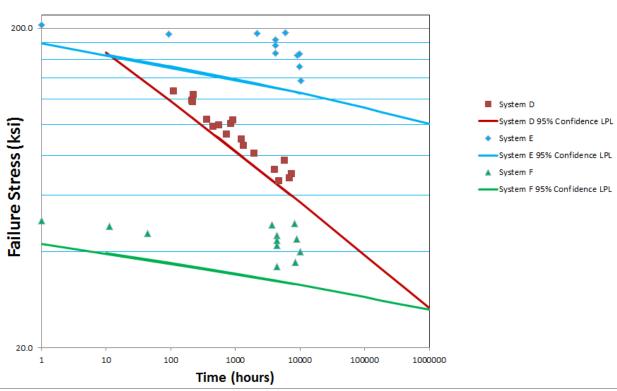


Figure 3A: Ultimate Tensile Test Results as a Function of Temperature



# Comparison of 120C Creep Testing Multiple Composite Repair Systems

Figure 3B: Creep rupture results

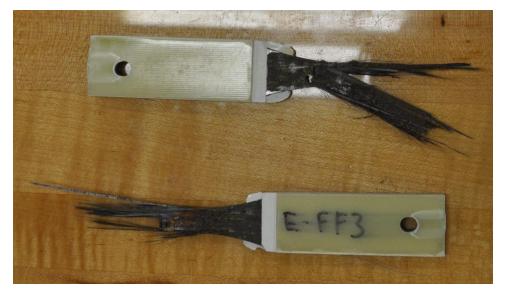


Figure 4: System D creep-rupture specimen – post failure

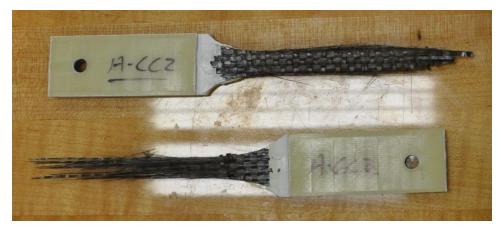


Figure 5: System E creep-rupture specimen – post failure

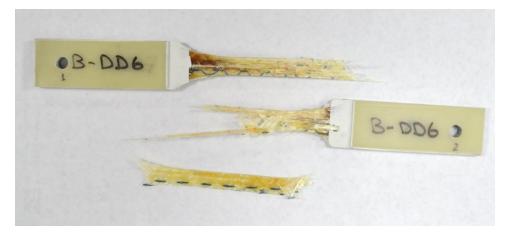


Figure 6: System F sample that failed at 65% of UIS. Large delamination suggests low epoxy content.



Figure 7: System F creep-rupture – post-failure

Repair System	Glass Transition Temperature, Tg (°F)	
System D	287.6	
System E	478.4	
System F	327.2	

Table 1: Measured glass transition temperatures, Tg for the three selected systems

Table 2: Test requirements of ASTM D2992, PCC-2 Article 4.1, Section V-2.3

Failure Time Range (hours)	Number of Tests Required	
10-1,000	4	
1,000-6,000	3	
6,000+	2	
10,000+	1	

Table 3: Results of load transfer material compression testing	
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	System D	System E	System F
	Calculated Modulus	Calculated Modulus	Calculated Modulus
	(psi)	(psi)	(psi)
Sample 1	9,914	177,732	20,316
Sample 2	12,426	232,039	54,101
Sample 3	13,285	234,626	12,979
Sample 4	15,143	209,267	30,282
Sample 5	9,574	172,817	14,792
Average	12,068	205,296	26,494