Can It Take the Heat: Analyzing the Behavior and Performance of Composite Repair Systems in Elevated Temperature Environments

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Abstract

Pipelines that operate at elevated temperatures present unique challenges to composite repair applications. The material performance of a composite repair is impacted both by its cure temperature and the operating temperature. These variations collectively influence the material mechanical properties of the applied composite system.

The first question explored in this paper is the degree to which the properties of a composite system, specifically the tensile strength and elastic modulus, change at various elevated temperatures compared to ambient temperatures. The second question explored in this paper is how the cure temperature of the composite affects the tensile strength and elastic modulus at various temperatures. An elevated temperature post-cure will increase the crosslink density of the polymer, which should result in a stronger, stiffer cured material. Increased crosslink density will also likely affect the elevated temperature properties of the composite repair system.

The testing program referenced in this paper developed a protocol to evaluate the abovementioned questions. Two commercially available, carbon fiber-based composite repair systems were tested. Each system was tested for tensile strength and modulus at various temperatures, ranging from 70 °F – 210 °F. All samples were analyzed based on tensile strength, tensile modulus, and glass transition temperature (Tg). The full test protocol was performed on both composite repair systems at two different cure temperatures. Ultimately, the intent of this testing program was to evaluate how the properties of composite repair systems change at elevated temperatures and to explore how variations in a composite's cure temperature affect these properties.

Introduction & Background

While established standards like ASME PCC-2 and ISO 24817 offer robust guidelines for general composite repairs, they lack specific recommendations for high-risk composite repairs that operate at elevated temperature environments. This gap underscores the need for further investigation into how composite materials must be cured and how they behave when exposed to elevated operating temperatures. Epoxies are the polymers commonly used for structural reinforcement with fiber reinforced polymers. This class of polymer generally benefits from elevated cure temperatures to maximize the cross-link density and relieve internally strained polymer configurations. The degree of crosslinking achieved during the curing process has a direct impact on the composite's mechanical properties. This is especially true in epoxy systems with a very high cross-link capacity, like many of the reinforcement systems marketed for high-temperature applications.

This study examines the impact of introducing a "post-cure" period on two composite repair systems that use different epoxy resins. To do this, coupons of composite repair panels prepared by Advanced FRP were subjected to tensile loading. Using the data collected from these tests, material properties could be calculated to better understand the impact of elevated curing and operational temperatures on composite repair performance.

Several key material properties were examined. Tensile modulus, tensile strength, and the glass transition temperature (Tg) were key focus areas of the testing procedure.

1. Tensile modulus is particularly important because it reflects the composite's stiffness and its ability to respond quickly to loading, which is critical for reinforcing defects like cracks

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that require immediate structural support. A higher modulus system can better withstand and distribute loads, offering enhanced performance in dynamic service conditions.

2. Tensile strength, on the other hand, represents the overall load-bearing capacity of the composite. Understanding how post-curing affects this property can have significant implications for the cure requirements and overall performance of composite repairs for higher-temperature applications.

3. Lastly, the Tg, or glass transition temperature, is a critical temperature range where the polymer matrix transitions from a rigid to a rubbery state. This property is vital for high temperature reinforcement systems, as it directly influences the composite’s tensile modulus and strength, with a sharp decline in both properties expected once the Tg is exceeded.

This paper reports the impact of multiple elevated temperature scenarios on composite repair mechanical properties, based on the results of the tensile testing protocol. The composite panels in this test program were subjected to controlled curing and post-curing processes, with a focus on the impact of a 150 °F post-cure condition. This temperature was selected for its relevance to typical field conditions. The panels were evaluated for key properties to determine if post-curing at elevated temperatures could enhance the composite's overall performance.

Preparing Composite Coupons with Varying Curing Temperatures

Tensile testing was performed on a total of 85 tensile specimens prepared by Advanced FRP Systems according to ASTM D3039-17 “Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials”.

Specimens were created with a bi-directional carbon fiber reinforcing fabric and two different types of epoxies, denoted as 211 HT and 221 C. Panels were prepared by Advanced FRP, and initially cured at room temperature on a vacuum bagging table. Each panel was post-cured at one of two temperatures: room temperature or 150 °F (65.56 °C). The post-curing process occurred over 24 hours for all panels within +/-2 °F of the assigned curing temperature. Coupon strips 0.5 inches wide and 6 inches long were cut from the panels.

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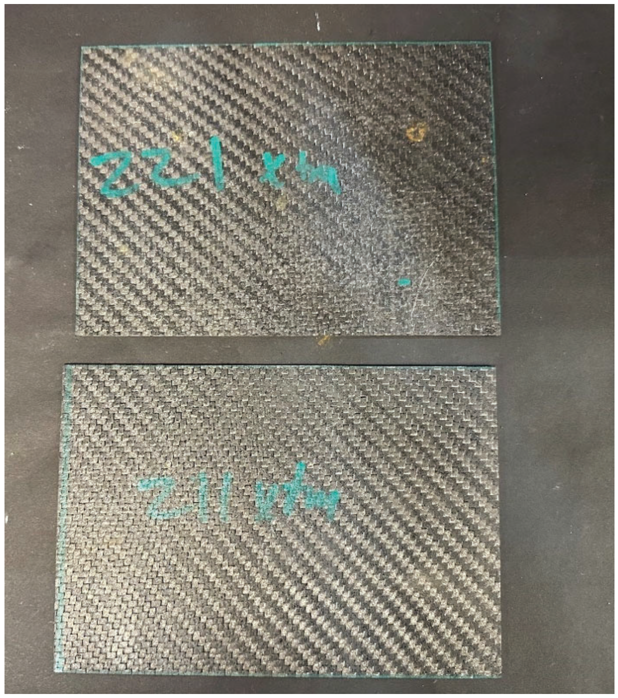

Figure 1: Prepared sample panels and specimens for tensile testing

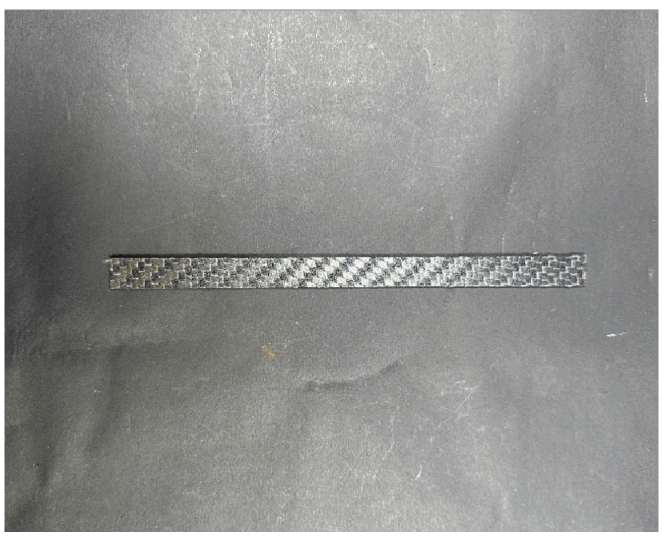

Figure 2: Prepared sample panels and specimens for tensile testing

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Table 1: Description of prepared testing specimens

Number of Specimens	Epoxy Resin Used	Curing Temperature
20	211 HT	Room Temperature
20	211 HT	150 °F (65.56 °C)
20	221 C	Room Temperature
25	221 C	150 °F (65.56 °C)

Tensile Testing of Coupons

Each configuration of resin and post-cure temperature was subjected to tensile testing at one of four temperatures: room temperature, 110 °F, 150 °F, and 180 °F, while the 221 C, 150 °F post-cure configuration had an additional test at 210 °F. Each test configuration of resin, post-cure temperature, and test temperature had a minimum of five (5) specimens tested.

Tensile loading was performed with a loading rate of 0.05 inches/min. Loading continued at the specified rate until tensile failure. Failure is defined as the point at which the measured load no longer increases or significantly decreases despite a continued increase in displacement.

From the data recorded from the tensile loading, the tensile strength and tensile modulus were calculated.

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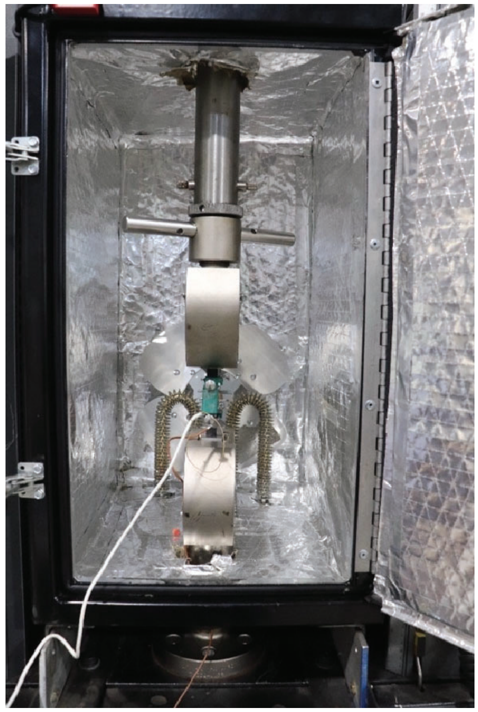

Figure 3: Elevated tensile testing set up

Glass Transition Temperature Testing of Coupons

The glass transition temperatures of each representative coupon were measured using the ASTM E1356-23 standard test method. A differential scanning calorimeter (DSC) was used with data collection devices capable of logging heat flow, temperature, and time. To minimize the effects of the heat during the DSC run, the glass transition temperature was taken on the first run.

Results

The average tensile strength (ksi), tensile strength standard deviation (ksi), average tensile modulus (Msi), and tensile modulus standard deviation (Msi) were calculated for each sample representing each resin, post-cure temperature, and test temperature combination.

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Table 2: Tensile strength and modulus results from tensile testing composite coupons

Resin	Post-cure Temperature (°F)	Test Temperature (°F)	Specimen Set ID	Average Tensile Strength (ksi)	Tensile Strength Standard Deviation (ksi)	Average Tensile Modulus (Msi)	Tensile Modulus Standard Deviation (Msi)
211 HT	Room Temperature	Room Temperature	211-RT-RT	137.3	12.78	9.91	0.84
		110	211-RT-110	120.3	8.96	9.65	0.54
		150	211-RT-150	108.5	3.87	9.77	0.78
		180	211-RT-180	101.1	6.67	6.63	0.95
	150	Room Temperature	211-150-RT	123.7	4.71	10.59	0.57
		110	211-150-110	101.7	10.12	9.03	0.65
		150	211-150-150	117.9	2.50	8.70	1.11
		180	211-150-180	122.3	5.24	8.30	0.62
221 C	Room Temperature	Room Temperature	221-RT-RT	146.8	7.77	10.90	1.24
		110	221-RT-110	120.3	8.96	9.65	0.54
		150	221-RT-150	121.3	2.98	9.55	0.84
		180	221-RT-180	84.2	3.64	6.73	0.62
	150	Room Temperature	221-150-RT	135.3	320	10.10	0.18
		110	221-150-110	129.7	4.93	9.73	0.63
		150	221-150-150	129.2	5.12	9.91	0.89
		180	221-150-180	102.02	6.82	9.67	0.95
		210	221-150-210	90.3	7.77	8.86	1.54

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The Tg, or glass transition temperatures of the four different configurations of resin and post-cure temperatures were recorded, as well. It is important to keep in mind that the glass transition temperature is not an absolute value. Rather, it is a temperature range where a phase change takes place in the polymer, from a rigid state to a more rubbery state. Each polymeric system has a maximum possible Tg based on full cross-linking of the system. In the field, these polymers rarely reach their maximum Tg. Instead, they operate at a lower Tg based on the cure temperature and time.

Table 3: Glass transition temperatures for each combination of resin and post-cure temperature

Resin	Post-cure Temperature (°F)	Glass Transition Temperature (°F)
211 HT	Room Temperature	137.0
	150	189.7
221 C	Room Temperature	186.9
	150	195.7

Tensile Strength & Test Temperature Results

For a better understanding of the relationship between the different variables in this testing protocol, tensile strength results were compared with the test temperature and glass transition temperature for each composite reinforcement option.

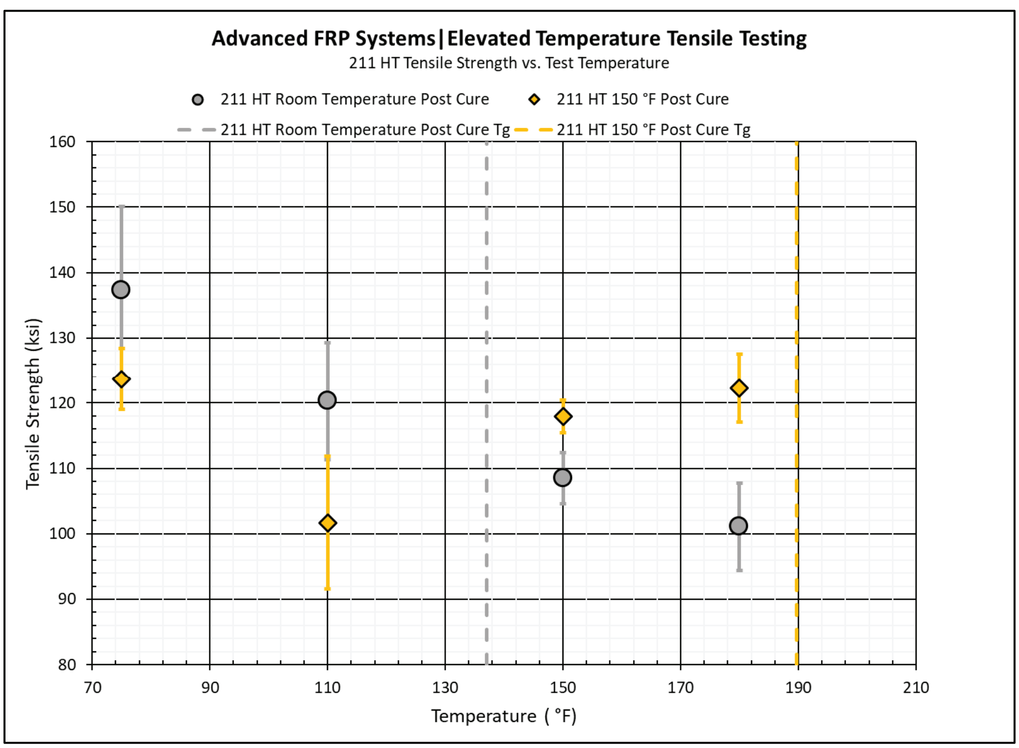

Figure 4: Elevated temperature tensile strength results for 211 HT composite samples

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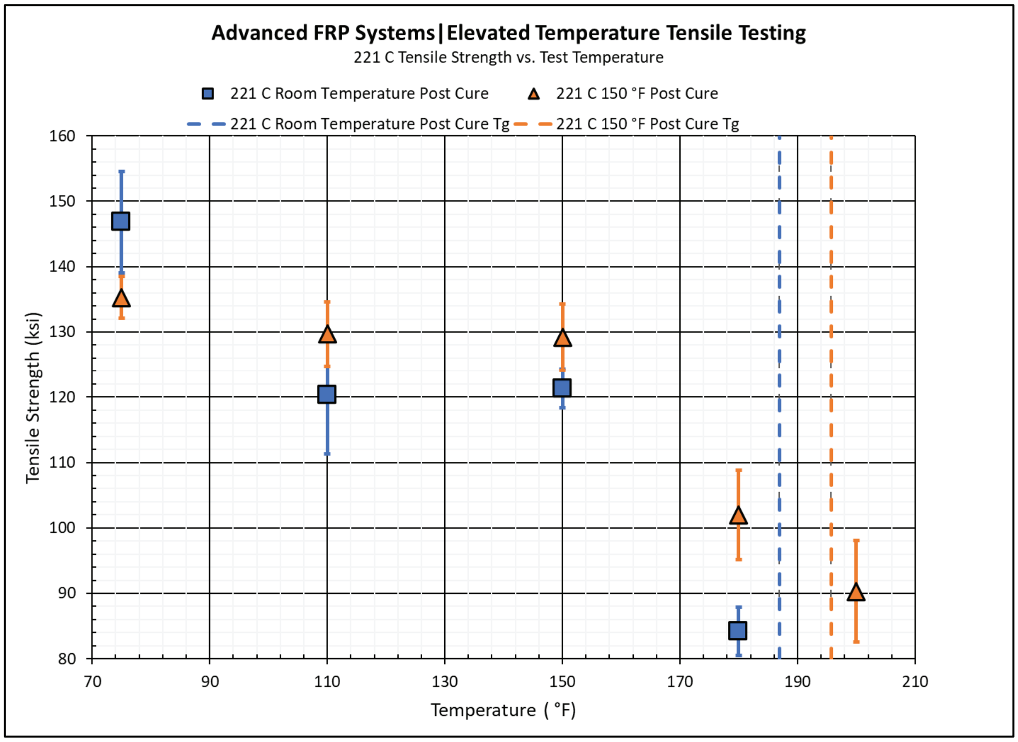

Figure 5: Elevated temperature tensile strength results for 221 C composite samples

As shown in the graphs, a post-cure at 150 °F consistently increased the tensile strength at the 150 °F and 180 °F test temperatures. The post-cure at an elevated temperature also consistently decreased the tensile strength at room temperature testing.

The 211 HT system, post-cured at 150 °F, showed an initial decrease in tensile strength, followed by a distinct increase at 150 °F and 180 °F. Conversely, the 211 HT system, without a post-cure, showed a fairly linear drop-off in tensile strength as the test temperature increased. The 221 C system showed a slow drop-off of tensile strength for both the ambient temperature and 150 °F post-cure systems until the test temperature reached 180 °F where they dropped off sharply.

Tensile Modulus & Test Temperature Results

The tensile modulus was also compared with the test temperature and the glass transition temperature for each resin.

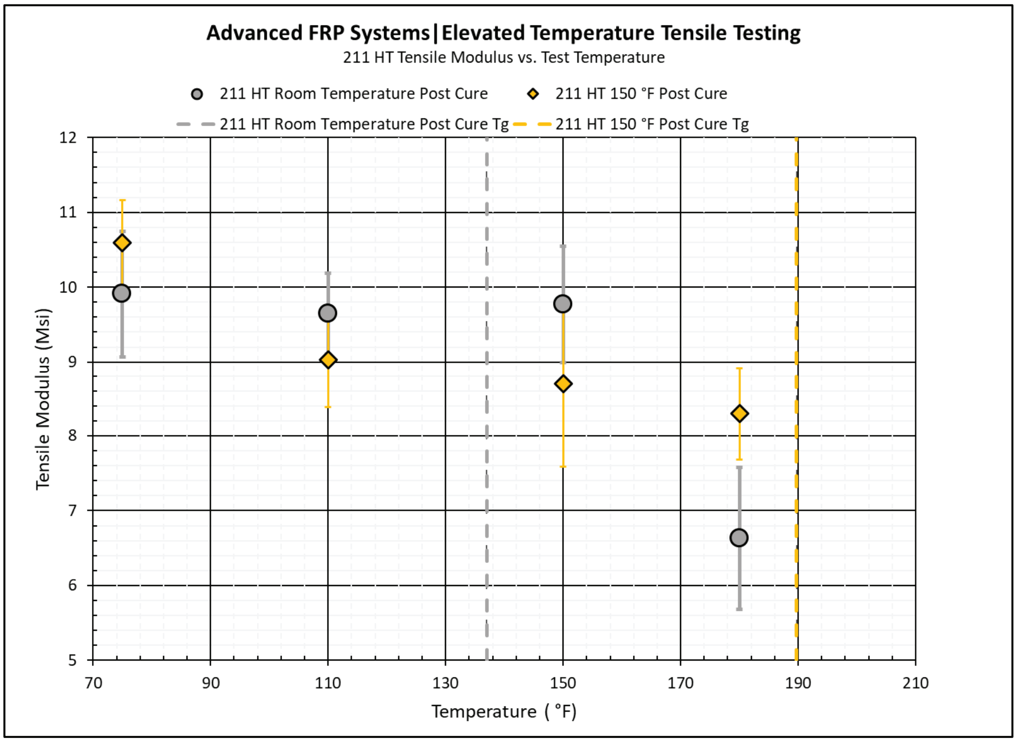

Figure 6: Elevated temperature tensile modulus results for 211 HT composite samples

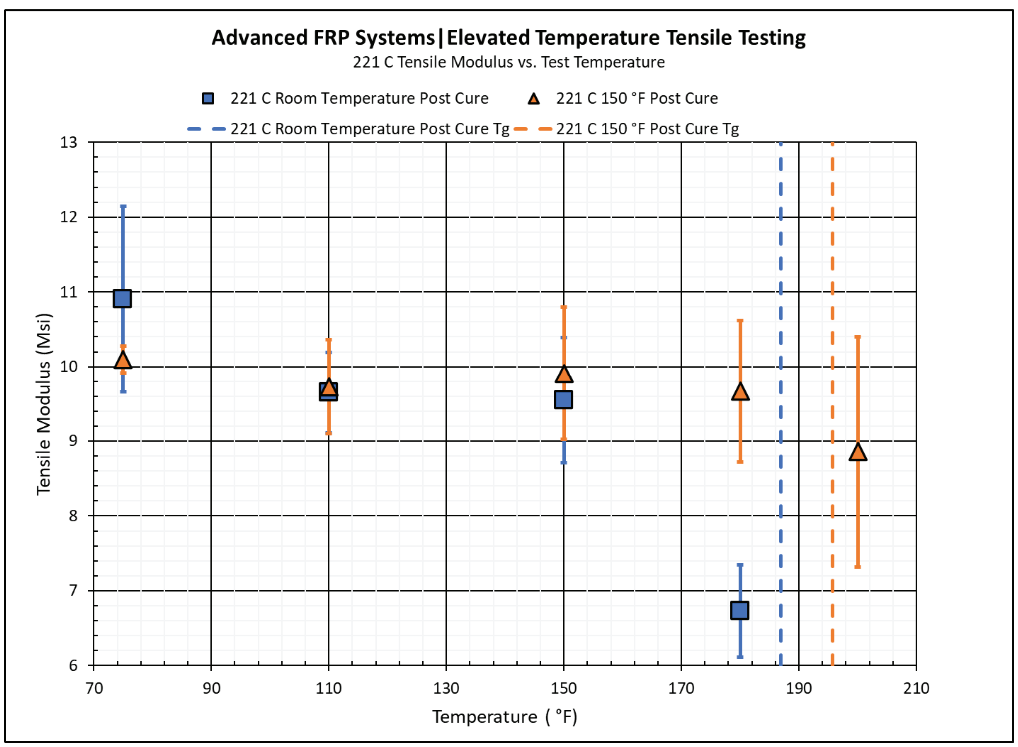

Figure 7: Elevated temperature tensile modulus results for 221 C composite samples

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As shown, a post cure at 150 °F consistently increased the tensile strength and tensile modulus at the test temperature of 180 °F.

Overall, the tensile modulus would be expected to trend downward as the test temperature is increased, then sharply decrease when the polymer begins to transition to a rubbery state at its glass transition temperature. All 4 systems showed this general trend, with the 211 HT dropping off sharply between 150 and 180 °F for the room temperature cured system. The 211 HT with a 150 °F cure most likely never reached its Tg and experienced a gradual reduction in modulus. The FRP 221 C with a room temperature cure showed a fairly consistent modulus until it passed 150 °F. The FRP 221 C with a 150 °F post-cure held steady until it exceeded 180 °F with only a slight decrease in tensile modulus even at 210 °F.

Conclusion

The results shared in this paper do not demonstrate distinct trends that shed light on how fiber reinforced composites behave at elevated temperatures. The results clearly show that each system behaves differently and must be tested to understand how it will behave with a certain cure temperature and service temperature. The 211 HT composite with a room temperature cure showed a steady decrease in tensile strength as the test temperature rose, while the tensile modulus held steady until a sharp decline at 180 °F. Conversely, the 211 HT composite with a 150 °F post-cure showed an initial decrease in tensile strength followed by an increase. While the test temperature increased, the tensile modulus decreased steadily over the entire test range. The 221 C with a room temperature cure had an extremely high initial tensile strength, which decreased steadily as the test temperature increased. The tensile modulus decreased only slightly until the 180 °F test point where it decreased drastically. The 221 C with a 150 °F post-cure also showed a decrease in tensile strength as the test temperature increased, however it experienced a much slower rate of decrease. The tensile modulus decreased slightly and even up to 210 °F experienced no significant drop-off.

In summary, all 4 systems behaved differently, and any composite repair done at elevated temperatures should use the appropriate properties when calculating the number of layers required to provide the hoop and axial stress required for the reinforcement.

It is also fairly clear that using the glass transition temperature, Tg—measured by DSC—is not a good test for the actual loss of modulus associated with the samples during elevated temperature physical testing. The Tg of the 211 HT with a room temperature cure was measured at 137 °F by DSC. However, no reduction in tensile modulus was observed until the 180 °F test point. For the 221 C with a room temperature cure, a Tg of 186.9 °F was measured, but at the 180 °F test point, a distinct drop in the tensile modulus was observed. It is the author's opinion that glass transition temperature is of limited value for determining the maximum temperature of a composite reinforcement system. Rather, a graph of the tensile modulus versus temperature is a much more effective means of determining what temperature a composite system will no longer deliver the required level of reinforcement.

Results from a study funded by Chevron on the elevated temperature performance of composites were presented by Alexander et al at the International Pipeline Conference in 2016. These papers do not explore different curing conditions for the composite repairs, but they do explore both coupon-level and full-scale survival testing. The tensile strength testing, performed from room temperature up to 250 °F, showed results similar to those presented in this paper. Specifically, the mechanical properties of some systems decreased with increasing temperatures, while others increased in tensile strength at elevated temperatures. When properly cured, several of the tested systems performed well in a full-scale pipe spool test, demonstrating that coupon testing does correlate to full-scale pressure testing.

Overall, while the trends identified in this paper do not help predict the behavior of fiber reinforced polymeric reinforcement systems in general, it should inform future testing protocols to better understand how composite systems behave in elevated temperature applications. It clearly shows the critical relationship between cure temperature and the physical properties of the composite system. Furthermore, it demonstrates that glass transition temperature, or Tg, is not sufficient to predict the properties of polymeric reinforcement systems at elevated temperatures.

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