Freeze–Thaw Resistance of Fiber-Reinforced Polymer Composites Adhesive Bonds with Underwater Curing Epoxy

Roberto Lopez-Anido, M.ASCE[1]; Antonis P. Michael, S.M.ASCE[2]; and Thomas C. Sandford, M.ASCE[3]

Abstract: A prefabricated fiber-reinforced polymer (FRP) composite shield or jacket was developed to repair wood piles in the field. Cylindrical E-glass/vinyl ester composite shells were bonded underwater to encase damaged wood piles. The resistance to freeze–thaw cycles of an underwater curing epoxy adhesive was evaluated. The standard test procedure for single-lap shear adhesion for FRP composite bonding, ASTM D5868, was selected. A standard procedure for freeze–thaw cycling exposure was adopted. The effect of freeze–thaw cycling exposure on the performance of the adhesive bond was discriminated by comparing the lap shear strength and the mode of failure of control and exposed samples. It was found that the lap shear strength is substantially reduced by exposure to freezing and thawing cycles. A change in the mode of failure from predominantly adhesive type to combined adhesive/cohesive type was noticed after exposure.

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Introduction

A novel method developed for protection and structural restoration of wood piles requires field installation of fiber-reinforced polymer (FRP) composite prefabricated shells around the piles (Lopez-Anido et al. 2004b). During field placement, FRP composite shells need to be attached with an underwater curing adhesive that produces a satisfactory structural bond. The adhesively bonded shells need to develop "composite action" to serve as a load bearing structural shield or jacket. In cold-weather coastal regions, the main concern for durability of the adhesive bond between the shells is resistance to freeze–thaw cycles.

A procedure for exposure to freeze–thaw cycling of FRP composites bonded to concrete and masonry substrates was developed by the International Conference of Building Officials (ICBO) Evaluation Service as part of an acceptance criteria (ICBO 2001). This procedure was applied in literature to evaluate freeze–thaw exposure of four different FRP composite materials including an E-glass/vinyl ester composites fabricated by the vacuum assisted resin transfer molding (VARTM) process (Lopez-Anido et al. 2001). In this reference, standard tensile tests and short-beam shear tests were used to evaluate residual mechanical properties after freeze–thaw exposure, and the corresponding material capacity reduction factors were reported.

In the present study, E-glass/vinyl ester composite plates that are representative of the shell material used for wood pile restoration (Lopez-Anido et al. 2003) were fabricated using a variation of the VARTM process, the licensed Seemann composites resin infusion molding process (SCRIMP) (TPI Technology 2001). To assess adhesive bond durability, the standard test procedure for lap shear adhesion for FRP composite bonding, ASTM D5868 (ASTM 1995) was selected. This procedure is based on a single-lap shear test configuration. An underwater curing epoxy adhesive was utilized, and the standard procedure for freeze–thaw cycling exposure (ICBO 2001) was adopted.

The objective of this technical note is to investigate the performance of an FRP composite material used for wood pile repair bonded with underwater curing epoxy after exposure to freezing and thawing cycles. The effect of freeze–thaw cycling exposure on the performance of the adhesive bond was discriminated by comparing the lap shear strength and the mode of failure of control and exposed samples.

Materials and Methods

Composites Fabrication

The E-glass fiber reinforcement selected was unidirectional woven fabric and chopped strand mat (CSM). The criteria for material selection and fiber layup was presented elsewhere (Lopez-Anido et al. 2004b). The fiber architecture of the plates was: [CSM/0/90/0/0/CSM], which corresponds to the actual FRP composite shells used for wood pile restoration. One CSM layer was added on each surface of the laminate to improve bonding

[1]Associate Professor, Dept. of Civil and Environmental Engineering and Advanced Engineered Wood Composites Center, Univ. of Maine, Orono, ME 04469-5711 (corresponding author). E-mail: rla@maine.edu

[2]Graduate Research Assistant, Civil and Coastal Engineering Dept., Univ. of Florida, Gainesville, FL 32611-6580.

[3]Associate Professor, Dept. of Civil and Environmental Engineering, Univ. of Maine, Orono, ME 04469-5711.

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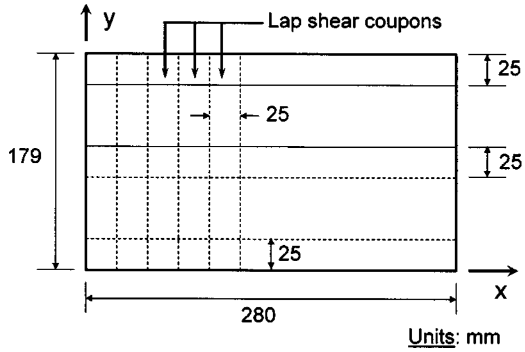

Fig. 1. Schematic of adhesively bonded fiber-reinforced polymer composite plates

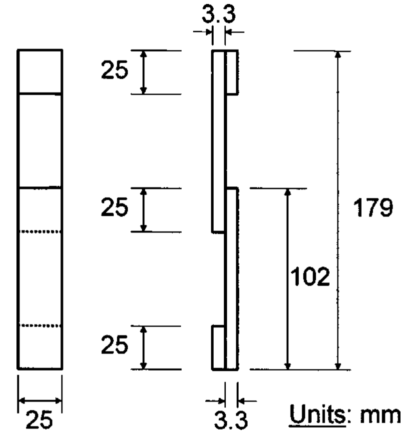

Fig. 2. Schematic of single-lap shear test sample

properties and to create a resin rich area that may provide environmental protection. An epoxy-based vinyl ester resin was selected as the matrix for the composite shells (Dow Chemical Co. 1999).

Using the VARTM/SCRIMP process (TPI Technology 2001), reinforcement layers were placed dry on a steel base mold and then sealed with a vacuum bag. A vacuum pressure of 102 kPa was applied using a vacuum pump. The applied vacuum pressure not only debulked (compacted) the dry fiber reinforcement, but also removed the entrapped air from the fiber layup. Once the required vacuum level was attained, resin was infused through a system of resin feed lines, flow distribution media, and vacuum lines. The pressure differential between the atmosphere and the applied vacuum allowed infusion of the resin into the fiber layup. After the resin impregnated the fiber reinforcement, the vacuum pressure was reduced to 51 kPa until the resin gelled. Once the resin gelled, the vacuum pressure was removed and the composite part was allowed to cure. From the manufactured FRP composite plates, plates of 280 mm×102 mm were cut using a precision wafering machine.

Adhesive Bonding

The FRP composite plate surfaces were wetted with tap water and then bonded together using an underwater curing epoxy adhesive (Superior Polymer 2000). This epoxy adhesive is specified for applications with a water temperature of at least 5°C. The epoxy adhesive was applied on one plate and then another plate was placed over the covered area creating an overlap of 25 mm. At the two edges, 25 mm wide strips, cut from the FRP composite plates, were bonded to align the bondline with the midplane of the sample for testing (see Fig. 1). To simulate actual field conditions, no preparation or cleaning of the plate surfaces was done prior to the application of the adhesive.

Underwater Conditioning

The bonded FRP composite plates were placed in a tap water bath at a temperature of 38°C with an uncertainty of ±0.5°C to allow the epoxy adhesive curing in an underwater environment. Water was heated by one submersible 250-W heater and circulation was provided by a 4-liter/min pump. The temperature was checked periodically with an electronic thermometer. The control bonded plates were removed from the water bath after 14 days, while the bonded plates used for the freeze–thaw exposure were left for additional 7 days to complete the 3-week conditioning period required by the acceptance criteria (ICBO 2001).

Freeze–Thaw Exposure

The freeze–thaw exposure specified in the acceptance criteria (ICBO 2001) requires 20 cycles consisting of a minimum of 4 h in the freezer and a minimum of 12 h in 100% relative humidity chamber. Based on the requirement of 20 cycles, a repeatable daily schedule (i.e., 24 h cycle) was adopted as follows: 8 h in the freezer and 16 h in the hot-water immersion bath. The hot-water immersion bath was implemented in lieu of the 100% relative humidity chamber. Heaters were set to maintain the immersion bath at 38°C and the freezer was set to −18°C, as specified in the acceptance criteria (ICBO 2001). Before placing the plates in the freezer, a clean cloth was used to remove surface water.

Table 1. Single-Lap Shear Experimental Results for Control Samples

Sample	Width b (mm)	Length L (mm)	Overlap area A_b (mm^2)	Maximum load P (kN)	Shear strength S (MPa)	Mode of failure
1R	24.97	27.31	681.9	11.07	16.2	adhesive
2R	27.18	27.05	735.2	12.30	16.7	adhesive
3R	25.91	27.18	704.2	10.64	15.1	60% adhesive/40% cohesive
4R	24.89	26.42	657.6	10.93	16.6	adhesive
5R	24.71	26.42	652.8	10.31	15.8	90% adhesive/10% cohesive
6R	24.71	26.04	643.5	9.64	15.0	adhesive
7R	24.89	26.80	667.1	11.60	17.4	adhesive
8R	24.84	26.42	656.3	11.02	16.8	adhesive
9R	25.02	26.92	673.5	10.90	16.2	85% adhesive/15% cohesive

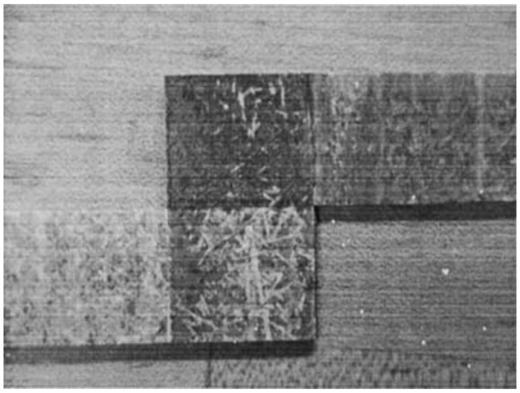

Fig. 3. Typical adhesive failure of control samples

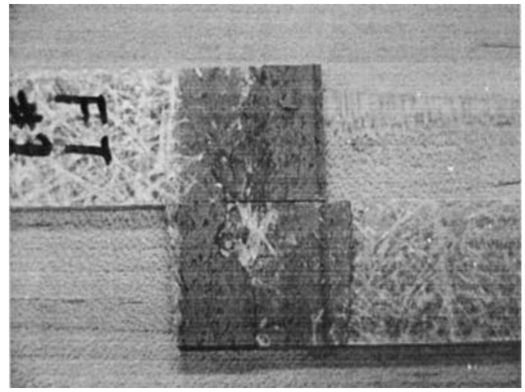

Fig. 4. Typical adhesive–cohesive failure of freeze–thaw exposed samples

Single-Lap Shear Test Evaluation

After conditioning and freeze–thaw exposure, the FRP composite bonded plates were cut into coupons according to ASTM D5868 (ASTM 1995) using a precision wafering machine. The lap shear test coupon dimensions were 179 mm in length by 25 mm in width as per ASTM D5868 (ASTM 1995) (see Fig. 2).

The lap shear tests were conducted using a 100-kN servohydraulic loading frame (Instron 1998) in a controlled ambient environment with a temperature of 22°C and a relative humidity of 45%. The samples were loaded in tension in displacement-controlled mode at a rate of 13 mm/min. The maximum applied load and the mode of failure were recorded.

The standard test method requires a minimum of five lap shear samples for each condition (ASTM 1995). A total of 18 lap shear samples were tested, 9 control and 9 exposed to freeze–thaw cycles. The scope of this study was restricted to discriminate the effect of freeze–thaw cycling exposure on the performance of the underwater curing epoxy adhesive selected for wood pile repair. Therefore, only a limited number of samples was tested to conduct comparisons between control and exposed bondlines. To develop quantitative data on residual shear strength, a larger number of samples need to be tested.

Results and Discussion

The standard practice for classifying adhesive failures in FRP composite bonded joints was applied (ASTM 1994). Adhesive (ADH) failure is defined as: "rupture of the adhesively bonded joint, such that the separation appears to be at the adhesive-adherend interface" (ASTM 1994). Cohesive (COH) failure is defined as: "rupture of an adhesively bonded joint, such that the separation is within the adhesive" (ASTM 1994).

Single-lap shear test results for control samples are presented in Table 1. The predominant mode of failure of the control coupons was ADH failure, with minimal or no COH failures. Only one lap shear control sample had a significant amount of COH failure. Typical ADH failure from one of the control samples is depicted in Fig. 3.

Experimental data from single-lap shear tests of freeze–thaw exposed samples are presented in Table 2. Most lap shear freeze–thaw exposed samples demonstrated an ADH mode of failure with a significant amount of COH failure. In some cases, COH failure accounted for 50% of the total overlap bonding area. A typical lap shear sample subjected to freeze–thaw cycles showing a combination of ADH and COH failure is depicted in Fig. 4.

The overlap bonding area, A_b, was calculated by multiplying the sample width, b, by the overlap length, L (see Tables 1 and 2). The apparent shear strength, S, of the ADH bond was determined by dividing the peak load, P, by the overlap area, A_b.

Comparative results for control and freeze–thaw exposed samples are shown in Table 3. The mean shear strength for the control samples was 16.2 MPa, while the respective value for the freeze–thaw samples was 9.2 MPa. Therefore, a reduction in the mean shear strength after 20 freeze–thaw cycles of approximately 43% was observed. Based on the coefficient of variation (COV), it was noticed that the test results had relatively low variability (e.g., COV of 4.9% for control samples and 2.4% for freeze–thaw exposed samples).

A statistical analysis of the apparent shear strength was performed using one-way analysis of variance (*ANOVA*) for the con-

Table 2. Single-Lap Shear Experimental Results for Freeze–Thaw Samples

Sample	Width b (mm)	Length L (mm)	Overlap area A_b (mm^2)	Peak load P (kN)	Shear strength S (MPa)	Mode of failure
1FT	24.89	26.42	657.6	6.07	9.2	adhesive
2FT	25.60	25.55	654.1	6.07	9.3	50% adhesive/50% cohesive
3FT	25.15	26.42	664.5	5.90	8.9	adhesive
4FT	25.27	26.16	661.1	6.04	9.1	80% adhesive/20% cohesive
5FT	25.65	25.27	648.2	6.19	9.6	60% adhesive/40% cohesive
6FT	25.48	26.67	679.6	6.34	9.3	50% adhesive/50% cohesive
7FT	24.94	26.16	652.4	5.71	8.7	80% adhesive/20% cohesive
8FT	25.32	26.62	674.0	6.20	9.2	90% adhesive/10% cohesive
9FT	25.35	25.83	654.8	6.08	9.3	80% adhesive/20% cohesive

Table 3. Single-Lap Shear Response After Freeze–Thaw Cycling Exposure

Condition	Control	Freeze–thaw exposed
Composite substrate	E-glass/vinyl ester	E-glass/vinyl ester
Adhesive	underwater epoxy	underwater epoxy
Conditioning	14-day water at 38°C	21-day water at 38°C
Exposure	not applicable	20 freeze–thaw cycles
Mean shear strength	16.2 MPa	9.2 MPa
Standard deviation	0.80 MPa	0.24 MPa
Coefficient of variation	4.9%	2.4%
Mode of failure	adhesive	adhesive/cohesive

trol and freeze–thaw exposed data sets. The analysis was conducted using the *SYSTAT* software package (SPSS 1999). The model for a one-way *ANOVA* (Dean and Voss 1999) is represented symbolically as follows:

$$Y_n = B_0 + B_1 \cdot X_n + \varepsilon_n \tag{1}$$

where Y_n=observed apparent shear strength for the data sets; B_0, B_1=coefficients of the model; X_n=code associated with the treatment under study (e.g., freeze–thaw exposure); and ε_n=random unit variation within the block of data.

The null hypothesis and alternative hypothesis are:

$$H_0 : B_1 = 0 \qquad H_A : B_1 \neq 0 \tag{2}$$

Pair-wise comparisons were performed with a confidence level of 95% ($\alpha = 0.05$). In order for the two data sets not to be significantly different, the p value, which is the probability of the coefficient B_1 to be zero, has to be greater than $\alpha (p > 0.05)$. In this study, the two sets are statistically different with a value $p = 0.000$.

It is hypothesized that the reduction in the bond shear strength is due to the presence of voids in the adhesive layer that facilitate water ingress. The void content in the adhesive layer is associated with the uneven spread of the adhesive on the FRP composite substrate combined with the lack of applied clamping pressure. During freezing, water expansion in the voids can generate crevices that in turn can degrade the epoxy adhesive bondline.

The retention of mean shear strength after freeze–thaw exposure was only 57%. However, the residual mean shear strength (9.2 MPa) is still adequate to transfer shear stresses between FRP composite shells in wood pile repair applications (Lopez-Anido et al. 2004a). It is worth noticing that in marine environments, where the adhesive layer is exposed to brackish or ocean water, the presence of salts may affect the epoxy curing reaction under water as well as the adhesive bond freeze–thaw durability. Furthermore, the effect of increasing the number of freezing and thawing cycles (beyond the standard 20-cycle exposure) on the performance of the underwater epoxy adhesive should be investigated.

Conclusions and Recommendations

The experimental study presented allows the following conclusions to be drawn:

1. The shear strength of the underwater curing epoxy studied is sensitive to freezing and thawing cycles.
2. Exposure to freeze–thaw cycles leads to a change in the mode of failure from predominantly adhesive type to combined adhesive/cohesive type.
3. The relatively low COV obtained in the experiments indicates that repeatability of the fabrication process, the testing protocol, and the shear strength measurement are satisfactory.

The following practical recommendations are proposed:

1. In field applications of FRP composite shells around wood piles, closely spaced straps can be used to increase clamping pressure and, therefore, reduce voids in the adhesive layer.
2. In the design of FRP composite bonded shells, a material capacity reduction factor for apparent shear strength needs to be introduced to account for the loss of strength that can occur during exposure to freeze–thaw cycles.

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References

ASTM. (1994). "ASTM D5573-94 standard practice for classifying failure modes in fiber-reinforced plastic (FRP) joints." American Society for Testing and Materials, West Conshohocken, Pa.

ASTM. (1995). "ASTM D5868-95 standard test method for lap shear adhesion for fiber-reinforced plastic (FRP) bonding." American Society for Testing and Materials, West Conshohocken, Pa.

Dean, A., and Voss, D. (1999). *Design and analysis of experiments*, Springer, New York.

Dow Chemical Co. (1999). *DERAKANE epoxy vinyl ester resins/chemical resistance and engineering guide*, Dow Chemical, Midland, Mich.

Instron. (1998). "Instron fast track 8802 materials test control system." *Rep. No. M21-10006-EN*, Instron, Canton, Mass.

International Conference of Building Officials (ICBO). (2001). "AC125—Acceptance criteria for concrete and reinforced and unreinforced masonry strengthening using fiber-reinforced polymer (FRP), composite systems." International Conference of Building Officials, Evaluation Service, Whittier, Calif.

Lopez-Anido, R., Harik, I., Dutta, P., and Shahrooz, B. (2001). "Field performance evaluation of multiple fiber-reinforced polymer bridge deck systems over existing girders—Phase I." *Rep. No. FHWA/HWY-2001/10*, Federal Highway Administration, Washington, D.C.

Lopez-Anido, R., Michael, A. P., and Sandford, T. C. (2003). "Experimental characterization of FRP composite-wood pile structural response by bending tests." *Mar. Struct.,* 16(4), 257–274.

Lopez-Anido, R., Michael, A. P., and Sandford, T. C. (2004a). "FRP composite-wood pile interface characterization by push-out tests." *J. Compos. Constr.,* in press.

Lopez-Anido, R., Michael, A. P., Sandford, T. C., and Goodell, B. (2004b). "Repair of wood piles using prefabricated FRP composite shells." *J. Perform. Constr. Facil.,* in press.

SPSS. (1999). "*SYSTAT 9*—Statistical analysis program." SPSS, Chicago, Ill.

Superior Polymer. (2000). "Technical data sheet: Hydrobond 500 underwater epoxy adhesive." Superior Polymer Products, Calumet, Mich.

TPI Technology. (2001). *An overview of the SCRIMP technology*. TPI Technology, Inc., Warren, R.I.