

Monitoring fungal degradation of E-glass/phenolic fiber reinforced polymer (FRP) composites used in wood reinforcement

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Abstract

The susceptibility of E-glass fiber reinforced polymer (FRP)/phenolic pultruded composite plates to fungal degradation was examined. Interlaminar shear strength (ILSS) by short-beam testing and ultrasonic non-destructive evaluation (NDE) techniques were applied to monitor fungal degradation of E-glass fiber reinforced polymer composites. Since the FRP material was designed for use as reinforcement with wood, the FRP material was exposed to two common wood decay fungi, a brown rot fungus and a white rot fungus. Light and scanning electron microscopy indicated that both wood decay fungi actively grew and penetrated into the FRP material, especially in high-void content areas. The reduction in apparent ILSS of the brown rot-exposed FRP material was not statistically significant at a 95% confident level. A weak relationship between decay exposure and ILSS strength loss, however, was observed. The experimental results indicate that both mechanical property evaluation techniques (ILSS and NDE) may be sensitive enough to detect the effects of fungal degradation in FRP materials.

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1. Introduction

Fiber reinforced polymer (FRP) composite materials are becoming increasingly accepted for use in the construction industry because they combine the advantages of both the fibers (typically E-glass) and the resin matrix (Lopez-Anido and Karbhari, 2000). These performance advantages include increased strength-to-weight ratio, hardness, wear and corrosion resistance, stiffness and improved creep behavior (Wagner et al., 1996; Mallick, 1993). As more applications are found for wood/FRP hybrids (e.g., laminated lumber for bridge applications, waterfront piers), their use in exterior and high-decay-hazard environments is expected to grow. Unfortunately, little information is available on the resistance of FRP composites to the activity of wood decay fungi

or the behavior of wood decay fungi in these composites. Since these reinforcements are used with wood, the mechanical strength and resistance to degradation of FRP material exposed to common wood decay fungi were evaluated in this experiment via qualitative (microscopic observation) and quantitative techniques (ultrasonic non-destructive evaluation (NDE) and mechanical testing).

It was only recently recognized that FRP composite materials were susceptible to biological attack (Gu et al., 1995a, b, 1996, 1997, 2000; Wagner et al., 1996; Thorp et al., 1994; Sampath et al., 1997; Sand, 1994). Gu et al. (1995a, b, 1996, 1997, 2000) reported that impurities and additives in FRP composites can promote fungal and bacterial growth and can serve as carbon and energy sources for these microorganisms. Furthermore, they concluded that biological damage to FRP composite materials may significantly affect their physical integrity and fatigue performance. Specific surfaces or voids in FRP materials may provide a place for nutrients to concentrate, thus providing a favorable

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microenvironment for microbial development. Fibers may serve as capillaries to improve the movement and distribution of moisture and chemical species within the material, and may enhance the spread of microorganisms along the fiber–matrix interface within the composite structure. Physical performance of composite material can be drastically affected by slight chemical changes in localized regions (Gu et al., 1995a, b, 1996, 1997, 2000; Wagner et al., 1996). Several mechanical and NDE (e.g., acoustic emission, electrochemical impedance spectroscopy (EIS)) techniques were previously examined for potential in evaluating the residual strength of FRP materials exposed to microorganisms. However, most of these techniques were found not sensitive enough to determine mechanical changes in the samples evaluated (Gu et al., 1995a, b, 1996, 1997, 2000; Wagner et al., 1996).

The application of ultrasonic techniques (acousto-ultrasonics) has been gaining popularity for non-destructive evaluation of various materials including FRP composites over the last decade (Beall et al., 1998; Emerson, 2000; Franklin et al., 2001). Ultrasonic methodology basically involves either the analysis of signals transmitted through materials along a fixed path to evaluate active changes in a material, or scanning the material to locate defective and weak areas (Beall, 1996).

In this work, an electronic pulser was used to generate repeatable elastic waves using a piezoelectric transducer. These waves propagate through FRP specimens and are received by a second transducer. The resulting waveforms can be recorded as voltage changes with subsequent waveform analysis performed using different techniques such as fast Fourier transform (FFT) to determine the changes in the signal content in the path. These changes result from microstructural modifications including potentially any internal flaws and loss of material integrity.

Mechanical characterization tests can directly measure strength properties, and therefore have had greater acceptance compared to NDE in material qualification for assessment of the action of degradative agents (UV exposure, weathering, chemical and thermal exposure, etc.) (Lopez-Anido and Wood, 2001; Prian and Barkatt, 1999). The standard test procedure “Interlaminar shear strength of FRP composites by short-beam method” (ASTM, 1997) is a widely accepted method for the determination of fiber/matrix interface characteristics (Mallick, 1993; Muszynski et al., 2000). The apparent interlaminar shear strength can be measured based on the short-beam test method according to ASTM D2344. In this method, a composite specimen with relatively small span-to-depth (l/d) ratio is tested in three-point bending to induce the interlaminar shear mode of failure. The apparent shear strength is computed assuming a continuous parabolic shear stress distribution in the cross section as predicted by elementary beam theory for homogeneous materials. However, it has been shown that the shear stress distribution is dominated by stress concentrations in the regions close to the loading

nose and the supports (Muszynski et al., 2000). In spite of these limitations, the short-beam test has become one of the most popular methods used by the industry to determine the interlaminar shear bond quality of composites due to the ease of specimen preparation and the simplicity of the experimental procedure. This method was used in our research as a performance indicator to assess the effects of fungal exposure on E-glass fiber/phenolic matrix composites.

2. Materials and methods

2.1. FRP material

One type of FRP was used in this experiment: E-glass/phenolic pultruded composite. This FRP composite, identified as K-1, was developed by the Advanced Engineered Wood Composites Center at the University of Maine (Dagher et al., 1998) and manufactured by Strongwell Corporation, MN. The FRP pultruded composite reinforcement consists of unidirectional (0°) E-glass continuous fiber rovings in the core with a melamine-coated E-glass randomly oriented chopped strand mat (CSM), 230 g/m^2 (0.75 oz/ft^2), at the surfaces. The fiber reinforcement is embedded in a phenolic matrix. In the fabrication process, the CSM mat was pulled dry in the pultrusion die to generate a resin-starved surface that improves bonding to wood. The corresponding average volume contents for the pultruded plate were obtained based on ignition loss tests according to ASTM D2584 and ASTM D2734 procedures (ASTM, 1994, 1998), as follows:

$$V_f = 54\%, \quad V_m = 21\%, \quad \text{and} \quad V_v = 25\%,$$

where V_f is the fiber volume fraction (both fiber roving and CSM), V_m the matrix volume fraction, V_v the void volume fraction.

The resulting high-void content leads to an open structure that favors movement and distribution of moisture within the composite material, and may enhance the growth of microorganisms. The density of E-glass fiber was 2.54 g/cm^3 and the density of the phenolic matrix was 1.2 g/cm^3 .

2.2. Sample preparation and decay testing

A total of 12, $25.4 \times 25.4 \times 3.175 \text{ mm}^3$ ($1'' \times 1'' \times 0.125''$) square coupons were wet cut, air-dried, oven-dried and autoclaved, respectively, from the same FRP plate (Fig. 1). Oven-dry specimen weights were determined prior to inoculation. Autoclaving was performed at 121.1°C (250°F) for 20 min to sterilize the coupons before incubation. Four coupons were exposed to the brown rot wood decay fungus, *Gloeophyllum trabeum* (Persoon: Fries) (ATCC # 11539) and four were exposed to the white rot wood decay fungus, *Trametes versicolor* (Linnaeus: Fries) (ATCC # 12679) utilizing a modified AWWPA soil block test (AWPA, 1999). In this test, four uninoculated FRP control

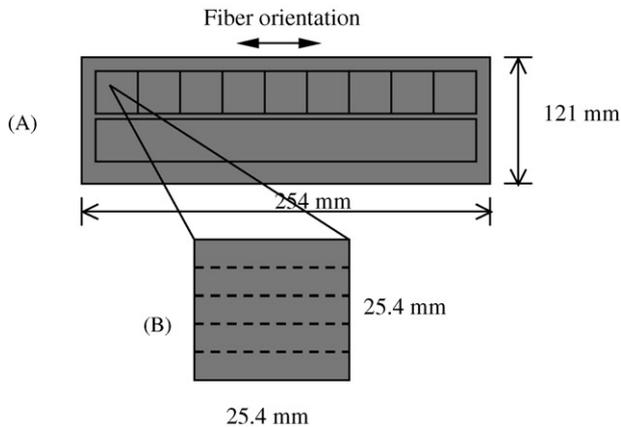


Fig. 1. Cutting schematic of FRP specimens: (A) 25.4 mm (1") square coupons for soil block and ultrasonic NDE tests, thickness 3.18 mm (0.125"), and (B) short beams were cut oriented with the unidirectional core fiber direction for interlaminar shear testing.

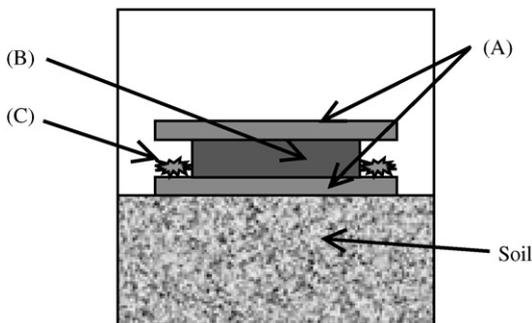


Fig. 2. Decay exposure details of FRP coupons in soil jars: (A) birch feeder strips, (B) square FRP coupons, and (C) fungal culture (transferred from petri dishes).

samples were maintained under the same soil block conditions but without exposure to the fungi. An equal number of $25.4 \times 25.4 \times 12.7 \text{ mm}^3$ (1" \times 1" \times 0.5") southern yellow pine (SYP) sapwood blocks were also cut, air-dried, oven-dried, autoclaved, respectively, then incubated with the same fungal species as the FRP samples. All test samples were sandwiched between birch feeder strips (Fig. 2) to simulate exposure conditions of an in-service FRP composite used to reinforce wood laminates in exterior environments. The test was modified to extend the exposure period to 24 weeks to allow a longer time for fungal attack of the FRP composite. The samples were then removed from the chambers, mycelium was brushed from their surfaces, and the samples were oven-dried at 103°C (217.4°F) before weighing. Weight loss was expressed as a function of initial oven dry (OD) weight.

2.3. Ultrasonic testing and waveform analysis

Each decay-exposed FRP coupon was tested ultrasonically using a square wave pulser, which excites a 1.0 MHz piezoelectric ultrasonic transducer. The transducer generates

an elastic pulse that propagates through the FRP coupon. The ultrasonic transducer is then excited by the received signal, which is sent through a preamplifier and displayed as a voltage versus time waveform on a digital oscilloscope (Fig. 3). FFT was used to analyze the energy distribution and changes in signal content of the decayed and control waveforms.

2.4. Interlaminar shear testing

Following fungal exposure, all decay-exposed and uninoculated composites were sized with a diamond tipped wet saw blade to strips of an approximate dimension of $25.4 \times 6 \times 3.175 \text{ mm}^3$ (1" \times 0.235" \times 0.125"). Three to four interlaminar shear strength (ILSS) strips were cut from all 1-in square coupons except from those that had become inadvertently contaminated in the decay tests. A total of 47 specimens were tested for all exposures representing unexposed, soil-exposed, and the two groups of decay-exposed samples. The specific distribution of multiple replicates from the tested exposures were: 13 miniature strips from four unexposed square coupons, 14 miniature strips from four soil-exposed square coupons, nine miniature strips from three *G. trabeum*-exposed square coupons, and 11 miniature strips from three *T. versicolor*-exposed square coupons. Specimens were tested in three-point flexure with a span-to-thickness ratio of 5:1 to promote interlaminar shear failure parallel to the plane of core lamination (ASTM, 1997) (Figs. 4 and 5). The experimental design was originally based on the assessment of individual decay exposures and failed to account for the jar to jar variability when multiple samples were cut from the coupons in each jar. To rectify this, the data were artificially regrouped according to their ILSS strength for statistical analysis to provide a 'worst case' significance evaluation (see Section 3).

ILSS depends primarily on the fiber/matrix interfacial shear strength and/or matrix properties rather than the fiber properties. This test has been used in material qualification programs (Lopez-Anido and Wood, 2001) as a mechanical property indicator to assess retained 'apparent' interlaminar shear strength after environmental exposures. ILSS is also considered a valuable screening tool to evaluate new fiber-resin systems, and compatibility of new fiber coupling agents (sizing) with matrix resins. In this research, we applied this technique to evaluate the effects of biodegradation/biodeterioration on FRP composites. Failure modes were noted and interlaminar shear properties were confirmed using scanning electron microscope (SEM) and light microscope analysis. The apparent interlaminar shear strength (MPa) at the mid-surface is computed according to beam theory (ASTM, 1997), as follows:

$$S_H = 0.75P_B/(bd),$$

where P_B is the ultimate applied transverse load prior to failure (N), b the width of specimen (mm), and d the thickness of specimen (mm).

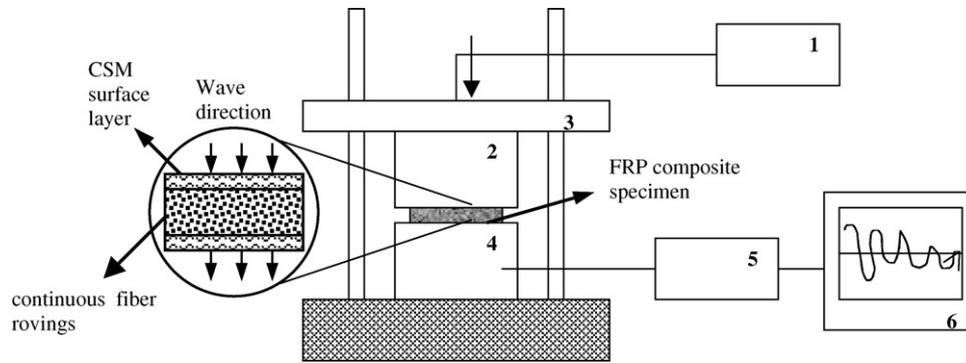


Fig. 3. Details of ultrasonic measurement of FRP coupons. Ultrasonic pulse generator (1), ultrasonic transducer (2), weight bar (3), ultrasonic receiver (4), ultrasonic pre-amplifier (5), digital oscilloscope (6).

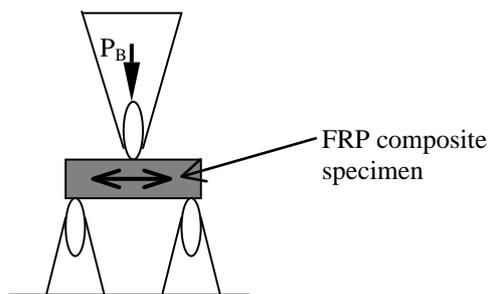


Fig. 4. Application detail of ILSS test on a short-beam specimen.



Fig. 5. Close-up of ILSS test of a FRP composite short-beam specimen.

2.5. SEM sample preparation

Additional samples of decay-exposed FRP composite material and resin films were prepared for SEM. The SEM samples were treated with 3% glutaraldehyde, buffered and washed with 0.2 M sodium cacodylate in DI water and fixed with 1% osmium tetroxide. Treated samples were mounted on aluminum stubs and coated with gold using a Polaron E5000 sputter device, and observed using a Cambridge S150 scanning electron microscope operated at 5, 10 and 20 kV. Fungal colonization, distribution, and

localized deterioration/disruption were observed on all fungal-exposed samples.

3. Results and discussion

3.1. Decay evaluation and microscopic analysis

The post-decay moisture contents and average weight losses of both wood and FRP samples are listed in Table 1. Following incubation, all test specimens were fully covered with fungal mycelium in both brown and white rot test chambers indicating that any leachable chemicals in the FRP material did not inhibit fungal growth. The FRP composite coupons all appeared in sound condition after the surface mycelium was removed (Fig. 6); however, the FRP surface beneath the hyphae displayed a bleached appearance, which could potentially be explained by the etching effect of organic acids produced by the fungus (Chung et al., 1999; Goodell et al., 1997a; Green et al., 1991).

The decay-exposed FRP coupons were weighed immediately after the surface mycelium was removed. Moisture uptake was calculated based on the post-decay OD weight and post-decay weights of FRP coupons. Coupons exposed to *T. versicolor* and *G. trabeum* gained 67% and 86% more moisture than sterile soil-exposed controls, respectively. The increased moisture uptake in the fungal-exposed samples was associated with fungal activity in the FRP coupons, perhaps affecting the fiber/matrix interface capillaries.

After drying, there was no detectable weight loss (based on oven-dried weight) of the FRP coupons after 24 weeks of exposure. SYP sapwood control blocks, however, sustained approximately 70% and 50% weight loss when colonized by *G. trabeum* and *T. versicolor*, respectively, over the 24-week exposure period (Fig. 7). This suggests that any degradation products produced in the soil block exposure of FRP were not metabolized by the fungi or that the fungal colonization of the FRP material produced only limited degradation products.

Table 1
Post-decay moisture content and weight loss of FRP material coupons after the soil block test

Specimen type	Average post-decay moisture content, % (Standard deviation) (based on post-decay oven dry weight)			Average weight loss, % (Standard deviation) (based on oven dry weight)		
	Soil-exposed control	<i>G. trabeum</i> exposed	<i>T. versicolor</i> exposed	Soil-exposed control	<i>G. trabeum</i> exposed	<i>T. versicolor</i> exposed
FRP material	0.84 (0.10)	1.40 (0.09)	1.56 (0.55)	0.1 (0)	0 (0.1)	0 (0)
SYP reference	—	—	—	0.8 (0.4)	69.6 (0.4)	51.4 (5.1)

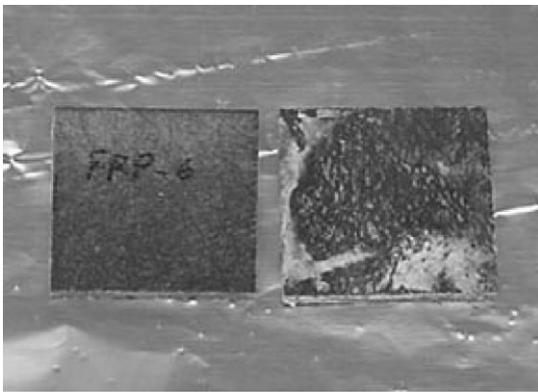


Fig. 6. Fungal growth and white mycelia mat on FRP composite surfaces and wood cross sections. Sterile soil-exposed FRP material control, left. *T. versicolor*-exposed FRP material, right.

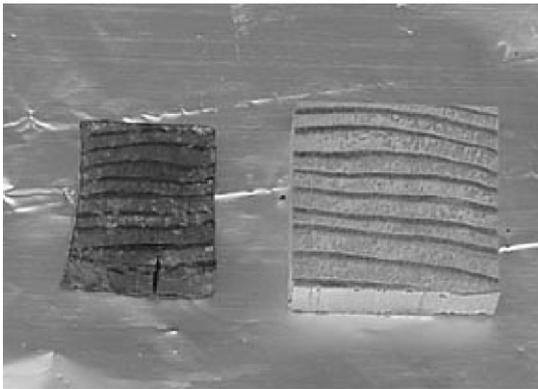


Fig. 7. Volumetric shrinkage of SYP reference blocks (*G. trabeum*-exposed, at left. Undecayed, at right).

3.2. Nondestructive evaluation

The waveform analysis and FFT results (Figs. 8 and 9) showed that the FRP coupons exposed to *G. trabeum* had considerable reduction in FFT magnitude of transferred energy as recorded by the shear transducer. This reduction in FFT magnitude can be attributed to the attenuation of the sound waves that travel through the FRP material. The average FFT magnitude recorded in the *G. trabeum*-exposed

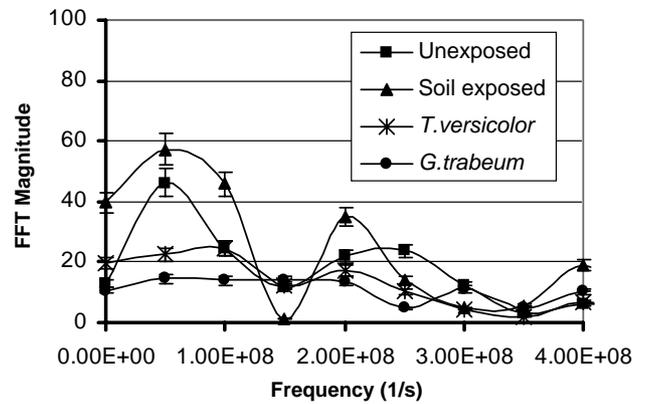


Fig. 8. FFT magnitude plots of control FRP material (sterile soil-exposed and unexposed) and FRP material exposed to *G. trabeum* and *T. versicolor* versus frequency for shear transducer data (each line represents an average of three specimens).

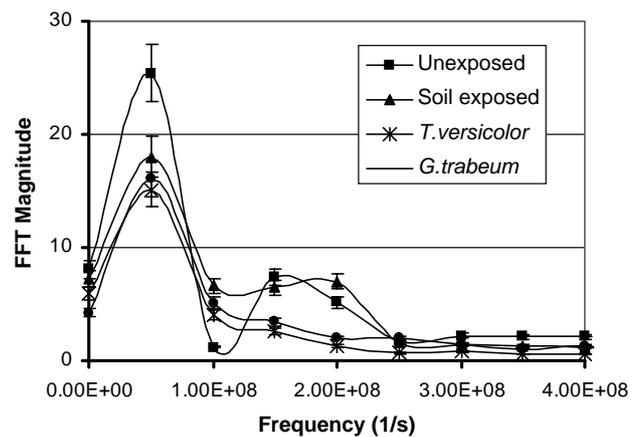


Fig. 9. FFT magnitude plots of control FRP material (sterile soil-exposed and unexposed) and FRP material exposed to *G. trabeum* and *T. versicolor* versus frequency for longitudinal transducer data (each line represents an average of three specimens).

FRP coupons was approximately 25% of that for the unexposed or sterile soil-exposed FRP. A 55% reduction in FFT magnitude was recorded in the *T. versicolor*-exposed samples with the same transducer. Unexposed and sterile soil-exposed FRP coupons, on the other hand, did not show

Table 2
Summary of ILSS values of FRP material differentially exposed for 24 weeks

Exposure conditions	Average ILSS (MPa)	COV (%)	ILSS change (%)
Unexposed	26.42	3.4	0.0
Soil exposed	26.72	4.5	1.1
<i>T. versicolor</i> exposed	25.39	7.2	-3.9
<i>G. trabeum</i> exposed	24.45	7.9	-7.4

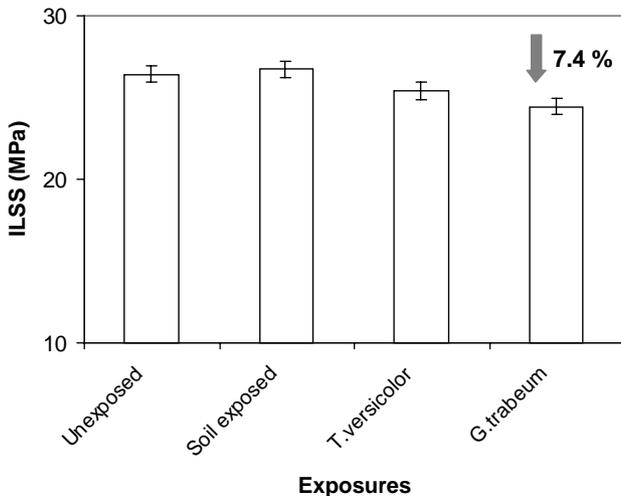


Fig. 10. Interlaminar shear strength values of control and decay-exposed FRP material after 24 weeks (the x - y error bars on the columns show standard error).

any reduction in FFT magnitude after 24 weeks of exposure. Internal changes (increased porosity or weakening of the fiber/matrix interface due to fungal growth or secretion of acids, etc.) may be responsible for the observed reduction of sound energy transfer in the *G. trabeum*-exposed FRP material. *Gloeophyllum trabeum* is known to use a non-enzymatic degradative system to attack cellulose in wood (Goodell et al., 1997b; Xu and Goodell, 2001), and this non-enzymatic degradation with production of powerful oxidants may play a role in FRP degradation.

3.3. Interlaminar shear strength test

Table 2 summarizes the average ISS values of the unexposed, sterile soil-exposed and decay-exposed (*T. versicolor* and *G. trabeum*) specimens after 24 weeks of exposure. An average ILSS reduction of 7.4% was recorded for the *G. trabeum*-exposed FRP material. The *T. versicolor*-exposed FRP material showed a non-significant 3.9% reduction (Fig. 10). The coefficient of variation (COV) values for decay-exposed FRP (Table 2) materials were greater than the COV values for the control (unexposed, soil-exposed) specimens. This is an important observation since the FRP composite allowable values depend on both the mean and

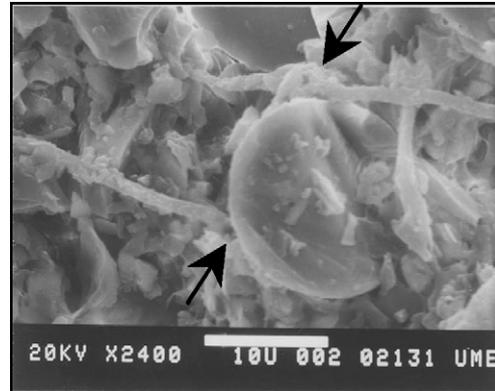


Fig. 11. A cross sectional view of decay-exposed FRP material showing fungal hyphae wrapped around (top arrow) a glass fiber and penetrating into the fiber/matrix interface (lower arrow).

COV. The higher COV values drastically reduce the allowable design values.

Unfortunately, the initial statistical analysis performed assumed that all strips in each treatment group had been independently exposed to decay fungi. This would be an appropriate assumption if each strip represented a sub-sample taken from individual (AWPA, 1999) soil block chambers. However, because a variable number of strip samples were taken from the 3–4 exposed coupons versus 12 independent samples, it is possible that the probability value ($p=0.003$), which shows a significant decrease in ILSS strength for *G. trabeum*, does not adequately reflect the true population variability.

A secondary attempt to analyze the data was performed using an artificial grouping technique (grouping the closest actual data points assuming they represented samples from the same exposure chambers) to test the effects of exposures and exposure chamber variability together. This technique allowed a ‘worst case’ statistical evaluation to be determined. The resulting p values varied upon the artificial grouping (p values 0.175–0.186) and indicated a weaker level of confidence than the original analysis where sources of variation were not partitioned adequately. Because of this, further work will be needed to verify that the ILSS reduction seen for *G. trabeum* is statistically significant at conventionally used confidence levels.

3.4. Microscopic evaluations

Visual, stereoscopic, and light microscopy (LM) inspections showed that, after FRP material coupons were incubated with decay fungi, mycelial mats were firmly attached to the surface CSM layer, with hyphal penetration by both fungi into the high-void-content region ($V_v = 38\%$ for surface CSM layer only). Further, LM and SEM analysis of the cross section of FRP material coupons incubated with fungi also revealed hyphal penetration (Fig. 11). The presence of large amounts of crystal development by both fungi was also

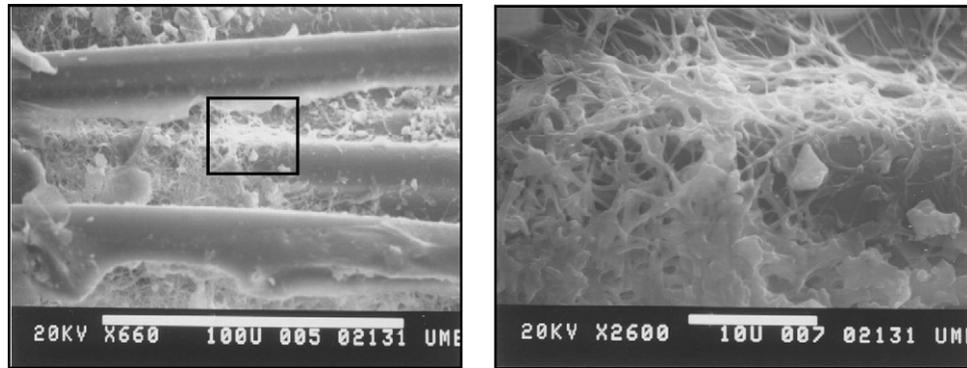


Fig. 12. Fungal growth on the fiber/matrix interface in FRP material exposed to *T. versicolor*. At left fungal hyphae traverse several glass fibers. At right, a close-up of boxed region in the left image showing fungal hyphae in the debonded fiber/matrix interface.

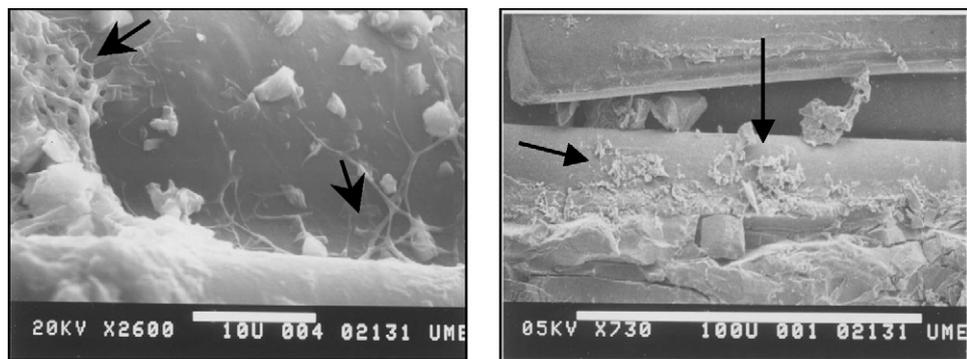


Fig. 13. 'Valley area' between the fiber and matrix of *G. trabeum*-exposed FRP material. At left: Fungal hyphae attached to a glass fiber surface (bottom), traversing the fiber and ramifying into resin coated glass regions (arrows). At right: Accumulated fungal residue (arrows) on a debonded fiber/matrix interface.

observed on the FRP material surface. Light, fluorescence and SEM observations all indicated that the FRP material is susceptible to fungal penetration by hyphae. The presence of fungal hyphae within the fiber–matrix interface of the unidirectional continuous fiber rovings of failed ILSS test specimens is shown in SEM micrographs (Figs. 12 and 13). This may be particularly important in helping to explain the reductions of interlaminar shear strength in the FRP material. Although it is unlikely that substrate-specific enzymes secreted by the fungi were responsible for the degradative effects, the production of acids which can reduce the pH of fungal microenvironments down to 2 or even less (Jellison et al., 1997) would likely have caused this type of degradation. The preferential distribution of bacteria and crystals along the fiber/matrix interface was also reported by Wagner et al. (1996) and Ray et al. (1997).

Our microscopic investigation supports a hypothesis involving the fiber/matrix interface weakening as a result of fungal attack. Since some sizing chemicals, including starch derivatives and acetylated celluloses, are biodegradable (Gu et al., 1997; Wagner et al., 1996; Sampath et al., 1997) enzymatic degradation of these compounds by wood decay fungi may be possible. Because of the proprietary nature of fiber sizing, the manufacturer has not disclosed the type of sizing used in our material. However,

silanes are often used with phenolic/glass FRP pultruded materials, and the powerful organic acids (oxalic acid) produced by wood-degrading fungi would be more likely to cause degradation of a non-organic sizing such as this. The CSM of our FRP material also contained a melamine resin binder, and the high nitrogen content of this resin may have promoted degradation in this region. The consumption or decomposition of fiber sizing or binders by fungi or their organic acids can introduce localized weakening on the matrix/fiber interface, which indirectly reduces the ILSS of decay-exposed FRP material. Addition of a fungicide to FRP components during manufacture might help to inhibit fungal degradation of these materials when they are exposed to moist environments that might promote fungal and other microbial activity. However, changes in surface energy and adhesion characteristics need to be taken into account and fabrication concerns may need to be addressed.

The addition of biocides to FRP materials introduces other important issues. Tascioglu et al. (2002) reported that some waterborne biocides (e.g. CCA) chemically attack glass fibers and induce spiral cracks and fissures on the glass surface reducing their tensile strength. These factors must also be considered if biodegradation is to be controlled using chemical preservatives (Tascioglu et al., 2002).

4. Conclusions

The following conclusions can be drawn from this research:

- (1) E-glass fiber/phenolic resin matrix pultruded composite materials designed for wood reinforcement (with high-void content) are susceptible to fungal penetration by common wood decay fungi.
- (2) Although no detectable weight loss of FRP material was recorded, there was a significant increase in the moisture content of both white and brown rot-exposed samples as well as a weak relationship (maximal p values 0.175–0.186) between the reduction in interlaminar shear strength and exposure of the FRP material to *G. trabeum*. Additional studies with improved design procedures will have to be performed to determine if a more robust relationship exists between decay exposure and FRP material ILSS values.
- (3) The combination of mechanical (interlaminar shear strength) and NDE (ultrasonic shear detection) techniques show promise for the monitoring of fungal activity in FRP composites. With further detailed work, these techniques might be useful in time exposure vs. strength loss modeling.
- (4) The fiber/matrix interface seems to be the key area for capillary distribution of fungal metabolites that may play an important role in interlaminar shear strength reduction.

Future work is planned at the University of Maine, AEWCCenter using different fungal and bacterial species on pre-stressed FRP material over a series several time exposures.

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