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# Evaluating the durability of wood/FRP bonds through chemical kinetics using a range of mechanical test methods

Jonathan Philip Alexander

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**EVALUATING THE DURABILITY OF WOOD / FRP BONDS  
THROUGH CHEMICAL KINETICS USING A RANGE OF  
MECHANICAL TEST METHODS**

By

Jonathan Philip Alexander

B.Sc. University of Wales, Bangor, 1998

A THESIS

Submitted in Partial Fulfillment of the  
Requirements for the Degree of  
Master of Science  
(in Forestry)

The Graduate School  
University of Maine  
December, 2000

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# **EVALUATING THE DURABILITY OF WOOD / FRP BONDS THROUGH CHEMICAL KINETICS USING A RANGE OF MECHANICAL TEST METHODS**

**By** Jonathan Philip Alexander

Thesis Advisor: Dr. Stephen M. Shaler

An Abstract of the Thesis Presented  
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Hybrid composites of wood and fiber reinforced polymers (FRP) exhibit a flexibility of design properties through choices in fiber type, amount, orientation and resin type. With this flexibility comes the need to measure the durability of the composite material system, especially the bond between these two materials due to their markedly different responses to moisture. This study is two-fold; the first section examines methods for the mechanical testing and durability of wood / FRP laminations. Six mechanical tests were evaluated and a modified block shear method was identified as the most suitable due to its ability to provide representative results independent of material integrity.

The Second section evaluated the wood / FRP bond durability for PRF and epoxy adhesives through chemical kinetics. The control maple laminates were projected to retain 30% of their post vacuum pressure soaked strength for 12 years. When PRF and

epoxy / HMR adhesive systems were used to bond the FRP reinforcement to maple the time projections to 30% bond strength degrade were 57 and 34 years, respectively. The epoxy resin was found to reinforce the wood / FRP at the bonded interface and this system, after exposure to a vacuum pressure soak cycle had a mean shear strength of 2,058 psi which was 23% greater than the PRF system. The ability of the epoxy to make the porous FRP impermeable to water was also recognized. The rate process assessment from chemical kinetics was combined with moisture cyclings, maple / PRF / FRP laminate shear blocks were evaluated. It appears that an interaction exists between the level of accelerated aging and the stress induced by moisture cycling. There is considerable experience with the successful use of wood / PRF systems in outdoor use and their durability has been proven. This study indicated the **PRF** and epoxy bonds between maple and FRP reinforcement are more durable than those between maple and PRF.

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## Part I

### **Methods for the Mechanical Testing and Durability Assessment of Wood / FRP Laminates**

## ABSTRACT

Hybrid composites of wood and fiber reinforced polymers (FRP) exhibit a flexibility of design properties through choices in fiber type, amount, orientation and resin type. With this flexibility comes the need to measure the durability of the composite material system, especially the bond between these two materials due to their markedly different responses to moisture. Currently there are no standardized performance qualification methods for bonded interfaces of hybrid wood composites. The need to develop (or modify an existing technique) which is able to characterize the effect of real or simulated exterior exposure of wood / FRP / adhesive systems is clear and is the objective of this study. The method should ideally be simple, low-cost, discriminating of differences in bond performance and exhibit **low** variability for a uniform set of samples. An additional criteria is the sensitivity to differences in joint properties. This difference in mean performance is important to indicate differential system performance. A third criteria to be evaluated is ~~the~~ ease-of-use and preparation of the specimen; a difficult to machine specimen may become a barrier when large numbers are required in quick time. Six mechanical test methods were evaluated based on the stated criteria. A modified ASTM D905 method was identified as the most suitable and able to provide representative results independent of material integrity. The bond between maple and FRP was found to **be** as durable as that between maple when bonded with PRF.

## Introduction

### Testing And Evaluation Of Bonded Products

Adhesives used to bond FRP reinforcements to wood must be durable and compatible with exterior conditions. Testing of adhesives used to bond these materials must provide a means of determining their level of performance and / or describe their durability. The ultimate aim of testing can be summarized as a reliable determination of pass / fail levels and prediction of service life for conditions similar to those in which the product is actually used (Marra 1992).

There are an infinite variety of adhesive types, substrates, joint designs, strength levels and durability requirements which has led to the development of a wide range of test procedures both in published forms as standards and in unpublished forms which tend to be developed for specific analyses by individual companies. The performance of the bond under investigation can **be** a function of the **structure**, composition and properties of the adhesive layer; the boundary layer between the adhesive and the adherend; or the **type** of adherends being used. Ultimate failure or delamination of an assembly can occur within the adhesive, adherend or adhesively within the boundary region. Hence the only satisfactory way to evaluate performance is through fabrication and testing of bonded systems as opposed to conducting tests on the adhesive itself.

In spite of documented difficulties with the use of block and modified block shear specimens for evaluating wood / wood adhesive interfaces and wood / fiber reinforced polymer (FRP) / adhesive interfaces, it remains the most popular method. Examples of this can be found in Barbero et al. 1994, Gardner et al. 1994 and Vick 1995. An objective of this study is to evaluate the suitability of other mechanical test procedures in quantifying bond quality between wood and FRP. This will be **based** on the level of variability and the ability to distinguish between processing parameters that each method exhibits and the 'ease-of-use' of the technique. The identification of such a technique is important for a reliable and rapid assessment of bond durability and investigating the differential response of system combinations ( wood / FRP / adhesive). The aim of this objective is to ensure that the question of "which was the most appropriate method for testing such systems" **no** longer needs to be asked.

Structural elements are subjected to different states of stress depending on structure, element size, connections and service environment. Small Scale testing cannot simulate such complex systems but this type of accelerating the aging of small specimens is based on a worse case Scenario. This is due to edge effects amplifying the response to fluctuating environmental conditions and the use of wet immersions as opposed to the less severe dry conditions which actual structures are subjected to.

### Shear Testing

Shear loading *is* frequently considered the most efficient of test techniques because the load is transferred over the entire bonded area. In contrast, peel and cleavage loading concentrate the force along one edge of the bondline (Caster **1981**).

*Block shear*; There are two standard block shear methods which are commonly used to evaluate shear strength of solid clear wood and adhesively bonded samples (**ASTM D143** and **ASTM D905**). Both originate from early test methods used to determine the shear strength of wood for military design (Hunt **1917**, Truax and Harrison **1922**). A single notched specimen ~~is~~ used in **D143** (designed for solid wood) and a double-notched geometry is used in **D905** (designed for adhesive joints). In **D143**, tests shearing takes place in a single plane, 2 inches wide and 2 inches deep, **but** the shearing edges of the tool are offset horizontally by 1/8 inch, promoting failure of the weakest plane within this zone. With **D905** testing the shearing takes place in a single plane, 2 inches wide and **1.5** inches deep. The shearing edges of the tool are in the same vertical plane which focuses the **stress** on this plane (McLeod et al. **1956**).

McLeod and his colleagues found that the restriction of the failure zone in the **D905** method created higher shear strength values than for the **D143** method. The increases were between **13** and **60%** with increases being greater for **softwoods** than hardwoods. The concentration of peak stresses on the adhesive plane makes **D905** a better indicator of adhesive strength. This was affirmed in a comparative study by Okkonen and River (**1989**) which used the two methods to test a variety of solid and bonded wood specimens. They concluded that **D905** gave a better indication of shear strength.

When seeking to gain a measure of the bond integrity between wood and FRP, investigators commonly use the **D905** method. It is able to indicate shear strength and percent material or cohesive failure for wood /

FRP samples. However since the FRP samples are rarely 0.75 inches thick an additional wood layer is bonded to the FRP to allow the sample to sit correctly in the shear tool.

Concerns with the non-uniform and non-linear stress distributions in the failure plane of standard D905 specimens have been raised for many years. Such concerns are also applicable to the modified test specimens. Comprehensive analyses of stress distributions were carried out as far back as 1935 by Coker and Coleman who conducted photo-elastic examinations on isotropic models of shear specimens. The existence of high stress concentrations near the re-entrant corners, and the non uniform shear stresses imposed over the test sections due to the geometry of the specimen and the method of loading were amply demonstrated.

The strain distribution at the interface of bonded D905 specimens was further examined by Yavorsky and Cunningham (1955) who used a brittle lacquer strain indicator coating. They concluded that neither pure nor uniform shear stress was induced in the plane of the bond interface due to stress concentrations at the re-entrant corners and tensile stresses normal to the interface plane.

A typical strain pattern obtained from the application of a brittle lacquer strain indicator coating is shown in Figure 1. The formation of strain cracks in the vicinity of the notches at low loads indicated high stress concentrations in these regions, and the variations in the slope of the cracks along the test section indicated a variable shear stress over this area.

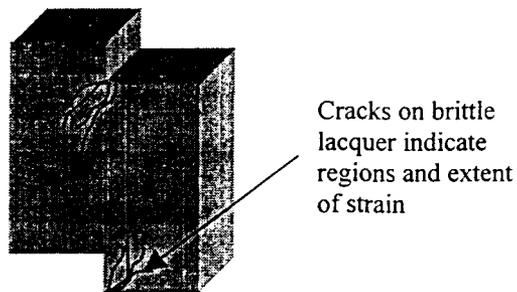


Figure 1 Strain pattern on a maple D905 test specimen

Radcliffe and Suddarth (1955) conducted an experimental stress analysis of single notched D143 specimens and found that in relation to the assumed constant shear stress, a concentration factor of approximately 2 existed at the re-entrant corners and the stress distribution was highly non linear. More recently, a finite element analysis were conducted by Cramer et al. (1984), Soltis and Rammer (1994) who reported stress concentration factors in this corner region of approximately 2.3.

Part of the Radcliffe and Suddarths study of the D143 test method involved cutting a slot terminated by a hole on the sample which was perpendicular to the grain at the reentrant corner. Although this modification was partially successful in reducing the undesirable stress concentration they stated; "the specimen is not recommended, since shear failures are often preceded by tension perpendicular to grain failures". The results of both types of shear test give the impression that the shear area is subjected to a uniform **stress**. Based upon a stress analysis employing electrical resistance **strain** gauges they **concluded** that this interpretation of results was incorrect and **stresses** on the bonded area ranged from 0 to 600 **psi** depending on position.

Yavorsky et al. (1955) evaluated a reduced glue bond area technique to evaluate standard D905 Specimens in failure. Shear blocks were prepared in the normal manner though adhesive was only applied to 50% of the normal bonded area (Figure 2). The average load at failure was reduced by 17% when the bonded area was decreased by this level of 50%, indicating that non-uniform loading occurs over the area of the test section. This is in agreement with Radcliffe and Suddarth. While they examined D143 specimens the method of loading was similar and they found the upper and lower bonded portions of the shear area to **be** subject to much greater levels of stress than the mid portion. In addition, the existence of tensile or 'tearing' stresses perpendicular to the plane of the glue joint have been shown to exist, both by photoelastic studies (Coker and Coleman, 1935) and the stress coat analysis. These normal stresses may result in failure of the specimen due to cleavage rather than shear.

*Compressive and lap shear*; Strickler (1968) examined a further type of shear strength testing technique, the compressive shear method. **He** found **it** provided a lower unit shear strength than the block shear specimens. These samples were found to fail in tension (cleavage) in some instances as the bending

moment induced by rotation tended to close the machined notches. Compressive shear specimens also developed lower unit shear strength than block shear specimens, and cleavage failure deep in the wood was a problem. Bending moments induced by rotation tended to close the notches in these compression shear specimens and to open the notches in lap shear. These findings caused lap shear and compression shear test modes to **be** dropped from Strickler's study.

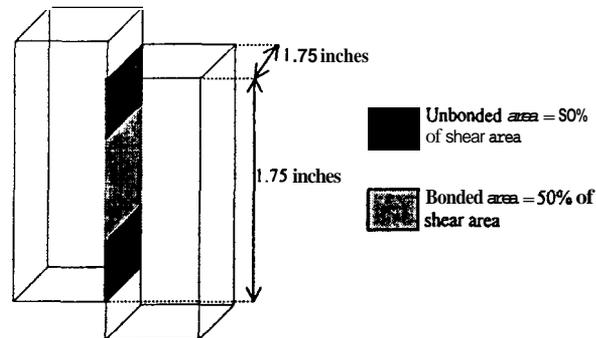


Figure 2 Reduced bond method block shear as used by Yavorsky *et al.* (1955).

*Compression shear;* This method has been found to provide an indication of the bond quality of a material subjected to shear deformation that correlates to the tensile strength perpendicular to the surface, (ASTM D1037). It's based on the principle that the test jig causes maximum stress to develop at an angle  $45^\circ$  to the force. In testing the specimen is positioned in the jig (Figure 3), so that the face planes of the specimen are at  $45^\circ$  to the compressive load. This configuration produces shear and compressive components (Hall and Haygreen 1983). Failure is not confined to a particular interface region as with block shear testing, but will occur in the weakest plane through the thickness of the sample. Results correlate well with those from tension perpendicular testing of **wood** based panels (Hall and Haygreen 1983).

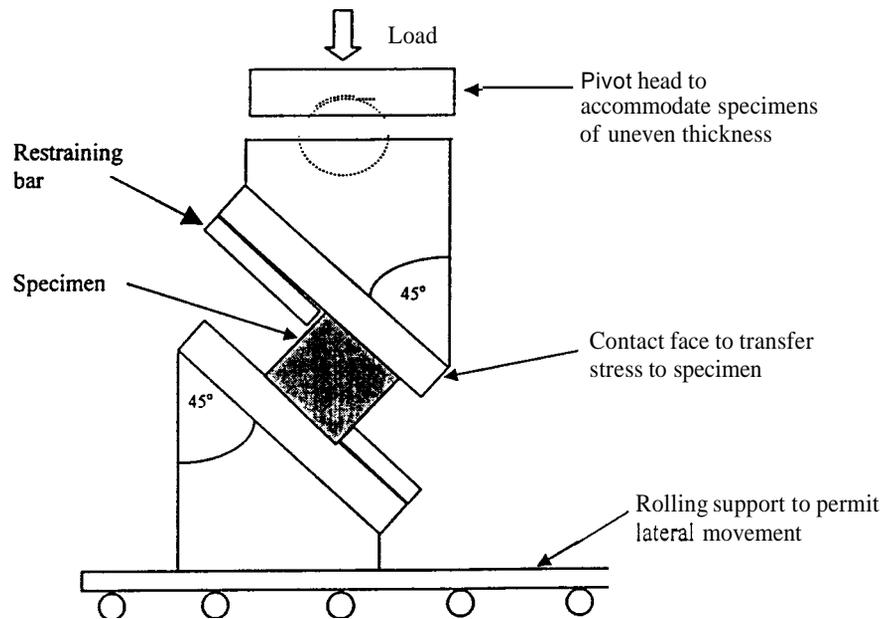


Figure 3 Diagram of Minnesota shear test jig as used in **ASTM D1037** testing

### Glueline Cleavage

Northcott (1952) was concerned **with** the amount of variability in testing wood / wood bonds, some of which appeared to **be** attributable to the wood or test method rather than the adhesive interface under examination. Four methods of test (tension normal to the glueline, tension shear, block shear and glueline-cleavage) were used to evaluate the quality of the bond between Douglas fir and phenolic resin adhesives.

Of the four methods trialed, the glueline-cleavage method appeared to offer the greatest promise of developing into a useful test. In conducting this test a knife edge was placed along the glueline of the specimen and the force required to cleave this joint measured. It was found necessary to grease the knife edge before testing each specimen to prevent pitch from collecting on the knife which altered the frictional component of the applied force.

When the results from these four test methods were compared by Northcott (Table 1) it can be seen that their means are several orders of magnitude apart. The coefficient of variation (**COV**) was determined to provide the best means of comparing variation between them as it expresses the standard deviation of each

population as a proportion of its mean. Applying the criterion that the test which gives the minimum COV (other factors being equal) incorporates the lowest error level, it can be concluded that the glueline-cleavage test with a greased knife was the most suitable of those compared for determining the gluing properties of Douglas fir bonded with phenolic resin adhesives.

Table 1 Results of four glueline evaluation test methods (Northcott 1952).

	Mean wood failure (%)	No. of specimens tested	Mean breaking load (lbs.)	Std. Dev. (lbs.)	COV (%)
Tension normal to glueline	97.7	35	337.4	91.8	27.2
Tension shear	89.4	35	308.4	42.2	13.1
Block shear	92.4	35	1754.6	356.2	20.3
Glueline-cleavage (no grease)	9.6	35	246.8	33.1	13.4
Glueline-cleavage (with grease)	10.3	15	241.6	23.0	9.6

The COV's of tension-shear and glueline-cleavage specimens tested with an ungreased knife were approximately the same. Northcott stated that had it been necessary to make a choice between these he would have been in favor of the glueline cleavage test because the tension-shear specimens had a much greater percentage wood failure while the glueline-cleavage specimens broke along the glueline.

For the types of break obtained using the ~~first~~ three test methods the percentage wood failure values (Table 1) show that the tendency is for these test specimens to break in the solid wood. Though this does indicate the adhesive is capable of withstanding the average strength value of the bonded assembly it would seem unsatisfactory for studies of the gluing properties of materials. The glueline-cleavage test minimized this factor.

### Tension Perpendicular To Surface

The intended scope of this test has been the determination of fiberboard cohesion in the direction perpendicular to the surface of the board and is believed to be a good indication of overall board quality. It is used in the majority of comparative board studies, and is considered a benchmark.

temperatures and moisture contents. They demonstrated that the J-integral method was able to describe the opening mode fracture in wood. It was found to be a more appropriate method than those which assume wood behave in a linear elastic manner when calculating the actual fracture strength of wood.

As mentioned, the samples in this study are required to be small and easy to machine to allow rapid sample production. The authors' previous experience is that the small size of wood / FRP / adhesive samples evaluated to contain such a level of variability which made the application of fracture mechanics very time consuming. The requirement of this study to identify a user-friendly test meant a modification to the ASTM method of analysis in that only the peak load was recorded.

## Materials

Wood species: A species having high shear strength was required so that the system wouldn't fail at a low level of stress. Red maple (*Acer rubrum*) was chosen on this basis. The lumber was locally sourced and delivered in kiln dried flatsawn ten foot boards of 1 X 6 inch nominal dimensions. The boards were straight grained, select grade or better and conditioned at 70°F / 65% rh (approximately 8% m.c.) prior to knife planing 24 - 48 hours before lamination.

FRP: Two FRP materials were used in the preliminary tests. The first FRP was a pultruded material supplied by Strongwell, Chatfield division, Minnesota. The pultrusion process produced a continuous plate 0.13 inches thick and 4.73 inches wide. The pultruded plate consists of a uni-directional E-glass roving embedded in a phenolic resin matrix, a porous chopped strand mat is placed on the upper and lower surfaces of the plate to facilitate mechanical interlocking with the adhesive.

The cured pultruded plate was passed through a drum sander to remove gloss and debris from the surfaces of the material. The surfaces were acetone wiped prior to priming with a PRF adhesive coat. The second FRP type was a pre-impregnated (wet-preg) type which had a PRF matrix and a uni-directional woven E-glass fabric reinforcement, with a weight of 26oz / yard<sup>2</sup>. The materials were processed using in-house pilot plant equipment (impregnator, hydraulic clamps). A characteristic of the finished wet-preg FRP sheets is their glazed surface. Initial trials were aimed at determining whether this inhibited a strong bond between

the FRP and maple. A sandblasting technique was used to remove this glazed surface from the upper and lower sides of the FRP sheet. This is often used in composite products to break the glaze. Block shear specimens were then prepared using this sandblasted FRP and FRP in its original (glazed) state. The mean shear strengths were 1580 psi for the blasted and 741 psi for the non-blasted material. A t-test found the blasted sample set to be significantly stronger ( $p < 0.001$ ). The predominant failure mode for this set was in the FRP material as opposed to complete glueline failure for the non-blasted material.

Resin: A phenol resorcinol formaldehyde (PRF) resin was used. The selection was made on the basis of promising performance when bonding wood and FRP in previous studies. PRF resins were examined in dry conditions by Brown (1998) and in wet and dry conditions by Gardner et al. (1994). The PRF resin was used for bonding the two systems, to prime the pultruded material, and as the matrix for the wetpreg. It was a two part system supplied by Georgia Pacific (4242/4554) with a viscosity of 2,200 cps, and was mixed immediately prior to application and applied at a rate of 0.25 g/inch<sup>2</sup>. The manufacturer recommended it be applied to wood of between 8 and 12% m.c.. The laminations were placed in a press after lay-up and clamped at 100 psi for 24 hours at ambient temperature. The priming coat was applied at a rate of approximately 0.05 g/inch<sup>2</sup> and was drawn across the FRP surface with a metal scrape. This coat was given a curing period of 48 hours at ambient temperature prior to lay-up.

## Sample Preparation, Conditioning and Testing

Laminations measuring 4.75 X 23 inches wide were manufactured and the test samples cut from these. The sample dimensions for block shear, compression shear and glueline cleavage were 1.5 X 2.0", 1 X 1" and 2 X 2" respectively. Peak load and failure mode were recorded for each. The zero time control specimens were tested in wet and dry conditions. Those samples immersed in high temperature water baths were allowed to cool in a bath of tap water prior to testing wet. These types of lamination were tested for quality using ASTM D1101 which is routinely used to assess the suitability of laminated products for exterior use. Three cycles were employed and two replicates of the maple / PRF and maple / PRF / FRP system tested. The mean delamination along wood to wood bonds was 8%, there was no delamination of the wood / FRP bonds.

Stainless steel tanks (250 liters) were used to immerse the sample batches at each of the elevated temperatures, circulating water baths maintained the tank temperatures ( $\pm 0.5^{\circ}\text{C}$ ). Plastic tanks were used for the immersion of samples at room temperature. While these required no hot water circulation a circulating pump was placed in each to prevent stagnation. VWR brand algicide was used through out to limit microbial growth. Sample batches were vacuum pressure soaked and placed in mesh containers prior to immersion. They remained immersed for the defined period; after which they were cooled in tap water, bagged and tested wet. The periods of immersion are given in Table 2. Samples were vacuum pressure soaked prior to immersion. To ensure that those samples immersed for the shorter time periods had the same level of water impregnation as those immersed for the longer periods.

Table 2 Sample immersion periods at each temperature.

Temperature( $^{\circ}\text{C}$ )	Immersion periods (hours)				
	1	2	3	4	5
95	6	12	42	90	148
85	24	48	144	340	484
75	144	600	936	1152	1752
65	336	1176	1776	2326	2760
24	336	1176	1776	2326	2760

## Results and Discussion

### Preliminary Test Results

Two laminate systems were tested (maple / wetpreg / PRF and maple / pultruded material / PRF ) by each of the five test methods at both room condition and following a vacuum pressure soak treatment (VPS). Eight replicates for each material / test combination were used. The results are summarized in Table 3. A summary of the predominant modes of stress induced by each of the tests and comments on their stress concentrations is given in Table 4. The tension perpendicular and compression shear tests induce a stress field throughout the specimen and are termed volumetric. The block shear test has been found to localize stress at the bond interface (Barbero) and the compact fracture test and glueline cleavage test concentrate stress at the crack tip (Smith and Kobayashi). Such a variety of stress modes was chosen so the sensitivity of each to accelerated aging treatments could be judged.

The absence of results for the **VPS** samples tested with the tension perpendicular method was due to delamination which occurred at the **epoxy** bond interface between the blocks and the sample. There was then no way of rebonding the wet sample to the test block. In terms of material failure, the aim of identifying a test which developed a greater percentage bond interface failure than achieved with block shear was not realized. However, the failure **mode** can be seen to differ by system. For the pultruded **FRP** system, it occurred consistently at the interface **between** the unidirectional core and the **CSM**. **Wetpreg** had no such surface mat and failure occurred in the uni directional rovings of the FRP.

		Block shear (psi)	Compression shear (psi)	Glueline cleavage (lb / inch)	Tension perp. (psi)	Fracture (lb/inch)
Room	Mean	2081	2040	504	343	185
	COV	7.1	13.0	10.8	10.6	10.9
	Failure	FRP	FRP	FRP	FRP	FRP
VPS	Mean	1196	1105	435	---	175
	COV	16.8	8.1	11.6	---	8.9
	Failure	FRP	FRP	FRP	---	FRP
Percent change		43	46	14	---	5
		Block shear (psi)	Compression shear (psi)	Glueline cleavage (lb / inch)	Tension perp. (psi)	Fracture (lb/inch)
Room	Mean	1562	2560	644	386	152
	COV	25	10.5	12.8	11.4	15.9
	Failure	FRP CSM	FRP CSM	FRP CSM	FRP CSM	FRP CSM
VPS	Mean	741	1955	499	---	140
	COV	30	9	30.5	---	16.3
	Failure	FRP CSM	FRP CSM	FRP CSM	---	FRP CSM
Percent change		53	24	23	---	8

While the failure mode was consistent by test, the sensitivity of each to the VPS treatment is not. **Block** shear (BS) was the most sensitive and fracture the least. The inability to test tension perpendicular samples in the wet state meant its sensitivity could not be gauged. The incorporated experimental error of each test was indicated by the COV, and the compression shear (CS) test was the best performer in this category. A matrix was drawn up to allow comparison of the test methods and this is shown in Table 5.

This indicates compression shear and glueline cleavage were the tests which best meet the criteria, whilst tension perpendicular and fracture could be described as the dunces of the class. It was decided that the two best performing tests, along with standard block shear method, should be used to evaluate bond

durability. Two laminated systems were used in the further analysis, the first was a maple / PRF / maple lamination (**M**), the second a maple / PRF / K1 type (**K**).

Table 4 Predominant mode and nature of **stress** application for five mechanical tests used in the assessment of wood / FRP laminates

	Predominant stress mode	Comments
Tension perpendicular	Tension	High stress throughout specimen
Compression shear	Shear / compression	High stress throughout specimen Less sensitive to defects due to compressive component
Block shear	Shear	Shear localized to interfacial region
Compact tension	Tension	Stress concentrated at crack tip
Glueline cleavage	Tension	Stress concentrated at crack tip

### **Applicability of Test Procedures to Durability Studies**

The test methods identified as being the most suitable were then used to test a large number of laminates following an accelerated aging treatment. Two hundred and fifty samples of each system (maple / PRF and maple / PRF / pultruded) were produced for each of the three test methods to give a total of one thousand five hundred. The groups of two hundred and fifty were randomly divided into batches of ten replicates for exposure to accelerated aging treatments. Five water baths, maintained at 95, 85, 75, 65 and 24°C, were used and five batches per system and per test method were placed in each of these to total 300. The immersion conditions were chosen to provide a range of deterioration rates within experimentally feasible time limits (Table 2). These were based on those used by Millet et al (1977) in the analysis of bonded maple. Batches were removed periodically for strength evaluation to allow calculation of the rate of loss with time to be calculated at each temperature. This data was also used in the analysis of each test methods variability. All samples were vacuum pressure soaked prior to immersion to ensure complete water penetration.

	Ease of use (Specimen prep. and testing)	Sensitivity	COV	GPA
Block shear	C	A	B-	2.89
Comp. Shear	A	B	A-	3.56
G. Cleavage	A-	C+	B-	3.00
T. Perpendicular	B	-	A-	2.22
Fracture	B-	C	B+	2.67

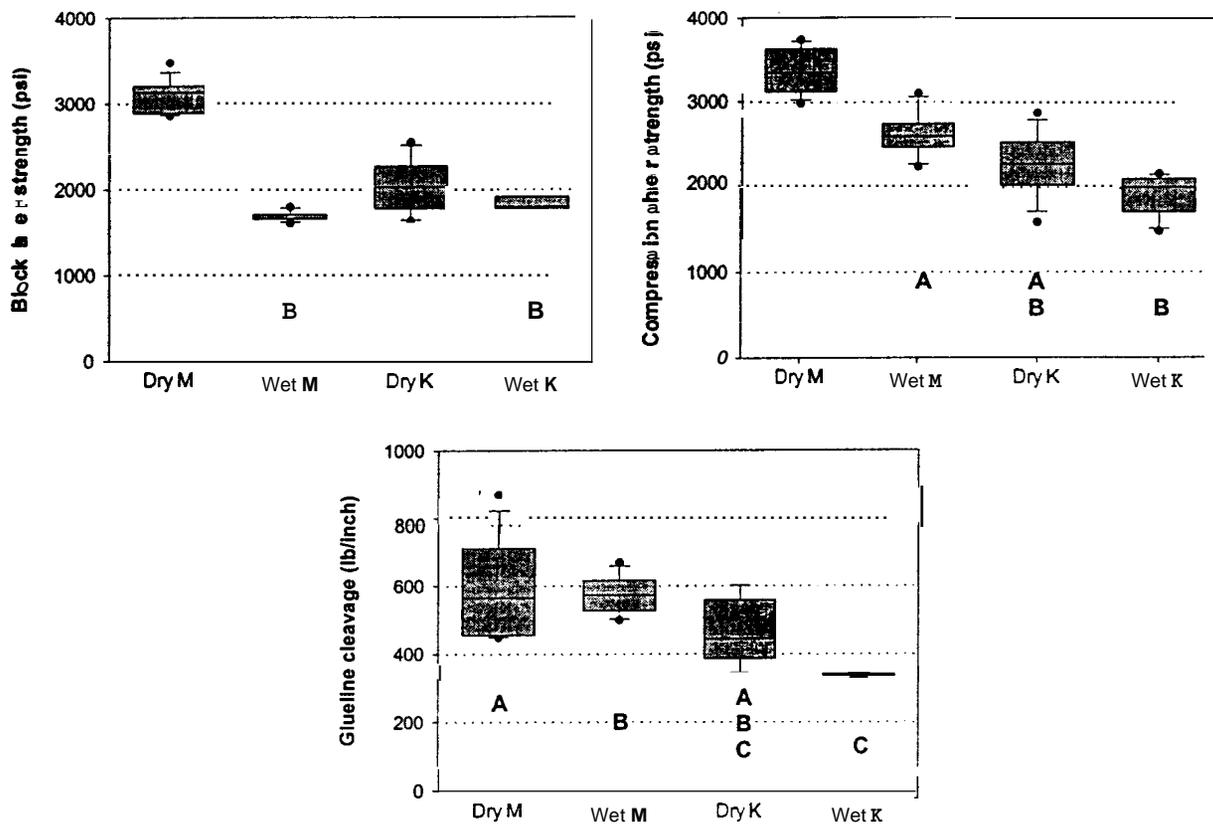


Figure 4 Test strengths of maple / PRF (M) and maple / PRF / FRP (K) laminations at room condition and after exposure to a vacuum pressure soak cycle as evaluated by three test regimes i) block shear, ii) compression shear and iii) glueline cleavage. Results of a Tukey multiple comparison test was run for the each of the test methods, (where matched letters indicate no significant difference between sets,  $\alpha = 0.05$ ).

In addition, matched control batches were produced. These control specimens were tested at room condition (70°F 65%RH) and following a VPS. The VPS results provided, a zero time immersion strength level to base the rate of strength loss with time of immersion at each temperature. The results are shown for each test method in Figure 4.

A Tukey test analysis ( $\alpha = 0.05$  level) of the results indicated that the VPS had a significant effect on the block shear and compression shear strength of the wood system. The glueline cleavage method was not able to detect a significant affect of the VPS treatment on the FRP system. This was unexpected when the differential swelling is considered. The graphs show the median of the wood results to be generally greater than for the FRP system following the *two* treatments. The Tukey test indicates these are not always significant ( $\alpha = 0.05$ ). The predominant failure mode of each system type was consistent for all test and condition types, ie. for the maple / PRF it occurred in the **wood** and for the maple / PRF / pultruded FRP in the **CSM** of the FRP.

An exponential decay regression equation ( $y = ae^{-bx}$ ) was used to fit the rate of residual strength loss versus time of immersion at each elevated temperature. The nonlinear coefficients (a and b) were determined using the Marquardt-Levenberg algorithm provided by the SigmaPlot software package. The time for each system to achieve a 25% drop in residual strength at each temperature was calculated from the appropriate regression. This approach was *used* to handle data for a chemical kinetic analysis of wood / FRP bond durability which predicted bond a theoretical **lifespan** for such bonds in actual use conditions (Alexander et al. 2000)

These equations are plotted and the strength level is expressed as a residual of the zero immersion time strength to allow cross test comparisons (Figure 5). A linear curve was required for the 24°C immersion data as it did not follow the pattern of exponential decay. The nature of the linear curve meant it was unable to pass through the zero time point of origin. Millet and Gillespie (1978) showed that 20 days immersion in 98.5°C water were required for maple samples to lose 20% of their OD weight and 280 days at 80°C. The curves produced with their data for immersion at these two and intermediate temperatures showed that weight loss to proceed at a rate similar to residual strength loss at corresponding temperatures. Weight loss could not be accounted for in this study as sample markings became illegible with prolonged immersion.

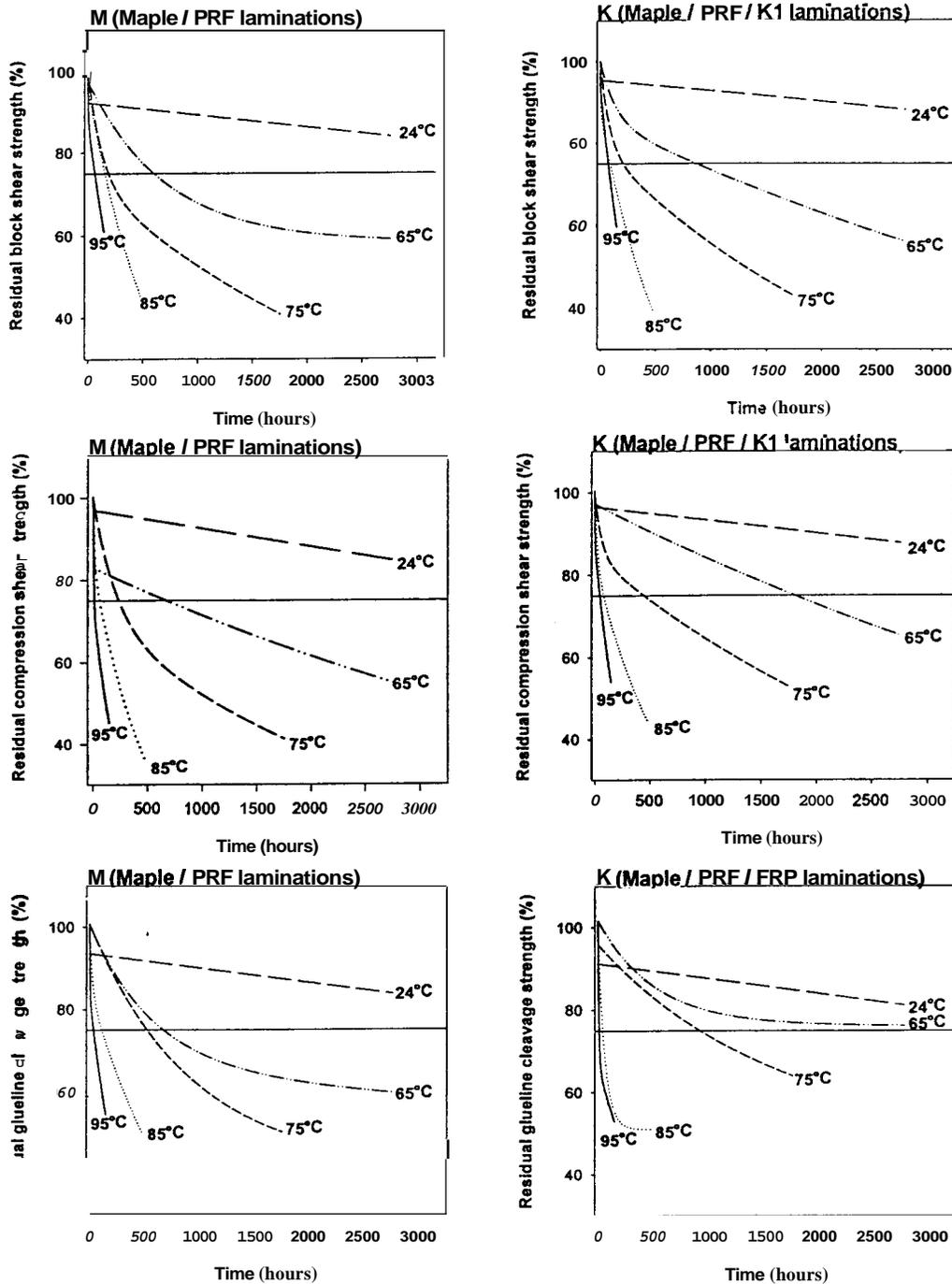


Figure 5 Rates of residual strength degrade for two systems when immersed in water baths maintained at five temperatures, (as measured by three test methods)

The time taken for the strength of these systems to exhibit a 25% reduction in strength by each method is shown in Table 6. At 95 and 85°C the time taken to attain a 25% loss in block shear strength was greater than both compression shear and glueline cleavage for both system types. At 75 and 65°C the time taken to reach the same level of block shear strength loss was less than with compression shear and glueline cleavage.

Table 6 Time to 25% loss in residual strength for maple / PRF and maple / PRF / FRP laminate systems.

	M (Maple / PRF)				K (Maple / PRF / FRP)			
	95°C	85°C	75°C	65°C	95°C	85°C	75°C	65°C
Block shear	64	157	185	604	72	93	205	863
Compression shear	11	59	233	668	47	81	445	1800
Glueline cleavage	35	110	520	668	12	44	924	2760

The severity of the two higher temperature immersions caused the specimens to lose considerable weight and integrity, which is an important factor when the compressive forces transferred to the specimens in compression shear and glueline cleavage tests are considered. In compression shear they occur as the faces of the jig press down on the upper and lower surfaces of the sample at 45°. They are induced in glueline cleavage where the edges of the cleaving knife press down into the notch shoulders as load is applied. These compressive forces contribute to a mixed state of stress being generated in the samples. The block shear test jig transfers minimum compression force to the parts of the sample which are not in the immediate vicinity of the bond line, causing the results to be more independent of wood integrity. The addition of this variable to the data by compression shear and glueline cleavage testing is a particular problem in this study where sample integrity changes by treatment.

An additional problem with the compression force exerted during compression shear testing was that the sample may fail in shear but load continues to be applied and the sample compresses further. This creates a problem when trying to identify the actual point of initial failure, which strongly detracts from the user workability of the test. The dependency of compression shear and glueline cleavage results on the integrity of the sample material would make them unsuitable for comparative studies between hardwoods and softwoods.

The families of curves in Figure 5 Error! Reference source not found. can be seen to exhibit similar behavior in that the rate of residual strength loss increases with temperature. While this degrade slows considerably beyond 80% loss at 65 and 75°C, it continues at a rapid rate beyond 50% loss at 85 and 95°C. These findings are in agreement with the chemical kinetic theory of first order reactions, where the rate of reaction (degradation) increases exponentially with temperature. The behavior in terms of rate and extent of residual strength loss with time is similar for **both** systems at 95, 85 and 24°C. At 65°C the rate is considerably slower for the FRP system and there was no clear pattern at 75°C. This suggests both systems are subject to similar degradation mechanisms.

To allow an assessment of each methods level of variability, the test results of all the immersed sample batches was used giving a total of 25 batches of 10 replicate samples per test method, per system. This resulted in six groups of twenty five batches. The **COV** values of each batch were determined to provide a data set for analysis. This set did not have equal variance so a Dunn's test was used to perform a multiple comparison means separation. The mean COV's and **results** of the Dunn's test are shown in Table 7. The variability of the compression shear test was significantly lower than that of block shear and glueline cleavage for both system **types**. The glueline cleavage **was** significantly higher than this for both systems. The block shear was statistically similar to compression shear for the maple / PRF system and the same as glueline cleavage for the maple / PRF / FRP system. A Mann Whitney comparison test was used to determine whether the level of variability was affected by system type. This was only found to be true for the block shear test results.

System / test combination	Mean COV (%)	Dunns pairwise comparisons	
		System	Significant difference
M block shear	9	MBS Vs. MCS	No
M compression shear	9	MBS Vs. MGC	Yes
M glueline cleavage	13	MCS Vs. MGC	Yes
K block shear	17	KBS Vs. KCS	Yes
K compression shear	10	KBS Vs. KGC	No
K glueline cleavage	16	KCS Vs. KGC	Yes

While the compression shear test exhibits very low COV it was believed that the block shear tests ability to evaluate systems independent of their integrity was a more important criteria and was selected as the most appropriate test method.

## Conclusions

This study identified modified block shear to be the most appropriate method of mechanically analyzing the integrity of adhesive bonds between wood and FRP. The results of compression shear and glueline cleavage techniques varied disproportionately with system integrity due to a mixed mode ~~of~~ applied ~~stress~~. This makes them unsuitable for studies such as this where the degradation of samples is accelerated and also for comparative studies which evaluate systems made of ranging material combinations. The ~~PRF~~ bond between these materials was found to be as durable as that between two wood members following prolonged immersions in high temperature water baths. When ~~the~~ successful history of phenolic wood laminated systems in exterior environments in ~~considered~~ this ~~study~~ would support the use of such resins for wood / FRP laminates. The comprehensive durability evaluation ~~can~~ also be ~~used~~ as a benchmark against which new wood / adhesive / FRP systems can be gauged against.

## References

ASTM D905 1996 Strength properties of adhesive bonds in shear by compression loading. Annual book of **ASTM** standards, Volume 15.06 West Conshohocken, PA.

ASTM D143 1996 Standard methods of testing small clear specimens of timber. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

ASTM D1101 1996 Integrity of glue joints in structural laminated wood products for exterior use. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

ASTM D1037 1996 Evaluating properties of wood-base fiber and particleboard materials. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

Aliyu, A.A; Daniel, I.M; 1985. Effects of strain rate on delamination fracture toughness of graphite / epoxy in delamination and debonding of materials. **ASTM STP 876**, W.S. Johnson ed. ASTM Philadelphia, 1985 pp. 336-348.

Alexander, J. P, Shaler, S. M; Gardner, D. J. 2000 Evaluating wood / FRP bond durability through chemical kinetics. **Proceedings of Wood Adhesives 2000**, Forest Products Society, Madison WI.

Barbero, E Davalos, J; Munipalle, U; 1994 Bond strength of FRP-wood interface. Journal of reinforced plastics and composites. Vol. 13 pp. 835-854.

Brown, M. 1998. Surface Characterization of pultruded FRP and its relation to bond performance with eastern hemlock. Master of Science thesis, University of Maine.

Caster, D.W; 1981. Testing and evaluation of bonded products. In Adhesive bonding of wood and other structural materials p.244. Eds: Blomquist, R.F; Christiansen, A.W; Gillespie, R.H; Myers, G.E. Materials Research Laboratory, The Pennsylvania State University, University Park, Pa 16802.

Coker, E.G; Coleman, G.P; 1935. Photoelastic investigations of shear tests of timber. Selected Eng. Papers No.174, Institution of Civil Engineers, London.

Cramer, S.M; Goodman, J.R; Bodig, J; Smith, F.w; 1984. Failure modelling of wood structural members. Struct. Res. No. 51. Civil Engineering Dept., Colorado State Univ. Fort Collins, Colo.

Daniel, I.M; Yaniv, G; Auser, J.W. 1987. Rate effects on delamination fracture toughness of graphite / epoxy composites. In composite structures - 4 (Proceedings of fourth International Conference on Composite structures, Paisley, Scotland, 1987) I.H.Marshall, ed. Elsevier Applied Science, New York, 1987.

Cavalos, J.F; Madabhushi-Raman, P; Qiao, P; 1997. Characterization of mode III fracture of hybrid material interface bonds by contoured DCB specimens. Engineering fracture mechanics, 58 (3). pp.173-192.

Davalos, J.F; Madabhushi-Raman, P, Qiao, P, Lang, E.M; 1998a. Mode I fracture toughness of fiber reinforced composite wood bonded interface. Journal of Composite Materials (in press).

Davalos, J.F; Madabhushi-Raman, P, Qiao, P; Wolcott, M.P; 1998b. Compliance rate change of linear contoured DCB specimen for Mode III fracture of bonded interfaces. Theoretical and Applied Fracture Mechanics (in press).

Gardner, D.J; Davalos, J.F; Munipalle, U.M; 1994. Adhesive bonding of pultruded fiber-reinforced plastic to wood. Forest Products Journal 44 (5) pp.62-66.

Hall, J.H; Haygreen, G.H. 1983. The Minnesota shear test. Forest Products Journal 33(9) pp. 29-32

Hunt, G.M. 1917. The preparation of specifications for propeller glue. Unpublished report, Project L-157-2. USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin.

Marra, A.A. 1992. Technology of Wood Bonding, Principles in Practice. Van Nostrand Reinhold, New York. ISBN 0-442-00797-3.

McLeod, A.M; Yolton, L.A; Sanborn, W.A; Phillips, R.S; 1956. A comparison of shearing strengths of glued joints at various grain directions as determined by four methods of test. Forest Products Laboratory Report No. 1522. Forest Products Laboratory, Madison, Wisconsin.

Miliet, A.M. Gillespie, 1978. Precision of the rate-process method for predicting bond durability. USDA Forest Service, Forest Products Lab. Madison, Wisconsin.

Northcott, P.L; 1952. The development of the glueline-cleavage test. Journal of the Forest Products Research Society. December 1952 pp. 216 - 224.

Okkenon, E. A; River, B. H. 1989 Factors affecting the strength of block shear specimens. Forest Products Journal 39(1) pp. 43-50.

Radcliffe, B.M; Suddarth, S.K; 1955. The notched beam shear test for wood. Forest products Journal. April 1955 pp. 131 - 134.

River, B.H; 1984. Accelerated, Real-time aging for four construction adhesives. *Adhesives Age*, February 1984 pp.16-21.

Smith, C.W; Kobayashi, A. S. 1987. Experimental fracture mechanics. In *Handbook of experimental mechanics*, edited by Kobayashi, A. S. Society for experimental mechanics, Prentice and Hall. ISBN 0 13 377706 5.

Soltis, L.A; Rammer, D.R; 1994. Shear strength of unchecked glued - laminated beams. *Forest Products Journal*, 44 (1) pp. 51-57.

Strickler, M.D; 1968. Specimen design for accelerated tests. *Forest Products Journal* 18 (9) pp. 84-90.

Truax, T.R. Harrison, C.A; 1922. Comparison of types of glue shear specimens. Unpublished report, Project L 157-3324. USDA Forest Service. Forest Products Laboratory, Madison, Wisconsin.

Vick, C.B; 1995. Hydroxymethylated resoranol coupling agent for enhanced adhesion of epoxy and other thermosetting adhesives to **wood**. In Christiansen, **A.W**; Conner, **A.H**; eds. *Wood Adhesives 1995* Portland, Ore. Proc. 7296. Madison, Wis. FPS 47-55.

Yavorsky, **J.M**; Cunningham, **J.H**; 1955 **Strain** distribution in Maple glue block shear specimen as indicated by a brittle lacquer. *Forest Products Journal*. February 1955 pp. 80 - 84.

Yavorsky, **J.M**; Cunningham, **J.H**; Hundley, **N.G**; 1955. Survey of *factors* affecting strength tests of glue joints. *Forest Products Journal* October 1955 pp. 306 - 311.

Yeh, B; Schniewind, A. P; 1992. Elasto-plastic fracture mechanics of wood using the J-integral method. *Wood and Fiber Science*, 24(3) pp. 364 - 376.

Part II  
**Evaluating Wood / FRP Adhesive Bond Durability  
Through Chemical Kinetics**

## Abstract

The durability of adhesive bonds between a pultruded phenolic – glass fiber reinforced polymer (FRP) and red maple (*Acer rubrum*) were evaluated. Maple arid maple / FRP laminates were exposed to various high temperature wet environments with batches of samples being removed periodically and tested for adhesive bond strength. The adhesive bond strengths were plotted as a function of exposure time to the said environments and evaluated using a chemical kinetics approach. The immersion conditions were chosen to provide a range of deterioration rates within experimentally feasible time limits. The extrapolation of strength deterioration rates from the kinetics analysis permits a durability forecast for each system in actual use conditions. Three laminate systems (maple / phenolic-resorcinol formaldehyde [PRF], maple / FRP / PRF and maple / FRP / Epoxy with a hydroxymethylated resorcinol [HMR] coupling agent) were exposed to the high temperature wet environments which ranged from 65°C to 95°C and one ambient temperature exposure. The adhesive bond strength was tested by a modified ASTM D905 block shear test.

The control maple laminates were projected to retain 70% of their **post** vacuum pressure soaked strength for 12 years. When PRF and epoxy / HMR adhesive systems were used to bond the FRP reinforcement to maple the time projections to 30% bond strength degrade were 57 and 34 years, respectively. The epoxy resin was found to reinforce the wood / FRP at the bonded interface and this system, after exposure to a vacuum pressure soak cycle had a mean shear strength of 2,058 psi which was 23% greater than the PRF system. The ability of the epoxy to make the porous FRP impermeable to water was also recognized. The rate process assessment from chemical kinetics was combined with moisture cyclings, maple / PRF / FRP laminate shear blocks were evaluated. It appears that an interaction exists between the level of accelerated aging and the stress exerted by the moisture cycling. There is considerable experience with the successful use of wood / PRF systems in outdoor use and their durability has been proven. This study indicated the PRF and epoxy bonds between maple and FRP reinforcement are more durable than those between maple and PRF.

## Introduction

The term engineered wood products (EWP's) describes a broad category of composites formed by combining resin and wood elements, which range in size from fibers to veneers through to dimensional lumber. Composites may take on the name of structural EWP's when their structural properties are confirmed by methods which go beyond visual grading, (Anon 2000). Glulam timber falls into this category, and has commercial construction applications ranging from large flat roof systems to complex arches. It performs well in demanding environments and can be used for bridges, utility poles, cross arms and marinas. North American Production is forecast to total 335 million board feet in 2000, up 12% from 1999 (Adair1999).

The combination of EWP's with fiber reinforced polymer composites (FRP) (eg. glass / phenolic pultrusions) has been shown to further increase structural performance and allow use of lower grade material (Dagher et al 1997). An advantage of combining wood with FRP is the flexibility of design properties through choices of fiber type, amount, orientation and resin type. With this flexibility comes the need to measure the durability of the composite material system, especially the bond between these two materials because of their markedly different response to moisture. The objective of this project was to devise a method which could assess the performance of different adhesives used to bond wood to FRP and to predict a product lifespan in actual use conditions.

Many products have been made which pass standard production quality control tests but, fail rapidly in service. Examples were cited (Caster and Perrine 1974, Caster 1980) one of which involved a North American beam producer who used PRF and melamine urea formaldehyde adhesives in lamination. The gluelines were tested after production and met the required quality control standards. Unfortunately these beams failed almost immediately in service and some during erection. When samples were compared against controls by a more comprehensive test based on loss of strength versus time of exposure in severe conditions (Caster and Perrine 1974) the difference in bondline performance was clear.

The rate process or kinetic approach can be used to predict the life span of a product in actual use conditions. The action of each factor affecting bond degradation is dependent on time, temperature and concentration. Thermal methods for accelerating the aging process have been applied to a wide variety of

materials to demonstrate the application of chemical kinetics to this degradation process. Thermal aging has been carried out on wood (Stamm 1956), paper (Western 1964) and other materials (Steiger 1958), which suggest the deterioration of strength properties results from chemical changes in the substance being aged. By increasing the temperature and concentration of variables, the rate at which a factor degrades a bond may be accelerated in the laboratory (Gillespie 1965), which permits a rapid assessment of product lifespan.

It was noted (Millet and Gerards 1972) that the use of accelerated aging for wood is predicted on it being an organic material comprised of an association of organic constituents, namely; cellulose, hemicellulose and lignin. The assumption is that the degradation of wood is a consequence of chemical changes in one or more of these constituents and follows a first order reaction rate. If one of these changes is rate determined, then it may be possible to relate the overall rate at different elevated temperatures by an empirical equation proposed by Arrhenius in 1890 and to use this relationship to predict system degrade at normal service temperatures. The workings of lifespan prediction used in this study are given in the material and methods section.

The Arrhenius equation is usually written as;

$$\frac{d \ln k}{dT} = \frac{E}{RT^2}$$

Where; k = rate constant

E = the activation energy (kilocalories per mole)

R = the molar gas constant (calories per gram per mole)

T = the absolute temperature (°K)

A plot of  $\log k$  versus  $1/T$ , should yield a straight line with slope equal to  $-E/4.57$  (Gillespie 1965). The temperature coefficient will be expressed in kilocalories per mole (E), It has not been proven that the systems examined in this study are subject to the same degradation mechanism, which brings the direct application of the Arrhenius equation into question. However, if there is scope to modify the equation the overall approach is very useful in extrapolating to temperatures other than those used for determining rate losses experimentally, provided the data are not extended greatly beyond the temperature range evaluated.

An early study (Stamm 1956) examined the thermal degradation of wood and cellulose. The degradation of the major components of wood ranged around the degradation of wood itself; ie. hemicellulose degraded four times as fast, a cellulose at the same rate as wood and lignin at only half the rate. The laminates examined in this study were bonded with phenolic and epoxy resins which have ~~been~~ found in the past to be more durable than the materials which they bond (and wood in particular), (Vick 1995, Gardner et al. 1994). It was subsequently expected that the rate of degrade was largely a function of the rate of wood / FRP deterioration. Stamm's work indicated that wood followed the first order kinetic rate of decay and gives validity to the application of this method in the analysis of the laminations.

Gillespie (1965) introduced the rate process technique and showed how it accurately describes the response of plywood test specimens to a range of moisture and temperature combinations. The study drew a number of conclusions; **1)** the log of wet shear strength under the experimental conditions used varied linearly with time of exposure to water-soaking, **2)** the temperature dependence of rate of strength ~~loss~~ due to water soaking conformed to the Arrhenius relationship, **3)** the adhesives studied could be ranked for water sensitivity on the basis of their degradation rates, **4)** this ranking **was** the same order indicated by past end-use experience and **5)** water soaking caused much greater rates of strength loss than shown by preliminary results with dry heat.

In a similar study small clear specimens of **wood** were exposed to **dry** heat from 115 to 175°C for a range of time periods (Milet and Gerhards 1972). Modulus of rupture was measured following exposure and was found to decrease with increasing time **of** exposure for **all** species. A series of Arrhenius plots were made with this data and the straight line relationships obtained indicate that despite an undoubted complexity and multiplicity of chemical reactions taking place within the degrading wood substance, the measured rates of overall change in physical properties proceed in an orderly manner.

Rate process derived estimates of the time required for joints of four elastomer based mastic type construction adhesives to lose half their original strength were compared with the actual performance of the adhesives over an eleven year period in three service conditions (River 1984). The rate process estimates were found to successfully rank three of the four adhesives. The inaccurate estimate was believed to be the

result of a change in the degradation mechanism (for one adhesive type) between the **2PC** and the lowest wet accelerated aging temperature of 40°C.

Solid and bonded pieces of hard maple have been predicted to retain 75% of their structural integrity for many thousands of years under dry room temperature conditions (Millet and Gillespie 1977). This was limited to tens of years with wet exposure. They formed Arrhenius plots using times to both 25 and 50% strength loss. The curves for the wet exposure samples showed a constant difference between the lines for 25 and 50% loss in strength over the entire temperature range. This was not the case with testing of dry samples indicating a more constant reaction mechanism over all the levels of thermal degradation for wet exposures.

A possible flaw in the rate process technique is the absence of alternating **stresses** on the bonded area through a cycling of the wood moisture content. An automatic **boil** test machine as described (Kreibich and Freeman 1968) was used in an attempt to determine the affect of this apparent deficiency (Northcott 1968). **This** study compared ten different accelerated aging schedules on the basis of their ability to rank a series of ten adhesives in accordance with their known order of durability in exterior service. Three laboratories participated in the study **so** as to provide a measure of the reproducibility of the procedures. They concluded that the supposed deficiency, resulting from failure to incorporate some manner of cyclic stressing of the bonded area during accelerated aging was not significant; at least when results are based on laboratory sized specimens, (Millet and Gillespie 1978).

More recently (Winandy 1998) has developed and verified kinetic models which predict the effects of fire retardant on the bending strength of wood subjected to on-going thermal degradation during exposure to elevated temperatures in service. The findings are currently being introduced into US design codes.

## Materials and Methods

Wood species: A species having high shear strength was required to prevent wood failure at a low level of stress. Red maple (*Acer rubrum*) was chosen on this basis. The lumber was locally sourced and delivered in flatsawn ten foot boards of 1 X 6 inch nominal dimensions. These were straight grained, select grade or better and conditioned to 8% m.c. prior to knife planing 24 – 48 hours before lamination.

FRP: A pultruded material supplied by Strongwell, Virginia was used. The pultrusion process produced a continuous band of FRP 0.13 inches thick and 4.76 inches wide. It consisted of a uni-directional E-glass core contained in a phenolic resin matrix which was encased by chopped fiber mat veil on each surface. It was passed through a drum sander to remove gloss and debris from the surfaces of the material. The surfaces were acetone wiped prior to a priming treatment appropriate to the resin system used in bonding.

Resin and Priming systems: Two resin systems were chosen for examination in this study with selection made on the basis of promising performance when bonding wood and FRP in previous studies. PRF resins were examined in dry conditions (Brown 1998) and in wet and dry conditions (Gardner 1994). The epoxy / HMR combination was found to perform well in both wet and dry tests (Vick 1994 and 1997).

The PRF was used as a primer and an adhesive, the mix is given in Table 8. It was used to prime the FRP in the maple / PRF / FRP system and as an adhesive to bond the maple / PRF and the maple / PRF / FRP systems. The priming coat was applied at a rate of approximately 0.05 g/inch<sup>2</sup> and was drawn across the FRP surface with a metal scrape. This coat was allowed a curing period of 48 hours at ambient temperature prior to lay-up. When used as an adhesive it was applied at a rate of 0.25 g/inch<sup>2</sup>, the manufacturer recommended it be applied to wood of between 8 and 12% m.c.. The laminations were placed in a press after lay-up and clamped at 100 psi for 24 hours at ambient temperature.

The Epoxy and HMR recipes were developed at the Forest Products Laboratory (Vick 1995). The constituents are detailed in Table 8. HMR primer was applied to the maple and FRP at a rate of 0.08g/m<sup>2</sup> by brush coating. These were given a 24 hour drying period in ambient conditions prior to lay-up. The HMR's method of coupling the adherends and the epoxy is described by the inventor (Vick 1995). The epoxy was applied at a spread rate of 0.29 g/inch<sup>2</sup> and hand clamped to the point of initial resin squeeze out in

ambient conditions for 24 hours and then given a post cure at 65°C for 5 hours. To avoid stresses on the bondlines from shrinkage of wood the EMC of the wood was maintained at 8%.

Table 8 PRF, Epoxy (resins) and HMR (primer) formulations

Resins		Epoxy		Primer	
	PRF Parts by weight (g)		Parts by weight (g)		HMR Parts by weight (g)
Hardner	12	DGEBA resin	100	dH <sub>2</sub> O	90.43
Water	18	Benzyl alcohol	12.5	NaOH 3M	3.34
Resin	70	Hydrophobic fumed silica	2.5	Formaldehyde (37%)	3.79
---	---	Triethylenetetramine hardner	11.1	Resorcinol (crystalline)	2.44
---	---	---	---	Dodecyl sulfate sodium salt, (added after reactiontime)	0.5% of total mix weight

### Sample Preparation, Conditioning and Testing

Laminations measuring 4.75 X 23 inches were made up and a central 2 inch **strip** cut from each to minimize edge effects. Rate process analysis may be conducted using high **temperature** dry or wet aging, wet was chosen in previous work (Millet and Gillespie 1978) as a more constant reaction mechanism was shown to occur with these conditions. Nine hundred shear **Mocks** suitable for testing by a modified ASTM D905 method were cut from these strips. The exposed edges of the FRP material were sealed with a vinyl ester resin to prevent excessive water penetration into the material through capillary action. Zero time control specimens were tested in wet and dry conditions. Those samples immersed in high **temperature** water baths were allowed to cool in a bath of tap water prior to testing wet. The laminate origin of each shear block was recorded and the samples *randomized* on this basis, a replication factor of 10 was used. The blocks had a shear area of three inches' with peak load and failure mode being recorded for each test using a 0.2 inch per minute rate of deformation.

Stainless steel tanks (250 liters) were used to immerse the sample batches at each of the elevated temperatures. Circulating waterbaths maintained the temperatures ( $\pm 0.5^\circ\text{C}$ ). Plastic tanks were used for the immersion of samples at room temperature. While these required no hot water circulation a circulating

pump was placed in each to avoid stagnation. VWR brand Algicide was used throughout to limit microbial growth.

Sample batches were vacuum pressure soaked and placed in mesh containers prior to immersion. They remained immersed for the defined period; after which they were, cooled, bagged and tested **wet**. The temperatures and periods of immersion are given in Table 9. As previously mentioned **all** samples were vacuum pressure soaked prior to immersion. This step was taken to ensure that those samples immersed for the shorter time periods had the same level of water impregnation as those **immersed** for the longer periods. These temperatures and times of immersion were based on a previous evaluation of bonded wood systems (Millet and Gillespie 1978).

Table 9 Sample immersion periods at each temperature.

Temperature (°C)	Immersion period (hours)				
	1	2	3	4	5
95	6	12	42	90	148
85	24	48	144	340	484
75	144	600	936	1152	1752
65	336	1176	1776	2326	2760
24	336	1176	1776	2326	2760

## Results and Discussion

### Rate Process Analysis

An exponential decay regression equation ( $y = ae^{-bx}$ ) was used to fit the rate of residual strength loss versus time of immersion at each elevated temperature (Figure 6). The nonlinear coefficients (a and b) were determined using the Marquardt-Levenberg algorithm provided by the SigmaPlot software package. The time for each system to achieve a 30% drop in residual strength at each temperature was calculated from the appropriate regression. An exponential growth regression of these times (in days) versus the inverse of absolute temperature  $\times 10^4$  ( $K \times 10^4$ ) was then made using an exponential growth equation ( $y = ae^{bx}$ ) which used the same Marquardt-Levenberg algorithm to determine coefficients of the independent variable (Figure 7). The resultant regression of time to 30% strength loss versus temperature was used to predict time to 30% strength loss at 20°C (Table 12).

The shear strengths and corresponding failure modes of the wet and dry control batches are summarized in Table 10, the wet values are termed the zero time strength as the samples have been VPS'd but not immersed. Of the two adhesives used to bond the maple to FRP, the epoxy / HMR combination gave a significantly stronger bond than PRF ( $p < 0.001$ ) following a VPS treatment and a statistically similar bond strength in dry conditions ( $p = 0.24$ ). A greater reduction in strength can be seen for the maple / PRF system following the VPS than for either of the two systems containing an FRP plaque. This was unexpected particularly in light of preliminary experiments which found the width of maple to increase by up to 8.8% following a vacuum pressure soak treatment while the FRP swelling was negligible in all planes. This differential would be expected to create a substantial **stress** on the **bondline** interface between the two materials causing this type of system to be more sensitive to VPS than the maple / PRF system. However, Table 10 shows this was not the case in this series of experiments, suggesting that both adhesives have sufficient ductility to cope with this differential. The predominant failure mode for the maple / PRF and maple / epoxy / FRP systems was in the wood material and in the continuous strand mat (CSM) of the FRP for the maple / PRF / FRP system.

System	Zero time mean block shear strengths (psi). COV in parentheses(%)			
	Dry		Wet	
	Strength (psi)	Failure mode'	Strength (psi)	Failure mode*
Maple/ PRF	3098 (6.4)	74%W 26%A	1710 (3.3)	59%W 41%A
Maple / PRF/ FRP	2051 (16.0)	56%FS 44%A	1578 (7.4)	60%CSM 40%W
Maple / Epoxy-HMR/ FRP	2211 (19.6)	80%W 20%F	2058 (7.0)	87%W 13%F

All systems displayed similar rates of degrade which increased dramatically with temperature (Figure 6) This behavior is typical for reactions which follow a first-order chemical kinetic mechanism of degradation (Gillespie 1965) validating the model approach used in this study. However, the decay rates of the systems evaluated in this study deviated from the typical degradation curves associated with 1<sup>st</sup> order chemical kinetic behavior. All systems show a rapid initial rate of degrade which slows with time but fails to produce a linear relationship even when plotted on a y-axis with a logarithmic scale. Such a response is discussed in ASTM D4502 which sites relief of internal stresses and rapid initial chemical degradation as common causes.

A linear y-axis is used here to create curves which allow for easier distinctions to be made between the rates of degrade.

Figure 6A is included to show a typical data series behind each of the curves and a visual regression shows that the two parameter exponential decay equation used in Figure 6B-D is a good representation. While the rates are similar for all systems a comparison of Figure 6C & D highlights the greater absolute strength retention of the epoxy. Subsequent analysis normalizes residual strength as a opposed to an absolute value to facilitate comparisons between the systems studied.

The two parameter exponential equation fitted the measured response with two notable exceptions. The fit of the curve for the epoxy system immersed in 95°C water Figure 6D was very poor ( $R^2 = 0.31$ ). Examination of the data revealed that degrade was occurring at a much greater rate than for the other two systems, indicating a differing degradation mechanism at this high temperature from that at lower temperatures. Epoxys mechanical properties were found to deteriorate at a faster rate than phenolics in high temperature / high moisture environments (McKague et al. 1973). The glass transition temperature ( $T_g$ ) of room temperature cured epoxies is typically 50-60°C (Pocius 1997) and immersion in the 95°C waterbath exceeds this by a considerable amount. When  $T_g$  is exceeded by such a degree the free volume increases allowing water penetration which can lead to partial plasticization of the resin (Pocius 1997). The experimental data for the epoxy / HMR system at 95°C was dropped from further analysis on this basis.

While the two lower elevated immersion temperatures (65 and 75°C) are also greater than the epoxy  $T_g$  their rates seem to be similar to those of the two PRF bonded systems. However, the degradation mechanism would be expected to change below the  $T_g$  and proceed at a reduced rate so the forecast of shear strength retention is somewhat conservative.

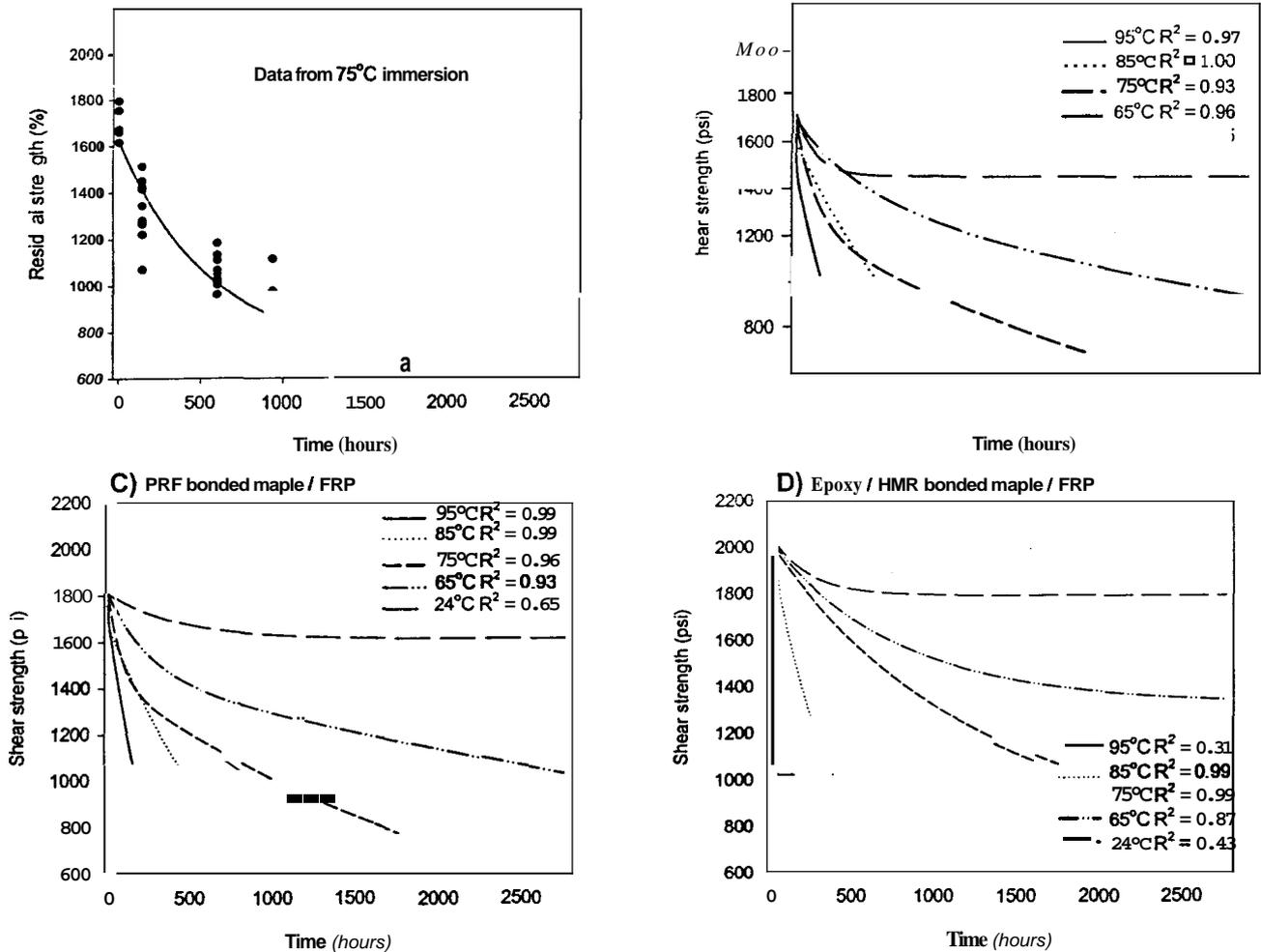


Figure 6 Typical scatter of block shear test results behind a plotted exponential decay curve of strength loss as a function of time of immersion. B - D, Exponential decay curves plotted for three laminate systems as a function of time of immersion in water baths maintained at 95, 85, 75, 65 and 24°C.

The  $R^2$  correlation was also poor for all three systems at 24°C. This was expected as strength degradation did not occur exponentially at this temperature over the time scale measured. The shear blocks (24°C immersion) tested after the first removal time (336 hours) showed an approximate drop of 10% which was found to proceed at an almost negligible rate with increasing time. This behavior was true for all three systems. The pH of the water baths was found to become more acidic with time and reached a peak of 4.6 in the 95°C bath. The pH change was due to extractive leaching, liberation of acetic acid and mobilization of hemicelluloses (McDonald et al. 1999).

The data from the degradation assessment were used in a rate process analysis that went beyond relative comparisons and permitted a theoretical forecast of product lifespan. This was determined by presenting all the test results obtained from the samples which had been immersed at the four elevated temperatures for increasing periods of time as a percentage of the original shear strength (taken from the *VPS'd* controls). This data set **was termed** the residual strength and **was** plotted versus time Figure 7 shows the maple / PRF / FRP system which was representative of the other two systems. Using a logarithmic scale for residual strength, the rates can be seen to approach linearity, true to first-order kinetic principles. The time required to achieve a 30% loss in residual strength for each system at the four elevated temperatures was predicted by the **fit** equation. These times are given in Table 11.

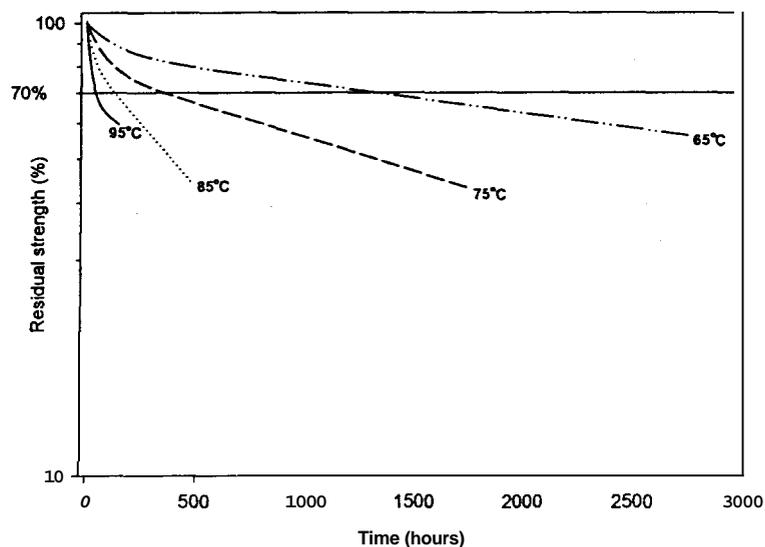


Figure 7 Residual strength loss versus time for maple / PRF / FRP block shear specimens.

An exponential growth regression was then performed for these times (given in Figure 8) versus the inverse of absolute temperature to obtain a regression equation which was used to predict the time to 30% loss of

strength when the laminations are immersed at room temperature. The equations and resultant times to this level of residual shear strength are shown in Table 12. A visual assessment indicates a similarity and when the experimental error involved in block shear testing (Figure 6A) is considered this impression would seem justified. However, when the equations are extrapolated to 20°C as in Table 12 the times required to achieve a 30% decrease are markedly different and the effect of apparently minor differences in the initial data are shown to have a significant influence when extensive extrapolations are made. Table 11 shows the maple / PRF system to be more sensitive to the lower temperature of the range evaluated causing its projected value to be the lowest. This system was predicted to experience a 30% loss in residual shear strength after 12 years immersion at 20°C which is a shorter time than that predicted previously (Millet and Gillespie 1978) who used a similar accelerated aging technique in the evaluation of phenolic bonded shear blocks. They concluded that the phenolic bonded maple would retain 75% of its initial shear strength for at least 40 years. Their use of hard maple (*Acer saccharum*) versus soft maple (*Acer rubrum*) used in this study and the tendency for the extrapolation method to magnify any experimental error may account for this discrepancy.

The time projections for both the maple / FRP / PRF and maple / FRP / Epoxy systems are longer than that of the phenolic bonded maple system and comparable to that found previously (Millet and Gillespie 1978). Phenolic wood bonds have long been considered suitable for service in exterior locations and there is considerable experience of their successful use in such environments. This study would indicate that both the maple / FRP system bonded with either the PRF or epoxy adhesives is as durable as phenolic bonded maple and should be considered suitable for use in exterior environments. The much greater initial shear strength of the epoxy system should be considered when its shorter projection to a 30% drop in residual strength is considered. Further support for the projections can be taken from Figure 6B-D. This shows laminates immersed in room temperature water baths exhibit an initial drop in strength followed by a sustained period of an almost negligible rate of loss which supporting the relatively long time projections made in Table 12. However, this twelve year prediction for the maple / PRF system is below the lifespan which such systems realize in actual use and may be explained by the amplification of environmental degradation induced by edge effects and the forecast being based on a water soaked condition.

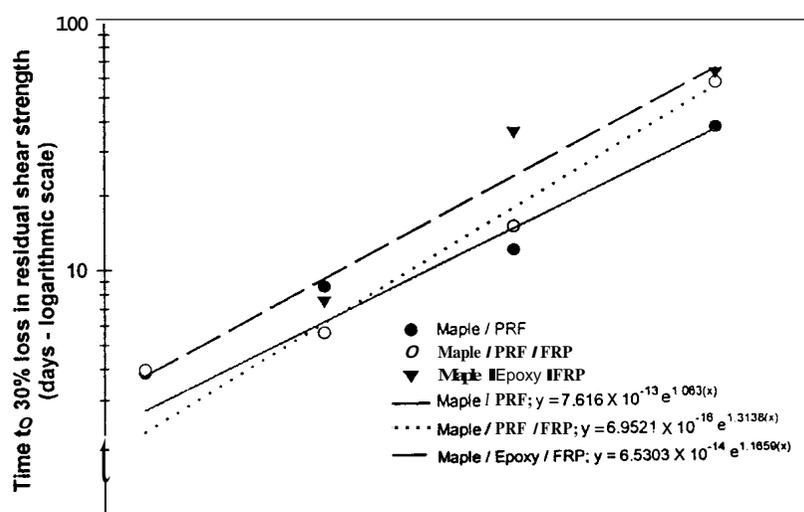


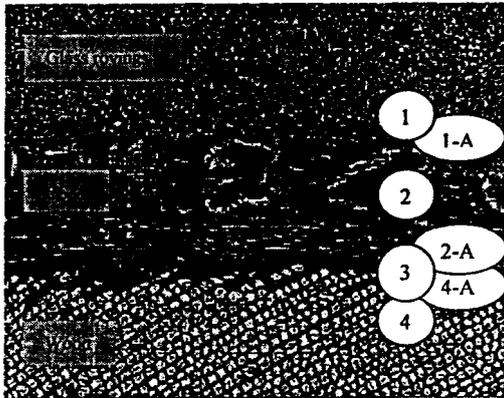
Figure 8 Regressions of time to a 30% decrease in residual shear strength for three laminate systems using a two parameter exponential decay equation.

Table 11 Predictions of time to 30% loss in block shear strength.

System	Equation	R <sup>2</sup> , goodness of fit	Predicted time to 30% loss in shear strength due following a 20°C immersion (years)	Primary failure mode
Maple / PRF	$y = 7.616 \times 10^{-13} e^{1.063(x)}$	0.98	12	Wood
Maple / PRF / FRP	$y = 6.9521 \times 10^{-16} e^{1.3138(x)}$	0.99	57	FRP continuous strand mat
Maple / Epoxy / FRP	$y = 6.5303 \times 10^{-14} e^{1.1659(x)}$	0.89	34	Wood

## Failure Mode Analysis

The anatomy of the bond interface in a wood / adhesive / FRP-reinforcement is similar to that described for wood / adhesive / wood bonds (Marra 1992). There are a number of links in such a system and failure will occur at the weakest, these are shown on the microtomatograph of a wood / FRP / phenolic system (Figure 9). The intra-adhesive bonds from previous work (Marra 1992) are not included in this figure as they could not be identified through visual examination of failure surfaces. Failure of the maple / PRF / FRP system occurred consistently at link 2-A (resin penetrated CSM) while link 4 (wood) was found to be the weakest for the maple / Epoxy / FRP system. It is believed the failure mode is strongly influenced by the contrasting curing behavior and mechanical properties of the two adhesive systems, which has a marked effect on the adherend / adhesive interface strength.



### Links (and hypothetical strength ranking)

- 1 Glass rovings in a phenolic matrix (1)
- 1-A Interface between rovings and chopped strand mat (CSM) (2)
- 2 Porous CSM (=5)
- 2-A Adhesive / CSM interface (=5)
- 3 Adhesive (3)
- 4-A Adhesive / wood interface (=5)
- 4 Deep wood (4)

Figure 9 Microtomatograph of a fiber-reinforced polymer / PRF / wood laminate interface and a hypothetical ranking of the integrity of the system components, (= indicates a joint ranking).

The epoxy resin had a low viscosity (1100 cps) and was presumably able to penetrate the permeable structure of the CSM layer of the FRP (link 2-A) material and increase its interlaminar shear strength (ILS).

There are no explicit formulas available for the ILS of random composites such as the CSM and it is typically approximated by the shear strength of the matrix (Barbero 1998). It is believed to be a function of;

1. The ratio of interlaminar shear modulus of the matrix ( $G_M$ ) and the interlaminar shear modulus of the fibers ( $G_F$ )
2. The fiber volume ( $V_F$ )
3. The void volume ( $V_V$ )

The ILS increases with an increase in  $G_M$  or a decrease in  $G_F$ ,  $V_F$  or  $V_r$ . Penetration of the epoxy resin into the porous mat (link 2-A) increases the interlaminar shear modulus of the matrix and decreases the void volume. The occurrence of a typically deep wood (link 4) failure indicated the interlaminar shear strength of the epoxy impregnated CSM is greater than that of maples. This observance of a deeper wood failure than with the maple / PRF system suggests the epoxy may also **be** reinforcing the permeable subsurface of the wood (link 4-A) in the same way as the CSM.

The PRF has a higher viscosity (2,200 cps) which reduces penetration into the permeable material surfaces and its condensation cure reaction causes it to shrink after application. Epoxy resins are known to exhibit < 4% shrinkage during cure (Pocius 1997). These factors combined with the lower modulus of the PRF resin in comparison to the epoxy prevent a significant increase in the interlaminar shear strength of the CSM (link 2-A). The shear strength of a maple / adhesive / FRP-reinforcement system is able to realize is dependant on its weakest link. A hypothetical ranking (**based** on working knowledge) from strongest to weakest **was** included on Figure 4. The epoxy / HMR system is shown to strengthen the grouping **of** the three weakest links leading to a deep wood failure and greater shear strength. The deep wood (link 4) failure **causies** the failure zone to **be** outside the plane of maximum **stress** concentration in testing and is an additional contributing factor.

A second effect of epoxy's gap filling characteristic is a sealing of the FRP surfaces, which when combined with the vinylester edge sealing makes the material impervious to water and less susceptible to chemical attacks. The attack of hydrochloric acid on laminates of polyester resins and E-glass fibers was observed (Ehrenstein et al. 1990). Water,  $H^+$  and  $Cl^-$  ions were able to diffuse through the resin matrix and reach the **glass** fibers which were attacked under cation exchange causing the cations of the E-glass to go into solution. The resin **acted** as a semi-permeable membrane (or as a strongly permeation retarding skin) and therefore limited a back diffusion of the ions which dissolved out of the glass. The hydrochloric acid accumulated along the glass fiber and tended to dilute (osmosis). This was only possible by diffusion of more hydrochloric acid from outside which increased the pressure of the salty solution within the laminate. Finally blisters occurred starting presumably from the smallest defect spots. These burst when pressure was high enough, causing cracks in the laminate, weakening the matrix and exposing the glass fiber to further chemical attack. The CSM of the maple / PRF / FRP system remains permeable due to significant adhesive

shrinkage (on curing). The pH of the water was found to reach 4.6 which is considerably less acidic than the dilute HQ documented in this case study. However it is hypothesized that the CSM is subjected to the same mechanism, albeit at a lesser rate.

#### Incorporation of Moisture Cycling into the Rate Process Analysis

The rate process analysis takes no account of the shrinkage and swelling stresses which wood / FRP laminates are subject to in actual use. This section was conducted to establish the change in degradation rate when a severe moisture cycling process was involved and whether the effects of immersion time and moisture cycling are additive or interactive. The trials were conducted at 24 and 85°C and immersion times of 24, 72 and 144 hours used. Three exposure series were made at each; ① continuous to provide a benchmark and allow comparison with previous data. ② the samples were given a dry (71°C) / VPS cycle prior to testing after each interval. And ③ the samples were tested after two dry / VPS cycles following a 72 hour immersion and three cycles following a 144 hour immersion.

The intention was to isolate the damage imparted by these discrete cycling events from that induced by continuous exposure and this is plotted in Figure 10. The decrease in residual shear strength due to continuous exposure ① is shown by the spline curve. The decrease in residual shear strength at the three removal times for immersion series ② minus the decrease due to continuous exposure ① is isolated as that attributed to the dry soak cycle. The decrease in residual shear strength at the three removal times for immersion series ③ minus the decrease due to immersion series ① is attributed to multiple dry soak cycles. Continuous exposure results are in agreement with previous results (Figure 6C) where degradation rate is initially rapid at high temperature and slows with time. At the lower temperature there was an initial gradual decrease followed by a sustained period of minimum change.

At 24°C, time of immersion seems to have a minimal effect on strength loss imparted by one dry / soak cycle (②), at 85°C this effect decreases with time. At the lower temperature, damage induced by multiple cycles (③) which was beyond that attributed to the rate effect increases with number of cycles, while at the higher temperature this same damage due to multiple cycles (③) increases and then falls. This implies an interaction between rate (being dependant on time and temperature) and moisture cyclings which is believed to be a function of increasing system degradation with increasing rate. As the maple loses integrity

the stress it is able to exert on the PRF bondline and FRP veil is reduced, leading to a reduction in damage imparted by dry / soak cycles with time.

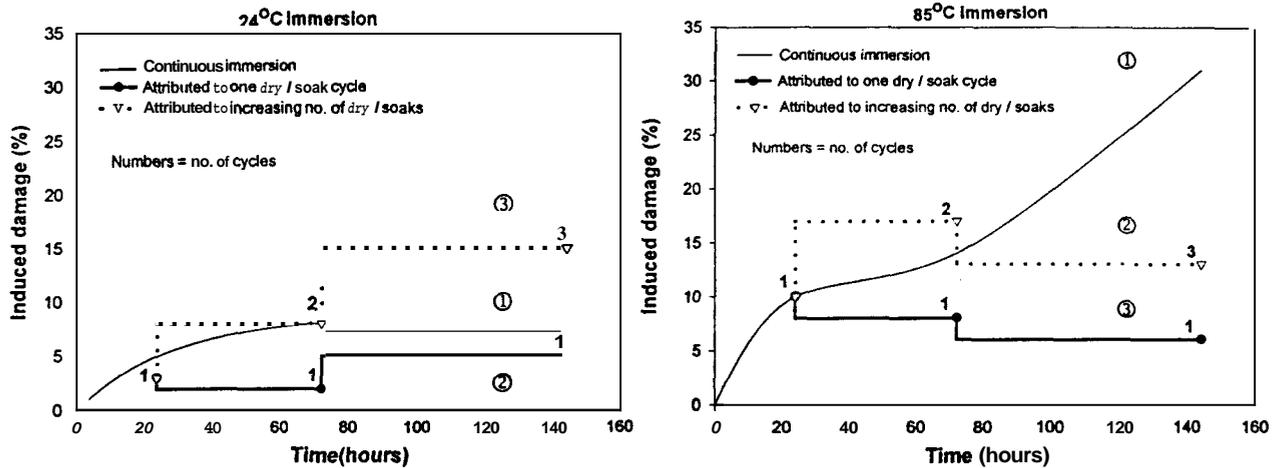


Figure 10 Combined rate process / moisture cycling effect on maple / PRF / FRP block shear specimens immersed at 24 and 85°C.

It is possible to implement the results and methodology presented here in further work evaluating this type of composite system. The rates of degrade have been found to be dependant on temperature. For the two FRP containing systems these rates occurred at differing rates which was largely attributable to the failure mode, this occurred in the FRP surface veil for the PRF bonded specimens and in the maple for the epoxy. It is proposed that these decay rates be used as master curves for new similar systems, the effect of moisture cycling at selected elevated temperatures could then be examined for the new system. Damage due to each component (ie. rate and cycling) can then be separated using the master curve, in a way similar to that shown in Figure 10.

## Conclusions

This study was conducted to evaluate the durability of a PRF and epoxy / HMR resin systems when bonding wood to FRP material. The epoxy resin was found to reinforce the material at the bond interface and had a greater dry and post VPS shear strength (2,211 and 2,058 psi respectively versus 2051 and 1578). The

epoxy also made the FRP impermeable to water penetration and therefore less susceptible to chemical degrade. A chemical kinetics analysis predicted times to 30% loss in shear strength for maple / PRF, maple / PRF / FRP and maple / epoxy / FRP systems to be 12, 57 and 34 years respectively. The maple / PRF system meets current durability requirements and it can be concluded from this study that both PRF or an Epoxy / HMR system can be used to bond FRP to maple in the lamination of reinforced glulam beams for use in exterior environments. The interaction of continuous immersion and moisture cycling were combined and as the maple **lost** integrity with time the stress it was able to exert on the bond interface diminished.

An underlying goal of this project was to develop a method of accelerating the aging of bonded systems which went beyond the wet / dry cyclic approach to **durability** assessment, and was based on a worst case scenario in terms of high moisture levels. The method **described** simulates material degrade induced by service in exterior environments and describes how moisture cycling may be incorporated into this to gain an understanding of how systems respond to cyclic stresses with increased degrees of accelerated aging. While the **full** experimental design is acknowledged as being time consuming it is shown how the assessment may be made by moisture cycling at one rate of accelerated aging. This **is** proposed **as a** comprehensive qualification method for systems requiring a high level of durability.

## References

- Anon 2000. Engineered wood products defined. Engineered wood products. [www.apawood.org](http://www.apawood.org)
- Adair, C. 1999. Engineered wood and lumber forecast. Industry data, News and information center. [www.apawood.org](http://www.apawood.org)
- ASTM D905 1996 Strength properties of adhesive bonds in shear by compression loading. Annual book of ASTM standards, Volume 15.06 West Conshohocken, PA
- ASTM D4502 1992 Standard test method for heat and moisture resistance of wood-adhesive joints. Annual book of ASTM standards, West Conshohocken, PA
- Barbero, J. E. 1998 Introduction to Composite Materials Design. Taylor and Francis, Inc. PA. ISBN 1-56032-701-4.
- Brown, M. 1998. Surface Characterization of pultruded FRP and its relation to bond performance with eastern hemlock. Master of Science thesis, University of Maine.
- Caster, D. Perrine, M.J; 1974. Screening wood adhesives with an automatic boil test. Adhesives Age, May 1974, pp.47-48.
- Caster, D; 1980. Correlation between exterior exposure and automatic boil test results. Proceedings of 'Wood adhesives research – application and needs' symposium. Madison, Wisconsin. Sponsored by USDA Forest Service and Washington State University.
- Dagher, H. J; Kimball, T. E; Shaler, S . M; Abdel-Magid, B. 1997 Effect of FRP reinforcement on low grade eastern hemlock beams. [www.fpl.fs.fed.us/documents/PDF1996/Daghe96b.pdf](http://www.fpl.fs.fed.us/documents/PDF1996/Daghe96b.pdf)
- Ehrenstein, G. W; Schmiemann, A; Bledzki, A; and Spaude, R. 1990 Corrosion Phenomena in Glass-Fiber-Reinforced Thermosetting Resins, in Handbook of Ceramics and Composites, Volume 1. Ed. Nicholas P. Cheremisinoff. Marcel Dekker.
- Gillespie, R.H. 1965. Accelerated aging of adhesives in plywood-type joints. Forest Products Journal, 15(9) pp.369-79.
- Gardner, D.J; Davalos, J.F; Muniapalle, U.M; 1994. Adhesive bonding of pultruded fiber-reinforced plastic to wood. Forest Products Journal 44 (5) pp.62-66.

Kreibich, R.E; Freeman, H.G. 1968. Development and design of an accelerated boil machine. *Forest Products Journal* 18(11) pp.24-26.

McDonald, A. G; Clare, A. B and Meder, A. R. 1999. Chemical characterization of the neutral water soluble components from radiata pine high temperature TMP fiber. In Proceedings of the 53rd General APPITA Conference. Rotorua, New Zealand. (19 –22 April. Vol. 2, pp. 641 – 647).

McKague, E. L. Jr.;Reynolds, J. D; Halkias, J. E. 1973 Thermo mechanical testing of plastics for environmental resistance. *Journal of Testing and Evaluation*. Vol 1. No.6 pp.468-471.

Marra, A. A. 1992 *Technology of wood bonding, principles in practice*. Van Nostrand Reinhold, New York. ISBN 0-442-00797-3

Millet, MA. Western, L.J. Booth, J.J; 1967. Accelerated aging of cellulosic materials: design and application of a heating chamber. *TAPPI* 50(11) pp.74a-80a.

Millet, AM. Gerhards, G.C; 1972. Accelerated aging: Residual weight and flexural properties of wood heated in air at 115 – 175°C. *Wood Science* 4(4) pp.193-201.

Millet, A. M; Gillespie, R. H. 1978. Precision of the rate process method for predicting bondline durability. Prepared for the Department of Housing and Urban development by the Forest Products Laboratory, Madison, Wisconsin.

Millet, AM. Gillespie, R.H. River, B.H; 1977. Evaluating wood adhesives and adhesive bonds. Report number 1057. USDA Forest Service, Forest Products Lab. Madison, Wisconsin.

Northcott, PL Kreibich, R.E. Currier, R.A; 1968. First replication comparing bond-degrade-accelerating systems. *Forest Products Journal* 18(5) pp.58-65.

Pocius, A. V. 1997 *Adhesion and adhesives technology*. Hanser / Gardener Publications, Inc. Cincinnati. ISBN 1-56990-212-7

River, B.H; 1984. Accelerated, Real-time aging for four construction adhesives. *Adhesives Age*, February 1984 pp.16-21.

Stamm, A.J. 1956. Thermal degradation of wood and cellulose. *Industrial and Engineering Chemistry*, 48(3) pp. 413-417.

Steiger, F.H. 1958. The Arrhenius equation in accelerated aging studies. *American dyestuff reporter*, 47 pp. 287-290.

Vick, C.B; 1995. Hydroxymethylated resorcinol coupling agent for enhanced adhesion of epoxy and other thermosetting adhesives to wood. In Christiansen, A.W; Conner, A.H; eds. Wood Adhesives 1995 Portland, Ore. Proc. 7296. Madison, Wis. FPS 47-55.

Vick, C.B; 1997 More durable epoxy bonds to wood with hydroxymethylated resorcinol coupling agent. Adhesives Age, July 1997 pp.24-29.

Western, J.L; 1964. Application of chemical kinetic principles to accelerated aging of cellulosic materials. M.S. Thesis, Department of Chemical Engineering, University of Wisconsin, Madison, Wisconsin.

Winandy, J. E. Using kinetics-based models to address serviceability concerns for fire retardant treated wood at elevated in-service temperatures. In Proceeding of 5<sup>th</sup> World Conference on Timber Engineering, Volume 1. August 17 - 20, 1998. Montreux, Switzerland. Eds. Natterer, J. and Sandoz, J-L.

## Bibliography

Adair, C. 1999. Engineered wood and lumber forecast. Industry data, News and information center. [www.apawood.org](http://www.apawood.org)

Anon 2000. Engineered wood products defined. Engineered wood products. [www.apawood.org](http://www.apawood.org)

ASTM D143 1996 Standard methods of testing small clear specimens of timber. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

ASTM D4502 1992 Standard test method for heat and moisture resistance of wood-adhesive joints. Annual book of ASTM standards, West Conshohocken, PA.

ASTM D1101 1996 Integrity of glue joints in structural laminated wood products for exterior use. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

ASTM D1037 1996 Evaluating properties of **wood-base fiber** and **particleboard** materials. Annual book of ASTM standards, Volume 4.10 West Conshohocken, PA.

ASTM D4502 1992 Standard test method for heat and moisture resistance of wood-adhesive joints. Annual book of ASTM standards, **West** Conshohocken, PA.

Aliyu, A.A; Daniel, I.M; 1985. Effects of strain rate on delamination fracture toughness of graphite / epoxy in delamination and debonding of materials. ASTM STP 876, W.S. Johnson ed. ASTM Philadelphia, 1985 pp. 336-348.

Alexander, J. P, Shaler, S. M; Gardner, D. J. 2000 Evaluating wood / FRP bond durability through chemical kinetics. Proceedings of Wood Adhesives 2000, Forest Products Society, Madison WI.

Barbero, J. E. 1998 Introduction to Composite Materials Design. **Taylor** and Francis, Inc. PA. ISBN 1-56032-701-4.

Barbero, E; Davalos, J; Munipalle, U; 1994 Bond strength of FRP-wood interface. Journal of reinforced plastics and composites. Vol. 13 pp. 835-854.

Brown, M. 1998. Surface Characterization of pultruded FRP and its relation to bond performance with eastern hemlock. Master of Science thesis, University of Maine.

Caster, D.W; 1981. Testing and evaluation of bonded products. In Adhesive bonding of wood and other structural materials p.244. Eds: Blomquist, R.F; Christiansen, A.W; Gillespie, R.H; Myers, G.E. Materials Research Laboratory, The Pennsylvania State University, University Park, Pa 16802.

Caster, D. Perrine, M.J; 1974. Screening wood adhesives with an automatic boil test. Adhesives Age, May 1974, pp.47-48.

Caster, D; 1980. Correlation between exterior exposure and automatic boil test results. Proceedings of 'Wood adhesives research - application and needs' symposium. Madison, Wisconsin. Sponsored by USDA Forest Service and Washington State University.

Coker, E.G; Coleman, G.P; 1935. Photoelastic investigations of shear tests of timber. Selected Eng. Papers No.174, Institution of Civil Engineers, London.

Cramer, S.M; Goodman, J.R; Bodig, J; Smith, F.w; 1984. Failure modelling of wood structural members. Struct. Res. No. 51. Civil Engineering Dept., Colorado State Univ. Fort Collins, Colo.

Dagher, H. J; Kimball, T. E; Shaler, S . M Abdel-Magid, B. 1997 Effect of FRP reinforcement on low grade eastern hemlock beams. [www.fpl.fs.fed.us/documents/PDF1996/Daghe96b.pdf](http://www.fpl.fs.fed.us/documents/PDF1996/Daghe96b.pdf)

Daniel, I.M; Yaniv, G Auser, J.W. 1987. Rate effects on delamination fracture toughness of graphite / epoxy composites. In composite structures - 4 (Proceedings of fourth International Conference on Composite structures, Paisley, Scotland, 1987) I.H.Marshall, ed. Elsevier Applied Science, New York, 1987.

Davalos, J.F; Madabhusi-Raman, P, Qiao, P, 1997. Characterization of mode III fracture of hybrid material interface bonds by contoured DCB specimens. Engineering fracture mechanics, 58 (3). pp.173-192.

Davalos, J.F; Madabhusi-Raman, P, Qiao, P, Lang, E.M; 1998a. Mode I fracture toughness of fiber reinforced composite wood bonded interface. Journal of Composite Materials (in press).

Davalos, J.F; Madabhusi-Raman, P, Qiao, P, Wolcott, M.P; 1998b. Compliance rate change of linear contoured DCB specimen for Mode I fracture of bonded interfaces. Theoretical and Applied Fracture Mechanics (in press).

Ehrenstein, G. W; Schmiemann, A; Bledzki, A; and Spaude, R. 1990 Corrosion Phenomena in Glass-Fiber-Reinforced Thermosetting Resins, in Handbook of Ceramics and Composites, Volume 1. Ed. Nicholas P. Cheremisinoff. Marcel Dekker.

Gardner, D.J; Davalos, J.F; Munipalle, U.M; 1994. Adhesive bonding of pultruded fiber-reinforced plastic to wood. Forest Products Journal 44 (5) pp.62-66.

Gardner, D.J; Davalos, J.F; Munipalle, U.M; 1994. Adhesive bonding of pultruded fiber-reinforced plastic to wood. Forest Products Journal 44 (5) pp.62-66.

Gillespie, R.H. 1965. Accelerated aging of adhesives in plywood-type joints. Forest Products Journal, 15(9) pp.369-79.

Hall, J.H; Haygreen, G.H. 1983. The Minnesota shear test. Forest Products Journal 33(9) pp. 29-32

Hunt, G.M. 1917. The preparation of specifications for propeller glue. Unpublished report, Project L-157-2. USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin.

Kreibich, R.E; Freeman, H.G. 1968. Development and design of an accelerated boil machine. Forest Products Journal 18(11) pp.24-26.

McDonald, A. G. Clare, A. B and Meder, A. R. 1999. Chemical characterization of the neutral water soluble components from radiata pine high temperature TMP fiber. In Proceedings of the 53rd General APPITA Conference. Rotorua, New Zealand. (19 -22 April. Vol. 2 pp. 641 - 647).

McKague, E. L. Jr.; Reynolds, J. D. Halkias, J. E. 1973 Thermo mechanical testing of plastics for environmental resistance. Journal of Testing and Evaluation. Vol 1. No.6 pp.468-471.

McLeod, A.M; Yolton, L.A; Sanborn, W.A; Phillips, R.S; 1956. A comparison of shearing strengths of glued joints at various grain directions as determined by four methods of test. Forest Products Laboratory Report No.1522. Forest Products Laboratory, Madison, Wisconsin.

Marra, A. A. 1992 Technology of wood bonding, principles in practice. Van Nostrand Reinhold, New York. ISBN 0-442-00797-3

Millet, M.A. Western, L.J. Booth, J.J; 1967. Accelerated aging of cellulosic materials: design and application of a heating chamber. TAPPI 50(11) pp.74a-80a.

Millet, A.M. Gerhards, G.C; 1972. Accelerated aging: Residual weight and flexural properties of wood heated in air at 115 - 175°C. Wood Science 4(4) pp.193-201.

Millet, A. M. Gillespie, R. H. 1978. Precision of the rate process method for predicting bondline durability. Prepared for the Department of Housing and Urban development by the Forest Products Laboratory, Madison, Wisconsin.

Millet, AM. Gillespie, RH. River, B.H; 1977. Evaluating wood adhesives and adhesive bonds. Report number 1057. USDA Forest Service, Forest Products Lab. Madison, Wisconsin.

Northcott, P.L; 1952. The development of the glueline-cleavage test. Journal of the Forest Products Research Society. December 1952 pp. 216 – 224.

Northcott, PL. Kreibich, RE. Currier, R.A; 1968. First replication comparing bond-degrade-accelerating systems. Forest Products Journal 18(5) pp.58-65.

Okkenon, E. A; River, B. H. 1989 Factors affecting the strength of block shear specimens. Forest Products Journal 39(1) pp. 43-50.

Pocius, A. V. 1997 Adhesion and adhesives technology. Hanser / Gardener Publications, Inc. Cincinnati. ISBN 1-56990-212-7

Radcliffe, B.M; Suddarth, S.K; 1955. The notched beam shear test for **wood**. Forest products Journal. April 1955 pp. 131– 134.

River, B.H; 1984. Accelerated, Real-time aging for four construction adhesives. Adhesives Age, February 1984 pp.16-21.

Smith, C.W; Kobayashi, A. S. 1987. Experimental fracture mechanics. In Handbook of experimental mechanics, edited by Kobayashi, A. S. Society for experimental mechanics, Prentice and Hall. ISBN 0 13 377706 5.

Soltis, L.A; Rammer, D.R; 1994. Shear strength of unchecked glued = laminated beams. Forest Products Journal, 44 (1) pp. 51-57.

Stamm, A.J. 1956. Thermal degradation of wood and cellulose. Industrial and Engineering Chemistry, 48(3) pp. 413-417.

Steiger, F.H. 1958. The Arrhenius equation in accelerated aging studies. American dyestuff reporter, 47 pp. 287-290.

Strickler, M.D; 1968. Specimen design for accelerated tests. Forest Products Journal 18 (9) pp. 84-90.

Truax, T.R. Harrison, C.A; 1922. Comparison of types of glue shear specimens. Unpublished report, Project L 157-3J24. USDA Forest Service. Forest Products Laboratory, Madison, Wisconsin.

Vick, C.B; 1995. Hydroxymethylated resorcinol coupling agent for enhanced adhesion of epoxy and other thermosetting adhesives to wood. In Christiansen, A.W; Conner, A.H; eds. Wood Adhesives 1995 Portland, Ore. Proc. 7296. Madison, Wis. FPS 47-55.

Vick, C.B; 1997 More durable epoxy bonds to wood with hydroxymethylated resorcinol coupling agent. Adhesives Age, July 1997 pp.24-29.

Western, J.L; 1964. Application of chemical kinetic principles to accelerated aging of cellulosic materials. M.S. Thesis, Department of Chemical Engineering, University of Wisconsin, Madison, Wisconsin.

Winandy, J. E Using kinetics-based models to address serviceability concerns for fire retardant treated wood at elevated in-service temperatures. In Proceeding f 5<sup>th</sup> World Conference on Timber Engineering, Volume 1. August 17 – 20, 1998. Montreux, Switzerland. Eds. Natterer, J. and Sandoz, J-L.

Yavorsky, J.M; Cunningham, 3H; 1955 Strain distribution in Maple glue block shear specimen as indicated by a brittle lacquer. Forest Products Journal. February 1955 pp. 80 - 84.

Yavorsky, J.M; Cunningham, 3H; Hundley, N.G; 1955. Survey of factors affecting strength tests of glue joints. Forest Products Journal October 1955 pp. 306 - 311.

Yeh, B; Schniewind, A. P; 1992. Elasto-plastic fracture mechanics of wood using the J-integral method. Wood and Fiber Science, 24(3) pp. 364 – 376.

## Appendix A Laminate mechanical test results

Table A.1 Maple / PRF system **block** shear results following **high** temperature water immersions

BLOCK SHEAR RESULTS						
Time of exposure (hours)						
95°C	0	6	12	42	90	148
Mean (psi)	1688	1526	1433	1330	1254	1005
St.dev.	58	95	224	175	160	115
COV (%)	3.4	6.2	15.7	13.1	12.8	11.4
85°C	0	24	48	144	340	484
Mean (psi)	1688	1547	1416	none	1257	965
St.dev.	58	227	98		139	166
COV (%)	3.4	14.6	6.9		11.1	17.1
75°C	0	144	600	936	1152	1752
Mean (psi)	1688	1325	1062	889	801	723
St.dev.	58	131	70	112	45	82
COV (%)	3.4	9.8	6.6	12.5	5.7	11.3
65°C	0	336	1176	1776	2328	2760
Mean (psi)	1688	1451	1177	1076	995	932
St.dev.	58	158	104	127	85	71
COV (%)	3.4	10.9	8.8	11.8	8.5	7.6
24°C	0	336	1176	1776	2328	2760
Mean (psi)	1688	1466	1445	1432	1444	1467
St.dev.	58	133	123	123	142	113
COV (%)	3.4	9.1	8.5	8.6	9.8	7.7

Table A.2 Maple / PRF system compression shear results following high temperature water immersions

COMPRESSION SHEAR						
Time of exposure (hours)						
95°C	0	6	12	42	90	148
Mean (psi)	2626	2149	1856	1642	1532	1107
St.dev.	281	165	128	272	91	42
COV (%)	10.7	7.7	6.9	16.6	5.9	3.8
85°C	0	24	48	144	340	484
Mean (psi)	2626	2147	1934	1734	1159	936
St.dev.	281	117	139	240	196	186
COV (%)	10.7	5.5	7.2	13.8	16.9	19.9
75°C	0	144	600	936	1152	1752
Mean (psi)	2626	2104	1597	1356	1248	1089
St.dev.	281	157	214	40	113	117
COV (%)	10.7	7.5	13.4	3.0	9.0	10.8
65°C	0	336	1176	1776	2328	2760
Mean (psi)	2626	1929	1940	1749	1532	1271
St.dev.	281	190	240	252	175	111
COV (%)	10.7	9.8	12.4	14.4	11.4	8.7
24°C	0	336	1176	1776	2328	2760
Mean (psi)	2626	2282	2384	2273	2215	2222
St.dev.	281	278	423	243	322	270
COV (%)	10.7	12.2	17.7	10.7	14.6	12.1

**Table A.3** Maple / PRF system glueline cleavage results following high temperature water immersions

<b>GLUELINE CLEAVAGE</b>						
<b>Time of exposure (houn)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean	576	496	464	424	375	310
(lb/inch)						
St.dev.	59	86	9	37	54	35
COV (%)	10.2	17.3	1.8	8.8	14.4	11.3
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean	576	509	472	413	349	279
(lb/inch)						
St.dev.	59	82	33	38	38	29
COV (%)	10.2	16.1	6.9	9.3	11.0	10.4
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean	576	none	430	354	none	None
(lb/inch)						
St.dev.	59		54	40		
COV (%)	10.2		12.5	11.2		
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean	576	492	377	374	336	339
(lb/inch)						
St.dev.	59	76	45	62	52	37
COV (%)	10.2	15.5	12.0	16.6	14.6	10.9
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
<b>Mean</b>	576	495	527	<b>none</b>	493	483
(lb/inch)						
St.dev.	59	91	86		62	62
COV (%)	10.2	18.4	16.3		12.5	12.9

**Table A.4 Maple / PRF / FRP system block shear results following high temperature water immersions**

<b>BLOCK SHEAR</b>						
<b>Time of exposure (hours)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean (psi)	1810	1713	1630	1353	1223	1161
St.dev.	134	160	316	287	292	144
COV (%)	7.4	9.4	19.4	21.2	23.9	12.4
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean (psi)	1810	1612	1548	1315	1227	1005
St.dev.	134	215	112	82	194	116
COV (%)	7.4	13.3	7.2	6.2	15.8	11.4
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean (psi)	1810	1421	1117	1055	980	737
St.dev.	134	364	224	176	133	72
COV (%)	7.4	25.6	20.1	16.7	13.6	9.7
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	1810	1482	1281	1095	1160	1001
St.dev.	134	403	259	255	247	174
COV (%)	7.4	27.2	20.2	23.3	21.3	17.4
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	1810	1690	1655	1503	1645	1632
St.dev.	134	179	187	92	315	221
COV (%)	7.4	10.6	11.3	6.1	19.2	13.5

**Table A.5 Maple / PRF / FRP system compression shear results following high temperature water immersions**

<b>COMPRESSION SHEAR</b>						
<b>Time of exposure (hours)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean (psi)	1914	1695	1564	1549	1181	1012
St.dev.	247	145	256	153	140	89
COV (%)	12.9	8.5	16.4	9.9	11.8	8.8
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean (psi)	1914	1714	1484	1315	1063	815
St.dev.	247	208	123	187	122	62
COV (%)	12.9	12.2	8.3	14.2	11.5	7.6
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean (psi)	1914	1586	1368	1298	1189	1007
St.dev.	247	269	223	89	108	88
COV (%)	12.9	17.0	16.3	6.9	9.1	8.7
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	1914	1648	1637	1562	1354	1153
St.dev.	247	173	269	155	238	115
COV (%)	12.9	10.5	16.4	9.9	17.6	10.0
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	1914	1800	1763	1706	1635	1768
St.dev.	247	271	128	343	272	222
COV (%)	12.9	15.1	7.3	20.1	16.6	12.6

**Table A.6** Maple / PRF / FRP system glueline cleavage results following high temperature water immersions

<b>GLUELINE CLEAVAGE</b>						
<b>Time of exposure (hours)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean (lb/inch)	342	301	241	218	188	182
St.dev.	6	21	36	26	17	31
COV (%)	1.7	6.8	14.8	11.9	9.3	16.8
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean (lb/inch)	342	294	239	210	182	157
St.dev.	6	29	36	24	39	26
COV (%)	1.7	9.8	15.3	11.4	21.6	16.5
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean (lb/inch)	342	295	281	261	234	217
St.dev.	6	89	68	18	46	68
COV (%)	1.7	30.1	24.4	7.4	19.8	31.6
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (lb/inch)	342	331	252	230	293	279
St.dev.	6	16	36	69	35	47
COV (%)	1.7	4.7	14.2	29.9	11.9	16.7
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (lb/inch)	342	286	286	283	267	320
St.dev.	6	6	40	66	70	46
COV (%)	1.7	11.3	14.1	23.3	26.2	14.3

Table A.7 Maple / Epoxy / HMR / FRP system block shear results following high temperature water immersions

<b>BLOCK SHEAR</b>						
<b>Time of exposure (hours)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean (psi)	2050	1474	1256	1100	none	965
St.dev.	145	344	326	114		129
COV (%)	7.1	23.3	25.9	10.3		13.3
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean (psi)	2050	1891	1786	1525	1105	870
St.dev.	145	134	83	51	165	104
COV (%)	7.1	7.1	4.6	3.4	14.9	11.9
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean (psi)	2050	1841	1559	1367	1206	1072
St.dev.	145	239	370	240	76	171
COV (%)	7.1	13.0	23.7	17.5	6.3	15.9
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	2050	1728	1532	1406	none	1320
St.dev.	145	341	200	276		158
COV (%)	7.1	19.8	13.1	19.6		11.9
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	2050	1858	1747	1624	1539	2115
St.dev.	145	300	334	281	219	379
COV (%)	7.1	16.2	19.1	17.3	14.2	17.9

Table A.8 Maple / Epoxy / HMR / FRP system compression shear results following high temperature water immersions

<b>COMPRESSION SHEAR</b>						
<b>Time of exposure (hours)</b>						
<b>95°C</b>	<b>0</b>	<b>6</b>	<b>12</b>	<b>42</b>	<b>90</b>	<b>148</b>
Mean (psi)	2438	752	644	484	455	347
St.dev.	118	118	150	181	168	109
COV (%)	15.7	15.7	23.2	37.3	36.9	31.5
<b>85°C</b>	<b>0</b>	<b>24</b>	<b>48</b>	<b>144</b>	<b>340</b>	<b>484</b>
Mean (psi)	2438	2140	1791	1494	1116	829
St.dev.	118	462	308	93	230	122
COV (%)	15.7	21.6	17.2	6.2	20.6	14.8
<b>75°C</b>	<b>0</b>	<b>144</b>	<b>600</b>	<b>936</b>	<b>1152</b>	<b>1752</b>
Mean (psi)	2438	1880	1752	1427	1359	1063
St.dev.	118	333	244	99	104	110
COV (%)	15.7	17.7	14.3	6.9	7.7	10.3
<b>65°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	2438	2134	2004	1782	1576	1347
St.dev.	118	271	167	153	151	176
COV (%)	15.7	12.7	8.3	8.6	9.6	13.0
<b>24°C</b>	<b>0</b>	<b>336</b>	<b>1176</b>	<b>1776</b>	<b>2328</b>	<b>2760</b>
Mean (psi)	2438	None	None	None	None	None
St.dev.	118					
COV (%)	15.7					

Immersion time (hrs.)	24°C immersion			85°C immersion		
	Continuous	1 soak / dry cycle	Multiple soak / dry cycles <sup>1</sup>	Continuous	1 soak / dry cycle <sup>1</sup>	Multiple soak / dry cycles <sup>2</sup>
0	100	100	100	100	100	100
24	95	92	92	90	80	80
72	92	90	84	76	68	59
144	93	88	78	69	63	56

<sup>1</sup>

<sup>2</sup> Removed at time interval and given a dry soak cycle prior to re-immersion or testing (dependant on defined treatment)

## Appendix B. Work Procedures

Work procedure; Wood preparation.

Material requirements	Equipment requirements
Lumber type required for study	Workshop planer Cross-cut saw Workshop table saw

Forward planning;

- Reserve equipment
- If lumber is required for sample preparation immediately after machining it should be pre-conditioned to 65% R.H. and 70°F.

Procedure;

- End use and required dimensions of lumber defined
- Use knife planer to achieve desired thickness and smooth surface
- Cut to length and width using the cross-cut and table saws respectively

Information to be recorded;

- Source of material
- Species
- Date of knife planing

## Work procedure Pultruded FRP preparation

Materials required	Equipment required
Pultruded FRP roll Acetone	Respirator Gloves, safety glasses and ear plugs Cross-cut saw Diamond coated blade Cloth
Workshop proficiency required. <i>See</i> Lab. Co-ordinator	
Respirator fitting required. <i>See</i> Lab. Co-ordinator	

### Forward planning

- Check on cross-cut saw availability
- Arrange for assistance when handling large FRP rolls

### Procedure

- Define end use and required dimensions of FRP sheets
- Cut to length
- Wipe surfaces clean with acetone soaked cloth
- Stack and label prior to use

### Recorded information

- Material source
- Roll number and prior surface treatment (typically sanded or un-sanded)

## Work procedure Mixing GP 4242/4554 PRF resin

Materials required	Equipment required
GP resin 4242 GP hardner 4554 Water Adherends	<b>Scales</b> Mixing containers Respirator Gloves and safety glasses Stirrer
Respirator fitting required. See Lab. Co-ordinator	

### Forward planning

- Prepare work area by covering table and immediate floor area with rosin paper
- Wood should be pre-conditioned to 8-12% moisture content as specified by the resin supplier

### Procedure

- Calculate requirement based on spread rate of 0.25g/inch, make allowances for waste. The mix ratio is;
  - 18% water**
  - 12% hardner**
  - 70% resin**
- Place mixing container on scales and weigh out required water
- Add hardner by weight and mix for several minutes until a lump free slurry is achieved
- Add resin by weight and mix for several minutes
- The mixed resin has a working life of approximately 35 minutes

### Recorded information

- Any prolonged pause in mixing procedure

### Relevant documentation

- Manufacturers mixing and application guidelines

## Work procedure Priming pultruded FRP with PRF adhesive

Materials required	Equipment required
Mixed PRF adhesive FRP material Acetone	FRP drying rack Scales Metal scrape Gloves and safety glasses

### Forward planning

- Cover work area and immediate floor area with rosin paper

### Procedure

- Label and weigh each FRP sheet
- Apply a large drop of PRF to the FRP surface and drag across with the metal scrape to produce a uniform coating on each side
- Place coated FRP in rack
- Allow 48 hours curing time in ambient conditions
- Reweigh each sheet

### Recorded information

- Weight of resin applied to each FRP sheet

### Relevant documentation

- Brown, M. 1998. Surface Characterization of pultruded FRP and its relation to bond performance with eastern hemlock. Master of Science thesis, University of Maine.

## Work procedure FPL1A Epoxy resin mixing

Materials required	Equipment required
DGEBA epoxy resin (DOW) Benzyl alcohol Hydrophobicfumed silica Triethylenetetramine hardner ( <b>DOW</b> ) Adherends	Scales Mixing containers Gloves and safety glasses Stirrer 10 ml pipette

### Forward planning

- Cover work area and immediate floor area with rosin paper
- The exo-thermic nature of this epoxy's curing reaction causes working time to be less than 5 minutes. All materials must be close at hand

### Procedure

- Calculate requirement based on a spread rate of 0.31 g/inch<sup>2</sup> and make allowances for wastage
- The mix ratio is (in parts by weight);
 

DGEBA epoxy resin	<b>100 g</b>
Benzyl alcohol	12.5 g
Hydrophobicfumed silica	25 g
Triethylenetetramine hardner	<b>11.1g</b>
- Weigh *out* required amounts of each prior to mixing
- Add hydrophobic fumed silica to resin and mix thoroughly until the powder is incorporated into the resin
- And **benzyl** alcohol to mix and stir vigorously
- Add hardner and stir vigorously
- Apply immediately
- The low viscosity of this resin means only a low level of pressure is required when laminating. Hand clamps have been found suitable and are able to facilitate resin transfer whilst minimizing squeeze out

### Recorded information

- Any deviation from mixing schedule

### Relevant documentation

- Vick, C.B; 1995. Hydroxymethylated resorcinol coupling agent for enhanced adhesion of epoxy and other thermosetting adhesives to wood. In Christiansen, A.W; Conner, A.H; eds. Wood Adhesives 1995 Portland, Ore. Proc. 7296. Madison, Wis. FPS 47-55.

- **Vick, C.B; 1997 More durable epoxy bonds to wood with hydroxymethylated resorcinol coupling agent. Adhesives Age, July 1997 pp.24-29.**



- **Vick, C.B; 1997 More durable epoxy bonds to wood with hydroxymethylated resorcinol coupling agent. Adhesives Age, July 1997 pp.24-29.**

## Work procedure Laminating procedure

Materials required	Equipment required
Lumber FRP (typically pre-primed) Mixed adhesive	Scales Mixing containers Spatula Gloves and safety glasses Pneumatic clamps

### Forward planning

- Prepare adherends
- Prepare adhesive
- Reserve hydraulic clamps

### Procedure

- **Wipe** lumber and FRP surfaces to remove dust
- Calculate and mix required amount of adhesive
- Place a secondary container onto scales and pour in the required amount of adhesive for one surface. apply this to the wood surface using a spatula and roller
- Lay-up FRP sheet on this
- Apply adhesive to second wood member using the same method and lay this on to the FRP surface to create a wood / FRP / wood laminate
- Repeat this procedure to make up the required sample size whilst keeping the working life of the adhesive in mind
- Transfer the laminate batch to the hydraulic clamps and apply pressure. Side bracings may be required to avoid laminate slippage as pressure is applied

### Recorded information

- Time between coating each surface with adhesive and placing a lamina on top of this (open time)
- Time between laying-up and pressing of each laminate (closed time)
- Time laminates are place in press so removal from press can be scheduled in accordance with the curing time required for the adhesive

## Work procedure Sample cutting

Materials required	Equipment required
Laminates	Respirator Gloves, safety glasses and ear pugs Delta table saw Walk-in vacuum hood Shear block cutting jig (for D905 specimens)
Workshop proficiency required. See Lab. Co-ordinator	
Respirator fitting required. See Lab. Co-ordinator	

### Forward planning

- Check equipment availability

### Procedure

- Draw up a cutting pattern for each laminate based on required specimen dimensions
- Cut according to this taking care to number specimens in a way which describes the laminate they came from and their position within this
- Wood / FRP laminates are a difficult material to cut and cause extensive blade wear; Jigs should be made where possible and close cutting to the blade avoided

### Recorded information

- Samples labeled to describe which laminate they came from and their position within this

### Relevant documentation

- Relevant ASTM standard or in-house test procedure describing specimen geometry

## Work procedure **Block** shear testing

Materials required	Equipment required
Machined laminate samples	Instron in room 1 Boardman Tinus Olsen shear tool Calipers

### Forward planning

- Reserve equipment

### Procedure

- Turn on Instron
- Place shear tool below cross head and fit load head to cell
- Adjust position safety control
- Set load rate to 0.2 inch / minute
- Allow machine to sit for 30 minutes from initial turning on to warm up
- Jog head to within 1 inch of top of shear tool when a specimen is in place and press **GL** reset
- Reset load balance
- Measure shear area of specimen, record and insert specimen
- Jog load head down to point of initial contact with shear tool
- Press down; load will be applied. Once peak load has been reached press stop, then return
- Remove specimen and press peak to obtain peak load and record this along side sample shear area
- Press reset peak then track
- Repeat steps from initial measuring of sample

### Recorded information

- Matched shear areas and peak loads to allow calculation of shear strength in psi

### Relevant documentation

- ASTM D-905

## Work procedure; Compression shear testing

Material requirements	Equipment requirements
Machined laminate samples	Instron in room 1 Boardman Compression shear tool (Prof. S. Shaler) Calipers

### Forward planning;

- Reserve equipment

### Procedure;

- Turn on Instron
- Place shear tool below cross head and fit load head to cell
- Adjust position safety control
- Set load rate to 0.2 inch / minute
- Allow machine to sit for 30 minutes from initial turning on to warm up
- Jog head to within 1 inch of top of shear tool when a specimen is in place and press **GL** reset
- Reset load balance
- Measure shear area of specimen, record and insert specimen with grain parallel to plane of shear direction
- Jog load head down to point of initial contact with shear tool
- Press down; load will **be** applied. Once peak load has been reached press stop, then return
- Remove specimen and press peak to obtain peak load and record this along side sample shear area
- Press reset peak then track
- Repeat steps from initial measuring of sample

### Information to be recorded;

- Matched shear areas and peak loads to allow calculation of shear strength in psi

### Relevant documentation

- ASTM D1037
- Hall, J.H; Haygreen, G.H. 1983. The Minnesota shear test. Forest Products Journal 33(9) pp. 29-32

## Work procedure; Glueline cleavage testing

Material requirements	Equipment requirements
Specimen laminates (2 X 2" with a machined notch at FRP / wood interface – 7mm deep made with thin kerf table saw blade)	22 kip AEWI Instron  Cleavage tool and base Calipers

### Forward planning;

- Reserve equipment

### Procedure;

- Turn on hydraulic power unit
- Turn on Instron and PC
- Restore position calibration
- Divert hydraulic power to Instron (by pressing buttons **1** and **11**)
- **Start** ramp generator and select relative ramp and rate of load (0.1 inch / min)
- Set position and load safety limits
- Mount cleavage tool and base in grips
- Measure length of sample's machined notch
- Position sample on base and jog tool down to point of initial contact
- Apply load and record peak load

### Information to be recorded;

- Matched notch lengths and peak load for each specimen to allow calculation of adjusted load (lb per inch)

### Relevant documentation

- Instron manual

## Work procedure; Vacuum pressure soaking of samples (VPS)

Material requirements	Equipment requirements
Samples to be immersed Water	Pressure vessel Forced air outlet (AEWC lab) Vacuum pump Weight to submerge samples in pressure vessel
Proficiency in use of vacuum pump, forced air system and pressure vessel	

### Forward planning;

- Check on equipment availability

### Procedure;

- Empty pressure vessel of water
- Place samples in tanks, weigh down and add water so that it covers samples by at least 6 inches
- Place lid on pressure vessel and seal
- Draw vacuum to 25 in. Hg, seal valve and maintain for 30 minutes
- Release vacuum and apply pressure of 75 psi and maintain constant pressure for 1 hour
- Remove soaked samples which can be stored intermittently in a bath of tap water if required

### Information to be recorded;

- Any deviation from procedure

## Work procedure; High temperature immersion of samples

Material requirements	Equipment requirements
Samples to be immersed Water WVR algaecide	Stainless steel tank (plastic may be used at temperatures below 50°C) WVR circulating water bath and associated tubing Insulation for tanks Mesh and stainless wire to contain samples

### Forward planning;

- Check with Lab. Co-ordinator with regards to space requirement

### Procedure;

- Become familiar with operation of circulating water baths
- Fill tanks with water
- Add required amount of algaecide (dictated by tank volume)
- Place inlet and outlet tubing of circulating water bath in tank and insulate
- Turn on circulating water bath; **select** required temperature, set pump **speed** to maximum and allow time for the circulation to establish this temperature in the baths
- Once temperature is reached, lower pump **speed** to medium flow rate
- Place VPS'd specimen batches into mesh bags (according to times of immersion), open tanks and immerse
- Remove batches at appropriate times and cool in tap water prior to testing or further conditioning

### Information to be recorded;

- Immersion periods for sample batches should be defined prior to initial immersion. Any deviation from this should be recorded.

## Work Procedure Handling of data for rate analysis

Sigmaplot was used as the analysis tool for the data in this study and was selected on the basis of its comprehensive equation library and graphing capability.

Simply put a rate analysis requires a large number of samples to be made up and immersed in high temperature water baths (or exposed in dry environments). Results typically show the strength of matched samples to deteriorate as a function of both time and temperature. The strength is expressed as a percentage of a zero immersion time control set and is termed residual strength. Initially each temperature is handled in isolation and the residual strength of each replicate is plotted versus time of exposure (in this case immersion). The following graph is a representative data set.

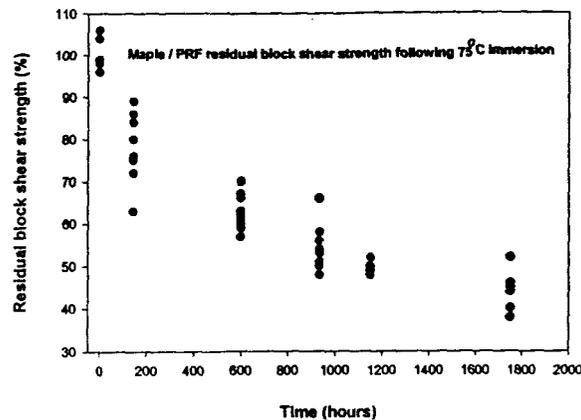


Figure B.1 Raw data for a single immersion series.

A two parameter exponential decay regression (from the regression wizard) is then used to fit the rate of strength loss using the Marquardt-Levenberg algorithm. This finds coefficients of the independent variables that give the 'best fit' between the equation and the experimental data. An incremental data set is produced by Sigmaplot and can be plotted and taken to be the rate of strength loss at that temperature.

This procedure is repeated for the data at each temperature for each matched sample set and a family of curves is then compiled. **The** time taken to achieve a defined level of residual strength **loss** is taken from the generated incremental data at each temperature. An exponential growth regression of these times (in days) versus the inverse of absolute temperature ( $\times 10^4$ ) is then made using **the** Marquardt-Levenberg algorithm. The resultant equation **is** then used **to** calculate time to the defined level **of** residual strength loss for samples immersed **at** 20°C

## **Biography of the Author**

Jonathan Philip Alexander was born in Liverpool, England on 29<sup>th</sup> October 1975. He was raised by his parents, Nora and Malcolm near Liverpool, England and Graduated from St. Anselm's College in 1994. He went on to attend the University of Wales, Bangor and graduated in 1998 with a degree in Forestry and Forest Products. He came to Maine in August 1998 to pursue a Master of Science in Forestry and entered the Advanced Engineered Wood Composites Research Program.

After receiving his degree Jonathan will be joining JELD-WEN Research and Development in Oregon to begin a career in the wood products industry. Jonathan is a candidate for the Master of Science degree in Forestry from the University of Maine in December, 2000.