Characterizing the Durability of PF and pMDI Adhesive Wood Composites Through Fracture Testing

by

Christopher R. Scoville

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Approved by:

_____
Dr. Joseph Loferski, Chair

_____
Dr. Charles E. Frazier

_____
Dr. Frederick A. Kamke

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Department of Wood Science and Forest Products

Blacksburg, Virginia

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ABSTRACT

The increased use of wood composites in building materials results in a need for a better understanding of wood adhesion. The effects of water and temperature exposure on the durability of wood products were assessed using the double-cantilever beam (DCB) method of fracture testing. The relative durability of phenol-formaldehyde (PF) and isocyanate (pMDI) adhesives was compared using a 2-hour boil test and an environmental test. The feasibility of using oriented strandboard (OSB), oriented strand lumber (OSL) and parallel strand lumber (PSL) for the DCB fracture method was assessed. The fracture resistance of PF was reduced significantly by the aging exposures. The fracture resistance of pMDI did not decrease after the 2-hour boil test. The DCB fracture method was shown to be useful with a square-grooved machined specimen using OSB and OSL.

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CHAPTER 1 - INTRODUCTION

Wood composites are commonly used in single-family home construction in the United States. Wood composite products consist of wood fiber used in conjunction with some other material, such as various adhesives, cement, plastic, or fiberglass. Some wood-based composites, like plywood and particleboard, are well known. Other common wood composites include Oriented strandboard (OSB), hardboard, medium density fiberboard (MDF), glulam beams, and laminated veneer lumber (LVL).

Defining the problem

The natural origin of wood brings about many of its highly variable characteristics. Solid wood alone is a complicated material to work with, compared to metals, plastics, or other materials used in construction. The mechanical properties of wood vary greatly and depend on such factors as growth rate and health of the tree it comes from, species, the direction that stresses act on the wood, and machining parameters. Shrinking and swelling stresses in addition to other internal stresses may cause lumber to warp, twist, and deform. Knots in wood cause localized weak points in wood. Wood is very weak in some directions; it is very strong others (Haygreen and Bowyer 1989). An understanding of wood and how it reacts to different environments will help us understand the behavior of wood composite materials.

By cutting wood material into smaller sizes and reconstructing the wood fibers in a specific orientation, we can design a composite wood product to have material properties that perform suitably for a desired function. Maximizing the efficiency of the use of wood material is the goal of wood composite materials research. Success in reaching this goal provides economic and environmental benefits.

Although there are certain advantages associated with composite wood products, there are also a few extremely important questions that are difficult to answer. The wood fibers must be reconstructed with some type of adhesive that holds them together satisfactorily. Due to the nature of interactions between wood and adhesive, wood based composites can perform poorly

when in contact with moisture. Some problems with the durability of wood composites have been studied in an attempt to improve the performance of wood composites for long-term applications. For example, hardboard siding can swell and lose integrity quickly, even when there is a protective overhang of the roof above. Some homeowners are unsatisfied with siding performance on their homes. Costly civil suits have affected construction companies and siding manufacturers. Glulam beams may have delamination problems when in humid conditions. Many types of plywood delaminate when they are exposed to long periods of cyclic moisture. OSB can lose the ability to maintain adhesion after a few days of wet exposure. Even while there is no liquid water contact, wood composites absorb moisture from air during humidity changes and irreversible thickness swell can occur.

Many questions may be asked to assess our understanding of the mechanisms of failure of wood products. At what point in the life cycle of a product do we consider the material as having finished its usefulness? How much degradation is accepted before reaching failure? What happens on a microscopic scale when wood composites fail? Is poor performance caused by wood failure or adhesive or cohesive failure? How do adhesives bond to wood?

Since the rise in popularity of composite wood products, laboratory tests have been developed in an attempt to correlate a short-term experiment with the long-term service life of the product. Many ASTM standards have been developed to measure the weatherability or longevity of materials (ASTM 2000). Boil-soak-dry or vacuum-pressure-soak-dry cyclic tests are used to expose wood composite materials to high temperature and moisture conditions to accelerate the long-term effects of weathering. After exposure, mechanical property tests are conducted to determine mechanical property loss and other performance indicators. Scientists endeavor to correlate these laboratory tests with varying durations of outdoor exposure. It is important to analyze what is being tested when we apply accelerated or extreme environmental conditions to wood composite specimens. One must consider to what degree does a boil-soak-dry test correlate to the 'real-life' service environment of the product in use.

Even considering the potential difficulties, composite wood products are very important and extremely useful in today's market. Wood composites will be utilized well into the foreseeable future. Long-term performance and failure mechanisms need to be further studied.

Definitions

Durability is defined as the ability of a material to withstand environmental stress over an extended period of time (Green 1999). The durability of a building, assembly, component, product, element, or construction can be thought of as the capability of that object to provide and sustain acceptable performance over a specified time under designed operating conditions. The service life of a product is the period of time after installation during which "all properties exceed minimum acceptable values and the system or assembly performs the functions for which it was designed, manufactured or assembled; based on assumed levels of use and maintenance, under anticipated internal and external environments" (Green). Durability is qualitative and service life is quantitative.

The temperature and relative humidity of the area immediately surrounding an object, and the exposure of the object to moisture, wind or UV light are considered the environment around that object. Failure can be defined as a decline in integrity or effectiveness, or not fulfilling expected performance.

Objectives

In order to better understand durability as it relates to wood composites, general and specific objectives were set. The global objective is to understand the effects of exposure to water and temperature on the durability of wood composite products. Specific objectives were developed to determine the effects of water and temperature on the fracture energy of wood composite products, accelerated aging tests, a comparison of two different adhesives, and the application of the fracture test method to composite products.

Specific Objectives:

1. Determine the effect of time, water exposure, and temperature changes of two types of accelerated aging tests on the degradation of adhesive bonds in wood composites.
2. To compare the fracture performance of phenol-formaldehyde and isocyanate adhesives during environmental exposure of bonded wood laminates.
3. To determine the feasibility of using the fracture test method to measure the fracture energy of various wood composite products.

Scope and Limitations

The topic of durability of wood and wood composites includes many diverse areas. Durability is affected by moisture or ultraviolet (UV) light exposure, temperature, stress level, insect attack, decay due to fungi, and in some cases, mechanical wear. In the topic of wood composites and adhesion, however, there may be many factors affecting durability that are not related to biological or mechanical deterioration. Those issues that are related to biological deterioration are beyond the scope of this project. Areas of focus for this project include moisture content cycling, temperature changes, dimensional stability, internal stress, and how these affect the wood-adhesive interphase.

CHAPTER 2 - UNDERSTANDING THE PROBLEM

Characteristics

A knowledge of the characteristics of wood composites will facilitate a study of the longevity of such products. Composite materials are a combination of two or more different materials. Any two pieces of solid wood can vary greatly in density and strength properties even if they both came from the same tree. Since lumber is cut from a log, it is very difficult to find a "perfect" piece of wood. There are many types of defects in solid wood, such as knots or drying checks. High grade lumber can be found at a high price; however, as timber harvesting changes from the larger trees of the past to the smaller diameter and faster-growing plantation trees of today, defect-free lumber will continue to become more rare.

Wood is an orthotropic material with three perpendicular property directions: tangential, radial, and longitudinal. In tension, the weakest direction for wood is perpendicular to the grain. A piece of wood that has a large knot in it will be weaker in bending or tension at the location of the knot. The procedure of processing wood into smaller components, and recomposing it into a product that may perform better than solid wood is an important way to engineer the properties of the product.

The common element in any wood composite product is the wood fiber. For example, medium density fiberboard (MDF) is made of fibers and adhesive and wax. Particleboard is made of small particles of wood material that are pressed with adhesives and waxes into a panel. OSB is a panel product made of thin wood flakes, typically 120 mm long, 12 mm wide, and around 0.6 mm thick (FPL 1999). Parallel strand lumber (PSL) is composed of strands and is marketed as a substitute for solid lumber. Plywood is made of veneers of wood that are usually around 2-3 mm thick and 2.5 meters long. These veneers are layered, alternating the grain directions, then pressed together with adhesives to make a panel. Wood composites are designed with balanced construction to maintain symmetry and avoid warping under changing moisture and temperature conditions. Balanced construction describes a symmetry of materials and properties about the neutral axis of a laminate or layered composite material. For example, a 5-ply sheet of plywood

has 2 plies above and 2 plies below the center ply (Figure 2). The center, top, and bottom plies are oriented in the longitudinal direction, and the second and fourth plies are oriented 90 degrees from the longitudinal, or in the transverse direction. This cross-ply orientation helps stabilize the shrinkage and swelling of the plywood and it reinforces the weak direction of wood with the strong direction.

Oriented strandboard (OSB) is similar to plywood in application, but it is made of thin, flat 'strands' of wood. Some types of OSB use flakes that are laid in a mat, in different layers that also have cross-ply orientation. The perpendicular orientation helps maintain dimensional stability and increases some properties of the panel. By using small flakes of wood, we reduce the size a tree must be before it is harvested. OSB panels also use less wood material to serve a certain function than can be achieved with solid wood. When the manufacturing process of wood composite materials is understood, we can observe some of the difficulties in maintaining mechanical properties after exposure to changing environmental conditions over time. When OSB is being made, flakes are prepared and mixed with resin, then placed into a mat. Flakes are oriented across the span of the panel to increase bending strength. Despite a general orientation of the flakes, the lack of precision and the inherent random character of flake placement causes variability in the alignment of flakes in the mat. Flakes are piled layer upon layer until the mat is approximately four to six inches thick. The mat is then placed in a hot press where pressure is applied at around 500 psi for a few minutes. Pressures, times, and adhesive content varies with type of adhesive and desired properties of the panel.

This pressure compresses the mat to a thickness that is smaller than the final thickness of the product. When the pressure is released, the mat expands to the final thickness of around ½ or ¾ inch. Many flakes may have been compressed more at points of contact with other flakes than at a void in the material. This causes localized bending of fibers in the flakes. The resin between the flakes, which is applied with as little as 2% to 5% by solids weight, holds the mat in this compressed state.

Plywood and OSB are commonly used in construction as 122 cm x 244 cm (4 ft x 8 ft) sheets for wall, floor, or roof sheathing. Sheathing panels of this size can save time and use less material

than lumber-sheathed construction. After the panel has been shipped and installed, if water comes in contact with the panel, capillary action pulls it into the many void spaces and into bound water and the flakes absorb moisture. Flakes expand and tend to straighten out. The tendency of wood composites to expand is one cause of problems with the long-term sustainability of mechanical properties. Characteristics of wood composite panels vary widely between plywood, OSB, and particleboard. The problems associated with different types of composites also vary, but there is an underlying mechanism that must be understood in order to quantify the longevity of a wood-based composite product.

Looking closer at the manufacture of wood composites may also help us to understand some of their advantages. Let us consider the differences between a piece of medium-grade solid wood lumber and a piece of laminated strand lumber (LSL) of the same size. We can compare a log cut into solid lumber with a log used to make LSL. Solid lumber usually has growth defects such as large knots; these knots come from the growth of branches in the stem of the tree. Knots limit the bending or tensile strength of wood by changing the direction of the wood cells with respect to the axis of the board. The effect of knots on the tension strength of solid wood is similar to the effect of drilling holes the size of the knots in the board.

LSL is made from flakes that are cut from logs, which also usually have knots in them. The knots are cut up and are distributed over many flakes. These flakes are subsequently randomly placed in the fabrication process. The knots or growth defects in LSL are thus spread throughout the volume of the material. The growth defect in LSL will not affect its strength and stiffness as much as a defect in solid lumber, because there is no concentrated strength-reducing location. Bending strength or modulus of rupture (MOR) of LSL is generally higher than an equivalent size piece of solid lumber. The basic homogeneous attributes of wood composites provide integrity and reliability. The manufacturing process also affects density and other properties.

In dry environments, wood composites can perform better than solid lumber in a structure for short-term loads. At higher moisture exposure, composite properties tend to degrade faster than

solid wood. High moisture exposure could be caused by high relative humidity or contact with liquid water.

Requirements

Since wood composites are engineered materials, they are designed to have specific performance advantages over solid wood products. In order to describe failure, we must ask some basic questions. What are the minimum performance criteria that must be achieved? What are the expectations of the engineer, construction manager and user? The influence of how long a product lasts affects decisions of economy, design, and use. The fear of unsatisfactory performance after a few years accompanies a new product that has not yet been tried for extended time periods. The design of a building may determine how long it lasts. Likewise, the durability of materials used in the construction of a building also affects how long it lasts.

A composite wood product must maintain minimum mechanical properties through its expected life. There are certain requirements for dimensional stability in thickness, length or width for many composite panel and lumber products. Design loads are determined for engineered wood products; these must be met and exceeded in order to have a product that performs acceptably. Failure of an adhesive bond can be classified as adherend failure, adhesive failure, or cohesive failure. Adhesive failure occurs at the surface between the adhesive and substrate. Cohesive failure occurs inside the adhesive, and adherend failure occurs in the substrate. Failure to withstand the design load can be affected by the configuration of the loads.

Adhesion

The construction industry has been using more adhesives than other wood product industries for decades (Blomquist *et al.* 1984). Adhesives are substances that can hold materials together at the surfaces. Adhesion is an attractive phenomenon between two material surfaces. Strength and stiffness of wood composites are attributed to the efficient transfer of stress from wood fiber to adhesive, and back to the wood. Cohesion is the 'force' that holds a material together.

Adhesion is the tendency of two separate entities to be held together. Adherends are the materials being held together with an adhesive (FPL 1999).

There are various mechanisms that create adhesive forces between two surfaces. It is difficult to determine to what extent a specific mechanism influences adhesion in a given material. Attractive forces are likely a combination of two or more mechanisms working together. The relative importance and occurrence of adhesive phenomena can be understood from a study of the five theories of adhesion: mechanical interlock, diffusion, electrostatic, adsorption /specific adhesion, and covalent chemical bonding theories.

The mechanical interlock theory accounts for flow of adhesive into microscopic and macroscopic surface pores and projections. After the adhesive enters these contours, the curing process results in a physical interlock between adhesive and adherend. Penetration of the adhesive into wood material two to six cells deep is considered effective interlocking (FPL 1999). If adhesive flows into cell cavities or diffuses into cell walls, there is molecular-level contact with the wood polymers.

The diffusion theory refers to interdiffusion of macromolecules of both adhesive and wood at the bonding surface, and is accepted as a contributor to adhesion of fibers in MDF or hardboard (Voyutskii 1963). In most cases, the diffusion theory does not apply to wood adhesion. A well-defined boundary surface between two materials is the interface between those two materials. An interface is slightly different from an interphase, which is a boundary region between two materials that is a gradient of the two materials (see Figure 2.1). There is no exact surface in an interphase. Any interface can be considered as an interphase region, if surfaces are examined under sufficient magnification.

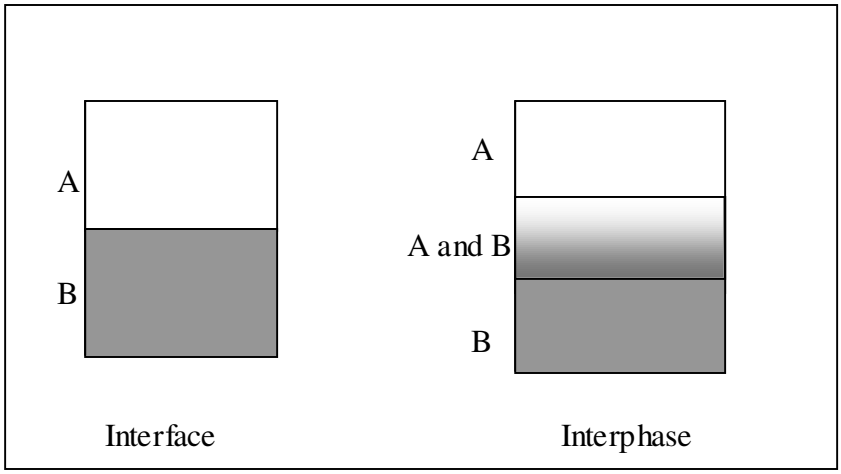

Figure 2.1 – Difference between interface and interphase.

Adhesion can occur even if there is no mechanical interlocking or diffusion. Positive charges in one material can be attracted to negative charges in another material. The electrostatic theory proposes that an electrostatic double layer is formed on the surfaces of adhesive and adherend. Transfer of electrons across the interphase of solid materials requires very intimate contact and very smooth surfaces. Smooth surfaces in wood are limited to the dimensions of the cells, and intimate contact only occurs if the adhesive can wet the surface well. Electrostatic attractions are thought to be less important than other theories for wood adhesion.

As an adhesive comes in intimate contact with wood material, physical attractions develop between adjacent molecules or atoms. These secondary interactions such as Van derWaals Forces, hydrogen bonding, and dipole-dipole interactions are weak but abundant. The adsorption/specific adhesion theory describes adhesion that results from intermolecular and interatomic forces between materials. The contribution of this mechanism to adhesion can be significant due to the large magnitude of occurrence of these forces along the bond surface. The adsorption/ specific adhesion theory is important for wood adhesion.

The covalent chemical bonding theory refers to formation of covalent bonding during adhesion. Covalent bonds are primary bonds that are created when two atoms share electrons, and are much stronger than hydrogen bonds or any other secondary bonds. If covalent bonds occur in an adhesive-wood interaction, long-lasting adhesive properties would result. Little evidence of

the existence of covalent bonding between adhesives and wood had been reported before 1999 (FPL 1999). The inter-penetrating network (IPN) theory describes what happens with certain isocyanate adhesives. The small, reactive monomers can penetrate deeply into the wood surface, then as they cross-link, they form a network that spans wood substance.

In order for the adhesive and wood to come close enough for intimate contact, adhesives must be liquid and must wet the surface. A surface-wetting liquid is one that spreads easily over and into pores of the surface and readily flows into capillaries. The flow of adhesive into contours of the wood surface increases surface area and opportunity for interactions between adhesive and substrate. Wood is highly polar; there are local differences in charge on the surfaces of cellulose and hemicellulose. Water is also highly polar. Charges on water molecules are attracted to charges on cellulose molecules when the distance between the two is small. Likewise, adhesives that are polar can develop strong initial attractions with wood in order to set the foundation for strong bonds. The adhesive development that follows is the mechanism that maintains these strong initial interactions over the service life of the bond.

Studies of surface wetting with wood adhesion began before 1960. The contact angle is measured as the angle of contact between a drop of water or other liquid and the wood surface. Zisman (1963) showed that a general linear relationship exists between the cosine of the contact angle and surface tension, Y, for a homologous series of organic liquids (Figure 2.2). The critical surface tension of wetting, Y_o, is the surface tension for a liquid that has a contact angle of zero, where optimum wetting takes place.

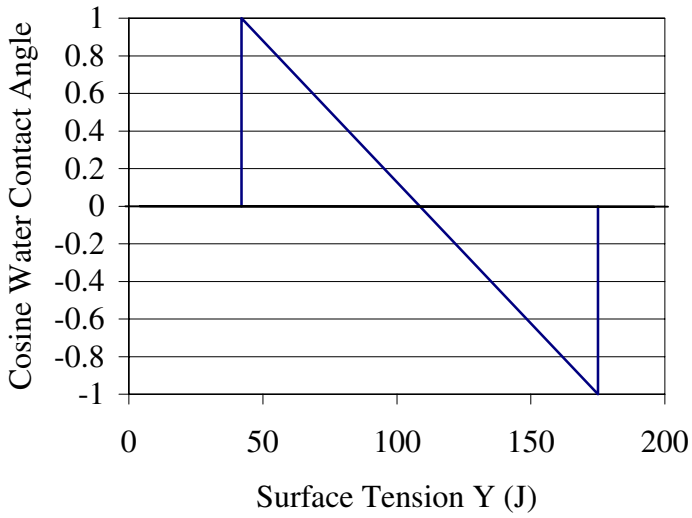

Figure 2.2 – Zisman's Rectilinear Relationship

As a recently cut wood surface is exposed to organic molecules in air, a rapid decrease in surface energy occurs (Herczeg 1965). The surface collects contaminants, which form a layer that is less polar than wood. The layer of contaminants is known as the weak boundary layer, which hinders wetting of liquid adhesives. Herczeg showed that the cosine of water contact angle decreases due to air exposure after machining the surface. Figure 2.3 relates an example of the trend in contact angle over time. Longer exposure to air results in higher contact angles. Surfaces that are freshly machined with sandpaper or a planer adhere better than surfaces that are exposed to air, heat or light.

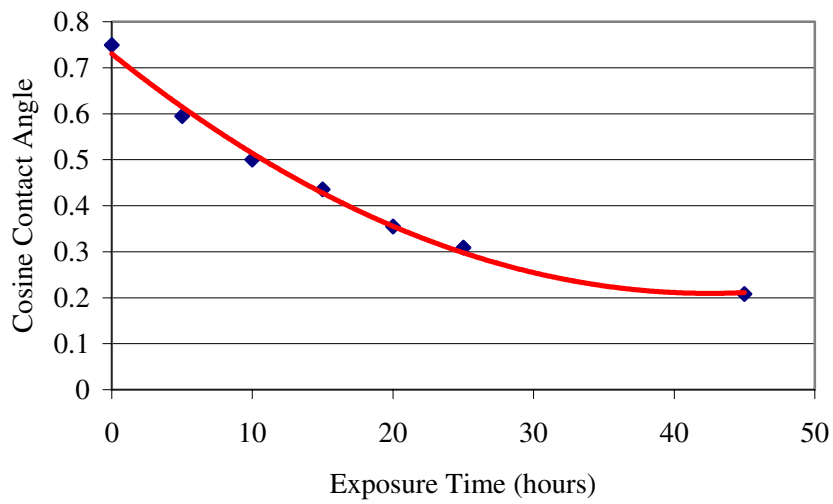

Figure 2.3 – Cosine contact angle over time exposed.

In order to understand adhesive failure, it is important to know where the failure is initiated. The failure can start as an adhesive failure at the interphase between adhesive and wood, or a cohesive failure in the bulk of the adhesive. Bikerman as well as Sharpe and Schonhorn have stated that separation at the interface rarely occurs, since there are higher attractive forces at the interface than there are within the bulk of the adhesive (Kinloch 1983). Another hypothesis is that interfacial failure is improbable, especially when chemical wetting has taken place (Kinloch/Good). As the bondline is exposed to water, moisture first displaces the adhesive from the adherend by rupturing some of the secondary bonds at the adhesive/adherend interface (FPL 1999).

Temperature

Adhesives are polymers that have a high sensitivity to temperature changes. An adhesive that is designed for use at 20° C may be strong but brittle at –50° C, and weak with a high ductility at 100° C (Adams *et al*. 1992). During fabrication in the hot press, heat makes the adhesive flow and wet the wood surfaces, fill gaps, and conform to the immediate shape adjacent to the adhesive. Heat also begins to cure the adhesive. In the process of curing, the adhesive shrinks,

and the adherend expands, which causes internal stresses in the adhesive. It is very important to use adhesives designed for the end-use temperature range that will be seen by the product.

Wood members under load are also affected by heat exposure. In a long-term heat exposure test, load capacity was reduced for higher heat exposure (Fridley, *et al.* 1989). Wood members that on average failed at 2000 seconds under a loading rate of 0.067 kN/second at room temperature would fail after 1000 seconds at 150° C and 500 seconds at 250° C (Lau, Barrett 1998).

Structural adhesives are those that contribute to the integrity and stiffness of a structure for as long as it is in use. The function of adhesives in keeping a building intact impacts the life safety of inhabitants. Plywood and exterior-grade OSB are wood composites made with structural adhesives such as phenol-formaldehyde or isocyanates. These adhesives are capable of maintaining adequate levels of performance after long-term exposure to water soaking and drying. Epoxide resins can withstand degradation of properties after short-term (a few days) soaking. Urea-formaldehyde and casein adhesives do not maintain properties after short-term humidity exposure. Polyvinyl acetate, polyurethane, and hot-melts are considered non-structural interior-use adhesives, because they cannot maintain minimum material properties after exposure to moisture (FPL 1999). Modifications to adhesives can improve resistance to degradation over time (River 1994, Mototani *et al.* 1996).

Understanding adhesion continues to challenge scientists in wood composites and other materials. As technology progresses, a better understanding of adhesive mechanisms and failures is desirable.

Internal Forces

Internal stresses are present in adhesives as well as solid or composite wood materials. Wood experiences considerable shrinking and swelling stresses during drying. Variable stresses are present at most temperatures, moisture contents, and relative humidity levels because as these levels change, a gradient is created in the wood material that also changes. Shrinking and

swelling of the wood, coupled with a dissimilar change in dimensions of the adhesive, cause high localized stresses and the physical opening of fracture surfaces at the interface, bulk adhesive, or wood material (Na, Ronze, Zoulalian 1996).

Moisture Effects

There is no single factor that has a more detrimental effect on wood composite products than water. Without moisture exposure, adhesively bonded joints would last almost indefinitely. Creep effects and temperature affect the longevity of bonds as well, but not as much as moisture does. Adhesion of metals, plastics, fibrous composites, or concrete is also affected by water (Kinloch 1983, Cardon 1991).

Since wood composite products are used in all parts of the US, many temperature and moisture situations exist. Dry, arid zones like Phoenix, Arizona will not experience the same types of problems as a humid area like Miami, Florida or Seattle, Washington. Moisture sources affecting building performance can be internal or external. Rainwater, ground water, or water vapor are external sources; respiration, cooking, plumbing leaks, and showers are internal sources. A comprehensive discussion of these sources has been presented by Angell and Olson (1988). Indoor relative humidity levels are usually below 60%. Properly installed vapor barriers reduce water vapor flow through walls, and reduce condensation and moisture buildup within a wall. Air barriers reduce bulk flow of water through convection in air. Building felt can protect wood from wind-driven rain when properly applied. Flashing is crucial to protect wood members and keep them dry (Quarles, Tully 2000).

Moisture effects can be analyzed at three levels: macroscopic, microscopic, and molecular or atomic. Some dimensional changes due to moisture absorption are easily seen without microscopes, such as drying cracks in oak lumber. On a microscopic scale, the anatomy of the components of wood cell walls can cause differences in swelling when water and adhesives come in contact with the wood cells. On a scale measured in angstroms, molecules of wood components and adhesives interact to create adhesive forces. Wood products exposed to changes in relative humidity of air will gain or lose water. As wood fibers come in contact with

water, the cell walls absorb water and the fibers tend to increase in diameter. The diffusion of water transfers moisture through the wood material from areas of higher to lower moisture content.

It is important to investigate the relation between dimensional stability and durability. Dimensional stability may not be related to the durability of a wood composite, although they are both affected by similar parameters such as water sorption and time of exposure to moisture. Composites made with juvenile wood have proven to have higher strength retention properties than wood composites from mature wood after exposure to moisture, but show more dimensional instability than those composites made of mature wood furnish (Pugel *et al.* 1990). Dense composite panels generally have more thickness swell than low-density panels. The extent and rate of change of water absorption in wood composites is well documented (Siaiu 1991). Several studies have shown the water concentration profiles of such composite products as MDF, OSB, and particleboard (Xu, *et al.* 1996). The flexural strength and stiffness of OSB were found to decrease substantially with increase in moisture exposure (up to 83% loss in MOE from 4% to 24% moisture content).

Weathering

During exposure to outdoor environments, chemical reactions and mechanical changes occur in the wood material. These are part of the natural weathering process of wood. Weathering is a different mechanism than decay due to microorganisms. With exposure to weather, smooth wood surfaces are converted to roughened surfaces with visible changes such as raised grain. Solar radiation, moisture, temperature, and oxygen exposure are responsible for weathering. Light can penetrate below the wood surface, causing a gray layer at the surface and a brown layer below it. The low molecular weight components are removed by wind and rain, resulting in higher polysaccharide content. Several chemical changes can take place during weathering. Free radicals are formed and lead to oxidation of lignin. This decreases lignin content, which causes an increase in polysaccharide content. The methoxyl content also decreases and the introduction of carboxyl groups causes an increase in the acidity of the wood (Feist, Hon 1984).

The degree to which plywood or OSB degrades over time is dependent on the quality of the exposed wood and the adhesive used. First, microchecks are formed due to exposure. Microchecks then increase in size and fiber bundles in the cells begin to separate due to loss of the middle lamella. Small particles of degraded components are removed through leaching, volatilization and mechanical action. Earlywood erodes faster than higher density latewood areas, which causes raised grain, a visual defect. Waferboard, flakeboard, chipboard and OSB typically degrade quicker than solid wood or plywood, due to the larger surface area of the wood particles (Feist, Hon 1984). Sunlight exposure can delignify a wood surface after only 4 hours. After 3 days, substantial delignification takes place, and 6 days of sunlight exposure causes severe surface degradation (Evans *et al.* 1995).

On the microscopic scale, wood cells change when water and adhesives come in contact with them. Attempts have been made to increase the dimensional stability of wood composites with varying degrees of success. The polymerization of some monomers such as methyl-methacrylate or styrene to solid wood using free radicals can improve dimensional stability(Meyer 1977). These improvements do not last very long because there is no reaction with the cell wall. Wood cell walls may react with some polymer matrix components in the presence of additives and provide cross-linking. One of these additives that have been studied is dimethylol dihidroxyethylene urea (DMDHEU). Polyethylene glycol (PEG) is another additive used to minimize dimensional changes. The main advantage of these compounds is that they are water soluble (Simonsen 1998). DMDHEU has been proven to reduce shrinkage of wood by 50% (Militz 1993). The cross-linked polyglycols had been thought to enhance the bulking effect and reduce dimensional changes (Simonsen 1998).

Acetylated wood treatments have been successfully shown to decrease dimensional changes associated with water sorption. Thickness swelling and water absorption were reduced significantly for acetylated wood fiber mixed with polypropylene fiber (Lee, Shin 1997). Acetylation has negative effects on other properties of composite products. Bending strength and IB strength generally decrease with acetyl treatment, which indicates that acetylation can be detrimental to adhesion.

On a sub-microscopic level, secondary bonds are being created and broken constantly. The magnitude of an individual bond is small, but millions of bonding sites will add up to a substantial force. Before being exposed to water, the locus or starting point of failure of the bond is within the adhesive layer. After substantial exposure to water, failure may begin in the interphase. River, *et al.* (1989) found that degradation during aging could be caused by depolymerization, chain scission, reaction of side groups, or crosslinking.

Test Standards

In an effort to determine how well wood-based composite materials stand up to long-term use in outdoor environments, scientists have attempted to accelerate the life cycle of the material. Short-term laboratory tests that attempt to simulate long-term exposure are called accelerated aging tests. The American Society of Testing and Materials (ASTM) has standards to evaluate wood properties, adhesive properties, and wood bonding performance.

ASTM D-3762 (98) Standard Test Method for Adhesive-Bonded Surface Durability of Aluminum (Wedge Test) is an attempt to reproduce the forces and reactions of the metal-adhesive interphase. It is used extensively in predicting the durability of surface preparations to the adherends. This test gives more reliable results than the other common lap shear or peel tests (ASTM 1999). ASTM D-3433 (93) is a double-cantilever beam fracture energy test for bonded metal joints. ASTM D-1151 (90) tests the effects of moisture and temperature on non-wood adhesive bonds.

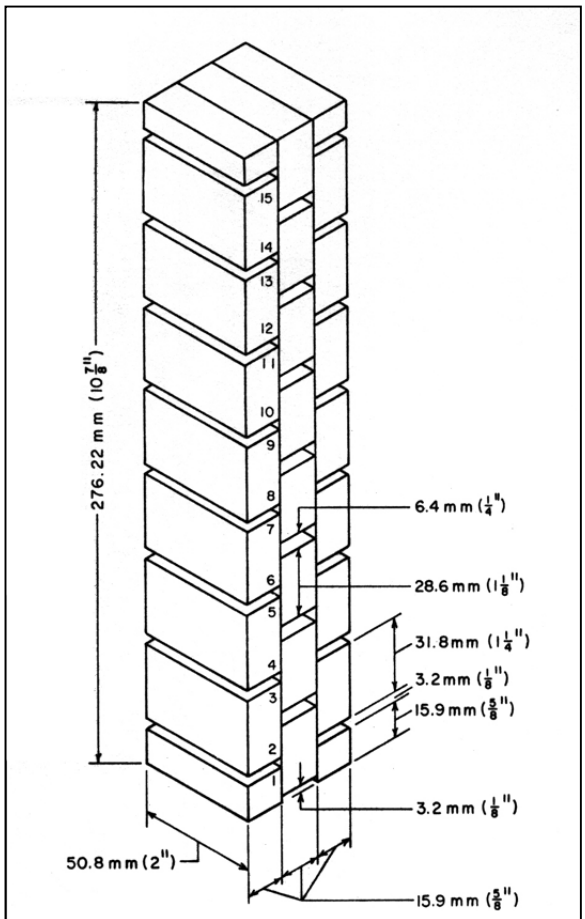

Figure 2.4 – View of Test Block Assembly

The purpose of test method ASTM D-4502 (92)

Standard Test Method for Heat and Moisture Resistance is to test the ability of adhesive-bonded wood joints to resist thermal and hydrolytic degrade. Chemical effects can be estimated for fire-retardant, preservative, or extractive components in adhesives. Small test specimens are bonded and placed in heat resistant aging jars and then subjected to constant states of temperature and moisture. The jars also contain a salt-saturated solution, which subjects the bonded wood specimens to 100% relative humidity. Physical properties are tested after exposure and compared to unexposed samples.

The Standard Test Method for Resistance to Deformation Under Static Loading for Structural Wood Laminating Adhesives Used Under Exterior (Wet-Use) Exposure Conditions (ASTM D-3535) compares the performance of Glulam beams under hot and humid conditions. A test block assembly is placed under spring-loaded constant load for seven days under two exposure conditions: 71° C at ambient relative humidity, or 26.7° C at 90% relative humidity (see Figure 2.4).

The Standard Test Method for Multiple-Cycle Accelerated Aging Test (Automatic Boil Test) for Exterior Wet Use Wood Adhesives (ASTM D-3434) is for testing the durability of adhesives for outdoor exposure wood products. It has proven useful for comparing different adhesives subjected to the same temperature and moisture cycles. An automated boil machine and 90 specimens of each type of adhesive being considered are required. Tensile shear loads are used to compare properties before and after exposure. See Figure 2.5 for the profile of temperature and moisture levels for 2 cycles of testing. Specimens are exposed to between 20 and 1600 cycles.

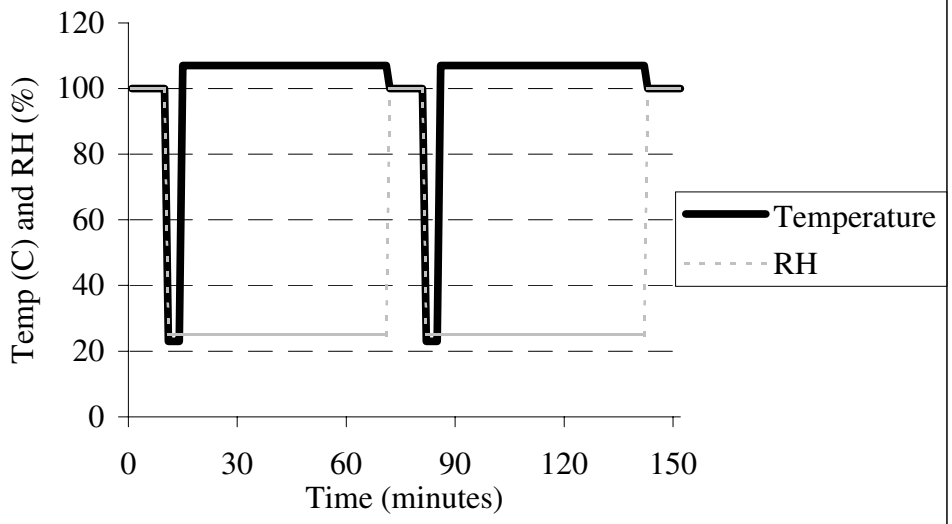

Figure 2.5 – ASTM D 3434 "Automatic Boil Test"

The Standard Specification for Adhesives for Structural Laminated Wood Products for Use Under Exterior (Wet Use) Exposure Conditions (ASTM D-2559) is also known as the "6-ply De-lam" test. The purpose of this test is to show the resistance to delamination of a six-level stair laminated wood sample during cyclic wetting and drying. Cycles of moisture between 12% and 100% and temperatures of 22°, 65°, and 100° C are administered along with a vacuum-pressure-vacuum-pressure change from –85 to 517 kPa, and then back to zero applied pressure. The percentage of delamination at the bondline is measured and compared.

ASTM D-1037 may be the most widely used and well-known ASTM test in use. It is specifically for evaluating the properties of fiber and particle-based panels, including OSB. Panels are tested in static bending, tension, compression, and hardness. Nail tests for lateral resistance, withdrawal, nail-head pull-through, and screw withdrawal as well as in-plane shear and block shear are specified. The two most commonly used tests from ASTM D-1037 may be the compression block shear test and the internal bond (IB) test. The properties of the composite products are to be compared before and after an accelerated aging exposure. The test comprises six cycles of boiling to freezing temperatures and 10% to 100% humidity. One cycle of the accelerated aging test is shown in Figure 2.6.

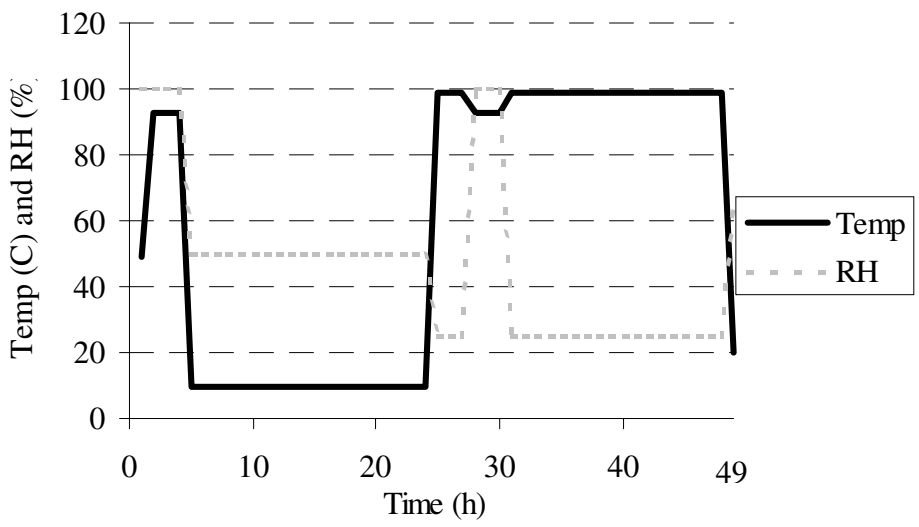

Figure 2.6 – ASTM D 1037 Accelerated Aging Cycle

An investigation at the Forest Products Laboratory in Madison, WI studied two alternate accelerated aging tests for wood-based panels (McNatt, McDonald 1993). The ASTM D-1037 accelerated aging test normally takes from 12 days to two weeks to administer. A test that would take less time to administer but give similar results was desired. Two variations of a four day-long test were used to simulate the ASTM six cycle test. Each cycle lasts 24 hours (see Figure 2.7). The variations of ASTM D-1037 resulted in a decrease of mechanical properties of wood composite panels that was similar to the decrease from ASTM D-1037 cycles.

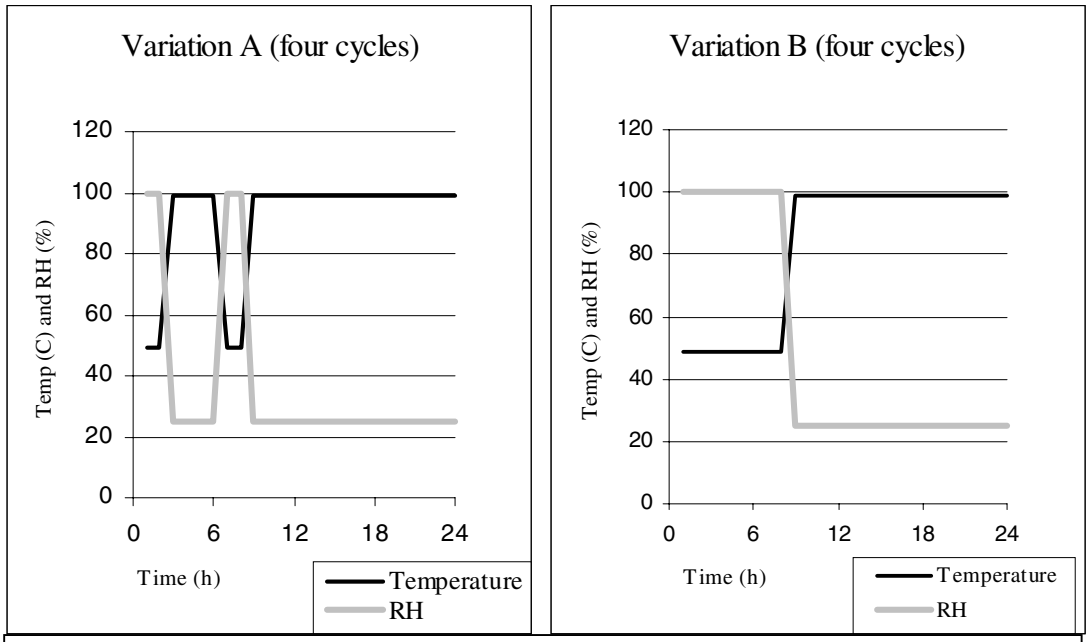

Figure 2.7 – 24 hour variations of ASTM D 1037

A newer test standard uses oxygen pressure to accelerate the aging of wood and wood-metal composite joints (ASTM D-3632).

Difficulties in determining how accurately accelerated aging tests predict the actual service performance of wood composites in use may create questions of the usefulness of these tests.
Long-term outdoor exposure tests are underway in Madison, WI, but these tests will not provide results for another twenty years, and the results will be characteristic of that specific climate. The degree of correlation between outdoor aging and accelerated laboratory aging were studied using five cycles of boil-dry treatment. This test was found to have equal property reductions as one year of outdoor exposure in Madison. Reductions in flexural MOR and modulus of elasticity of plywood panels was similar to those after 1 year of outdoor exposure (Okkonen, River 1996). Repetitions ranging from 1 to 40 cycles of both boil-dry and vacuum-pressure-soak-dry treatments were also compared to ASTM D- 1037 accelerated aging treatment.

In an effort to describe the durability of structural adhesives, eight UF glues and a PF resin were compared with Casein and RF glues (Raknes, 1996). Long-term accelerated aging treatments cycled between 20° C, 90% RH and 50° C, 50% RH once a month for 3 years. These tests were

compared to a monthly cycle between 20° C, 90% RH and 30° C, 30% RH for 5 years' duration. RF and PRF resins withstood aging satisfactorily, whereas UF resin decreased in integrity after the aging exposure. Small lap-shear specimens were tested according to the tensile strength of a 1- inch overlap bond.

Tests on urea formaldehyde (UF) adhesives related strength and accelerated aging. River *et al.* (1994) failure mechanisms with accelerated aging and unmodified, modified, or PF adhesive. Modification improved durability of UF bonded joints. DCB fracture toughness tests were implemented. Accelerated aging included 10 cycles of Vacuum-pressure-soak-dry (VPSD) treatment, and then tested for mechanical properties.
Specimens were held at 70° C and 80% relative humidity for 40 days before being subjected to a shear test. VPSD treatment weakened the wood-adhesive interphase.

Modeling

In order to model something as complex as a wood composite failure over time with different environmental exposure regimes, specialized mathematical and statistical procedures are necessary. One point of view is that it is impossible to model or to predict the aging of wood products that are exposed to weather. The concept is that humidity, temperature, and weather conditions are too random to be analyzed statistically; they never repeat any cycles or patterns. Although there is no way to predict the temperature, relative humidity indoors or out, accelerated aging tests demonstrate relative resistance to mechanical property degradation in a controlled, systematic artificial weathering state. If an adhesive lasts twice as long as another adhesive in a cyclic accelerated aging test, it can be concluded that it will also be more resistant to degradation than the other in real outdoor exposure or under normal humidity changes indoors.

Dunn *et al*. (1990) have stated that the properties of a composite should be calculable from the knowledge of properties of such components as the wood, adhesive, and assembly configuration. Establishing a credible database is an important objective in durability studies. An infrastructure of standard test methods should be created and accepted. Long-term data are

as important as short-term data because they offer real evidence of longevity of properties. Durability assessment hinges on the ability to predict: the course of events at times greater than those of the experiments, the difference of behavior of one composite structure from that of another, the synergisms between influential variables, and the departures from the established patterns of behavior.

No accurate and accepted procedure to model and predict composite performance has been presented as of the date of this literature review.

CHAPTER 3 - FRACTURE TEST METHOD

Introduction

In many wood composites, the bondline can be stressed beyond its capacity to resist crack formation. These cracks greatly affect the mechanical properties and durability of the composite material. There are many factors during the manufacturing of a wood-adhesive interface that can affect the integrity of the bondline. In order to characterize the robustness of this interaction between wood and resin, scientists started looking at the fracture energy, or the energy it takes to pull apart the two wood faces of the bondline. Fracture toughness is a material property and describes the ability of a material to resist crack propagation.

Theory

There are three modes of fracture (Figure 3.1). Mode I opening does not create a net moment because the forces are co-linear. A net moment can only be created by two forces that have some distance of separation, as in the case of Mode II or Mode III. Mode II is longitudinal shear, and mode III is transverse shear. Mixed mode fracture consists of two or more of these modes. Mode I fracture generally occurs at the lowest energy, and is considered the critical fracture mode. Modes II and III fracture have also been investigated (Yoshihara 2000; Ehart 1999).

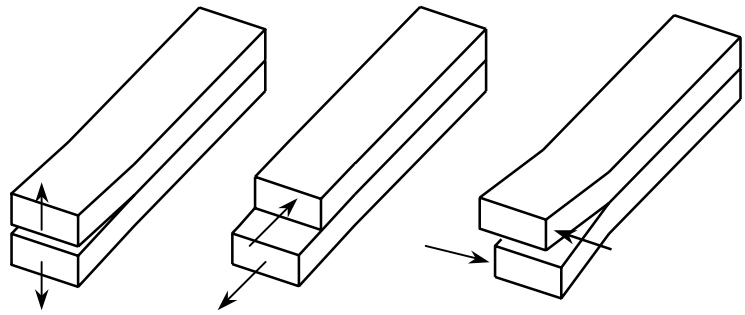

Figure 3.1 – Fracture Modes

In mode I fracture, the stress intensity factor (K) is related to fracture energy (G_{Ic}) by the Poisson's ratio (ν) and the modulus of elasticity (E):

$$G_{Ic} = \frac{K^2}{E}\left(1-\nu^2\right) \qquad \textbf{[3.1]}$$

The stress intensity factor characterizes the stresses and displacements near the crack tip and is related to the fracture energy, as shown above (Anderson, 1995). The total energy of the system (G) is assumed to remain constant and can be described by:

$$dG = dD + d\gamma = 0 \qquad \textbf{[3.2]}$$

where D is the potential energy of deformation, and γ is the surface energy of the new surfaces. The general stain energy release rate (G_{Ic}) for the compliance method of fracture analysis is:

$$G_{Ic} = \frac{P_c^{\,2}}{2b}\frac{dC}{da} \qquad \textbf{[3.3]}$$

where P_c is the load at which crack propagation occurs, C is the compliance of the beams, a is the crack length, and b is the width of the specimen. As the crack length increases along the bondline in a DCB specimen, the stiffness of the beams decreases and the compliance C of the beams increases. A linear relationship exists between the cubic root of compliance, $\sqrt[3]{C}$, and the crack length a:

$$\sqrt[3]{C} = ma + b \qquad \textbf{[3.4]}$$

where m is the slope of the line, and b is the y-intercept (Williams 1989). The correction factor χ is the distance from the origin to the x-intercept of the line, and can be found using the slope and the y-intercept:

$$\chi = b/m \qquad \textbf{[3.5]}$$

Figure 3.2 illustrates the correction factor and the correlation between cube root of compliance and the crack length for the DCB fracture specimen.

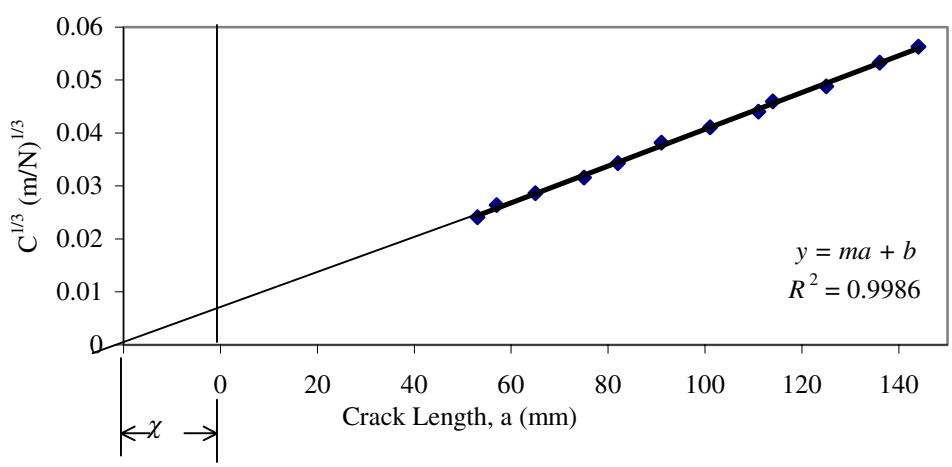

Figure 3.2 – Correction Factor and $C^{1/3}$ vs. a

Solving for *b* in equation [3.5] and substituting the result into [3.4] gives:

$$\sqrt[3]{C} = ma + m\chi \qquad \textbf{[3.6]}$$

Equation [3.6] can be rewritten as follows:

$$C = m^3 (a + \chi)^3 \qquad \textbf{[3.7]}$$

Differentiating Equation [3.7] with respect to C gives:

$$\frac{1}{3} C^{-\frac{2}{3}} = m \frac{da}{dC} \qquad \textbf{[3.8]}$$

Solving Equation [3.8] for $\frac{dC}{da}$:

$$\frac{dC}{da} = 3mC^{\frac{2}{3}} \qquad \textbf{[3.9]}$$

Taking the value of [3.7] to replace *C* in Equation [3.9] leads to:

$$\frac{dC}{da} = 3m\left[m^3 (a + \chi)^3\right]^{\frac{2}{3}} \qquad \textbf{[3.10]}$$

Which can be briefly expressed as:

$$\frac{dC}{da} = 3m^3 (a + \chi)^2 \qquad \textbf{[3.11]}$$

Returning to Equation [3.3] and substituting $\frac{dC}{da}$ for Equation [3.11] gives:

$$G_{Ic} = \frac{P_c^2}{2b} 3m^3 (a+\chi)^2 \qquad \textbf{[3.12]}$$

Recalling $EI_{eff} = \frac{2}{3}\frac{1}{m^3}$, the end result will be:

$$G_{Ic} = \frac{P_c^2}{b}\frac{(a+\chi)^2}{EI_{eff}} \qquad \textbf{[3.13]}$$

The DCB fracture test method used in this study follows the form of Equation [3.13].

Contoured dual-cantilever beams (CDCB) were used in the form of a pure epoxy as well as aluminum beams with an epoxy adhesion line. Fracture surfaces were examined to determine fracture ductility and resistance at several temperatures. A method of finding the optimum bond thickness was established, and the effects of bond thickness and temperature on fracture energy were also described (Hunston, *et al*., 1980).

Ebewele, River, and Koutsky (1979) showed that the adhesive thickness and grain orientation affect the fracture energy. They also found that increases in cure time increased the fracture toughness considerably. The test specimen configuration they used evolved over time and is currently quite simple to manufacture. First, a very precisely machined curvature was used on the two bonded wooden beams in order to have constant stiffness as the crack length increased.

Since then, others such as River and Okkonen (1993) used an aluminum tapered backing reinforcement to keep the stiffness of the beams constant for all crack lengths. This required additional adherents between the wood and the aluminum, which may have caused additional experimental variation. Blackman, *et al.* (1991) developed four linear elastic fracture mechanics methods for analyzing the data from load-displacement measurements from testing machines. This allowed them to use double rectangular wooden members as the beams, which are bonded together and subjected to cantilever pulling forces. The dual cantilever beam (DCB) method

uses a setup similar to Figure 3.3. This methodology has been widely accepted and is now generally used for testing of fracture strengths (Scott, *et al.* 1992; Lim, *et al.* 1994).

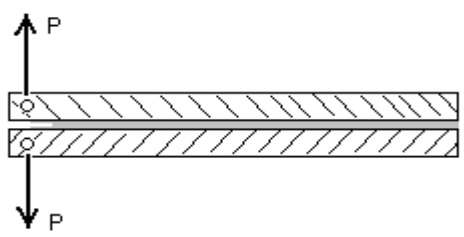

Figure 3.3 – DCB test specimen.

The DCB method for analyzing fracture mechanics data depend on the specimen behaving in a linear elastic manner. The test gives a load – displacement history as well as crack lengths at arrest. The goal in fracture testing is to keep the fracture within the adhesive bondline. If the wood fails, we do not learn anything about the bondline except that the adhesive is stronger than the wood. But if the adhesive fails, then we can analyze and compare the effects of different environmental exposures, different adhesives, and production parameters on fracture energy.

Studies at Virginia Tech have investigated the fracture energy of DCB specimens before and after a two-hour boil test (Gagliano and Frazier, 2001). Fracture testing is an energy-based test and makes possible direct comparisons of the relative quality of bonding. Differences in performance of types of resins, different press times, or different treatments before or after pressing may be subtle and difficult to see from strength-based tests. The fracture test method has shown significant differences in adhesives that were not shown using the IB test (Gagliano, Frazier 2001).

Fracture testing is valuable for durability studies of composite materials. The fracture characteristics of Parallam PSL have been compared to solid wood and particleboard. PSL had

a higher fracture toughness in both weak directions as well as the principle stress direction, using a wedge fracture test (Ehart, *et al.* 1998). Fracture energy was shown to be significantly reduced when a specimen was loaded for long time periods.

Although common tests such as the compression shear block test and IB test can give an average stress over the area of the bondline, we can learn more about adhesion using an energy based test. For a compression shear block test, 100% wood failure occurs when the adhesive bond is stronger than the wood in tension perpendicular to the grain. Since the adhesive is stronger than the wood in shear parallel to grain, the fracture occurs completely in the bulk wood material and we can not learn much about the adhesion in the bondline. The DCB fracture test method focuses the failure in the bondline, and the analysis is more indicative of surface variations and interactions between the resin and the wood material.

CHAPTER 4 – USING THE FRACTURE TEST METHOD TO COMPARE THE DURABILITY OF PF AND PMDI ADHESIVES

Introduction

Resin characteristics affect the behavior of adhesive bondlines during cure and during the useful life of the product. The resistance of the adhesive to moisture and temperature changes has a great affect on the durability of the adhesive bond. This chapter addresses the effect of adhesive properties on the fracture energy of bonded wood laminates.

The objective of this study is to compare the durability of phenol formaldehyde (PF) and polymeric methylene diphenyl diisocyanate (pMDI) adhesives. PF and pMDI are two common structural adhesives used for various composite wood products. Since structural adhesives are used in products that are rated for use in outdoor exposure, it is important to learn as much as possible about how these adhesives perform in wet or humid conditions.

The effect of 2-hour boil cycles and 4-day accelerated aging cycles on the fracture energy of wood laminates bonded with PF and pMDI was investigated, and the performance of PF and pMDI was compared.

In order to compare PF and pMDI adhesives, it is important to understand the differences between them and the similarities they have. The following is a brief background about the basic characteristics, manufacturing process, and properties of PF and pMDI.

PF Resin

Phenol formaldehyde resins are thermosets. Thermosets are resins that cure in the presence of heat by an irreversible cross-linking process. The most common PF starts out as a liquid with small polymers of phenol and formaldehyde suspended in an aqueous solution. PF is also used in the form of a powder or an impregnated paper. As temperature increases in the hot-press, the resin softens and flows into the wood surface contours. Provided the wood surface is sufficiently active, the flow of the adhesive allows intimate contact between wood and adhesive. As the temperature continues to increase, the molecular weight of the PF polymers increases due

to cross-linking that occurs, and the resin stops flowing, hardens, and turns into a brittle glassy solid. This solidification is the mechanical development of the adhesive.

There are two types of PF resins; novolaks are catalyzed in acidic conditions, and resols are catalyzed by alkaline conditions. Resol PF resins are normally used for wood composite products. PF resins are normally used for exterior grade structural products such as plywood, OSB, and PSL. The speed of cure can be controlled by the formaldehyde to phenol (F**:**P) ratio, by the pH of the adhesive mixture, and by temperature.

Most PF resins used in the industry are water based solutions, but too much water from other sources, either in the wood or introduced during hotpressing, may cause washout of the resin. Washout occurs when water affects the concentration and viscosity of the resin enough to weaken the adhesion to the wood fibers. The basic components of the resin, such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) are hydrophilic and absorb water and may cause detrimental thickness swell. PF adhesives are not harmed by temperatures up to 250 C, so the products can be hot-stacked, or placed in a pile without having to be cooled down. PF resins are less expensive than isocyanates, but generally cure slower.

pMDI Adhesive

Like PF, isocyanate resin is also a cross-linking thermoset. The isocyanate starts out as a monomeric, low viscosity liquid. It is also nonpolar. The liquid readily wets the wood surface, and the small molecular weight facilitates deep penetration of the adhesive into the wood material. PMDI resins penetrate further than PF resins. The resin cures by reacting with the water in the wood and creating urea linkages, which creates a rigid, polar network. This adhesive network has been shown to create urethane linkages with molecules in the wood. This is an important contributor to the properties of adhesion of isocyanates.

Isocyanate resin is used primarily in LSL and OSB. It is highly stable in the presence of water; conversely, the resin does not cure in the absence of water. This hydrolytic stability allows the use of higher MC wood, without risking washout. Some isocyanate wood composite products

with larger cross-sections use steam injection hot-pressing to transfer heat uniformly throughout the cross-section.

Since the synthesis of isocyanate resin involves the use of phosgene, a toxic gas, it is more expensive than PF. Isocyanates are not as thermally stable as PF, but they are faster curing. Despite the added difficulty and expense of handling the toxic isocyanates, these adhesives have the advantage of not producing formaldehyde emissions.

Experimental Design

Specimens made with each adhesive (PF and pMDI) were exposed to 2-hour boil cycles ranging from 0 to 4 cycles, and other specimens were exposed to 4-day environmental cycles ranging from 0 to 2 cycles (see Tables 4.1 and 4.2). Details of these aging conditions are given later.

Table 4.1 – Number of specimens tested for boil cycles.

Adhesive	control	1 cycle	2 cycles	3 cycles	4 cycles
PF	5	5	5	5	5
PMDI	5	5	5	5	5

Table 4.2 – Number of specimens tested for environmental cycles.

Adhesive	control	1 cycle	2 cycles
PF	4	4	4
pMDI	4	4	4

Specimen Preparation

The nature of the fracture test requires that specific specimen preparation be followed. The 25 specimens of each adhesive were made within a two day period, according to the following paragraphs.

Materials

Yellow-poplar (*Liriodendron spp.*) flat-sawn sapwood lumber (55 mm thick, 170 – 200 mm wide)

Phenol-formaldehyde (PF) resin (GP® 3121 Resi-Strain OSB)

Polymeric di-isocyanate (pMDI) resin (Huntsman Polyurethanes® RUB 9257)

Wood Machining

Clear 300 mm sections were cut from the lumber. Edges were machined on a jointer. Blocks were equilibrated to 12% MC in Conditioning Room (20° C (+/-1 C) and 65% RH +/- 1%).

Lines were drawn on the edge of the board at a 5 degree angle to the grain. A 35 mm thick slab was cut parallel to the 5 degree lines. The slab was cut into two 15mm thick plates, which were marked and kept together to be bonded. Each plate was planed on both sides using a Delta® planer to a thickness of 10.2 or 10.3 mm. The plates were equilibrated in an environmental conditioning room to 12% MC.

Laminate Preparation

Pressing was done in a hot press that was preheated to 200° C. The wood plates were removed from the conditioning chamber immediately before planning them to 10.0 mm thickness. Each plate was weighed and then resin was added to each face. The resin was spread immediately after application with a rubber roller and the plates were weighed again to verify resin weight. pMDI specimens had 0.0116 g/cm^2 of solids added to each face, and the PF specimens had 0.00626 g/cm^2 added to each face.

The laminate was closed, using the two sides from the same block. The laminates were wrapped in aluminum foil and placed in the hot press. Pressure was maintained at 100 psi for 20 minutes. The actual temperature of the platens was maintained at 200° C. After pressing, the aluminum foil was removed from the laminates and they were cooled under a fume hood. After cooling, the laminates were cut into 20 mm wide strips and trimmed to 200 mm long on a table saw. The specimens labeled and placed in the conditioning room and allowed to equilibrate to 12 % MC again.

DIELECTRIC ANALYSIS

In order to determine that the adhesive was fully cured in the hotpress, micro-dielectric analyses were performed. A laminate was prepared by following the same procedure as the fracture specimen laminates for PF adhesive and one was prepared using pMDI. A sensor for the dielectric analysis and one for temperature readings were placed in the bondline immediately prior to closing the laminate. The *HCSI – A* sensor was connected to a Eumetric System III Microdielectrometer. Conductivity readings were taken at 22 second intervals throughout the 20 minutes of hotpress time.

FRACTURE

A 35 mm pre-crack was cut into each specimen at the bondline using a bandsaw. Two 3 mm holes were drilled in the ends of each specimen, 10 mm from the end, and 5 mm from the top and bottom face (see Figure 4.1). The bondlines on each specimen were painted white using a liquid correction fluid, at least 24 hours before testing. A photocopy of a millimeter ruler was cut into strips and glued onto the side of each fracture specimen before testing. The paper ruler allows the user to record the length of the crack for each loading cycle. Crack lengths within the range of 50-150 mm were used in the data analysis. The load cell in the MTS machine was calibrated using the MTS software at the start of each test session. The load cell calibration was checked using a weight hanging from the load cell. The MTS machine reading was 0.5% higher than the known weight.

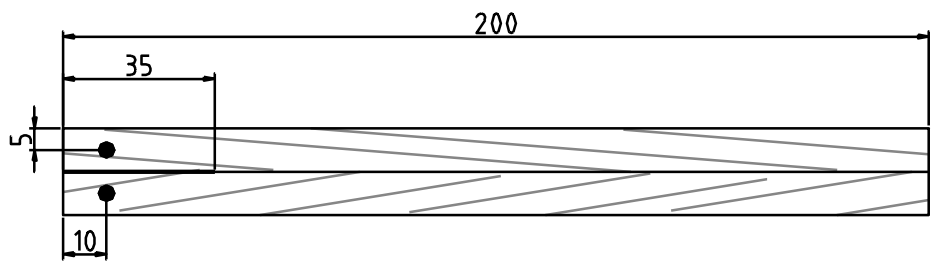

Figure 4.1 – Fracture specimen (dimensions in mm, thickness = 20mm).

Each fracture specimen was preloaded to 20 N to reduce the tendency of lateral movement in the test grips and pins. The MTS software is programmed to start the test at a loading rate of 1 mm/min. Once the crack tip starts extending, the load drops. When a 5% decrease in load is detected, the test machine holds the cross-head in that position for 45 seconds. During the 45 seconds, the operator records the crack length. The crosshead lowers to the starting position and the next cycle begins. These cycles are repeated until the crack has propagated completely through the specimen, or until the operator stops the test. The MTS software recorded the load and crosshead position. Both the critical crack initiation energy (G_{max}) and the crack arrest energy (G_a) are computed from the data using Equations [4.1] and [4.2] as shown below.

$$G_{\max} = \frac{P_{\max}^{\ 2}}{b} \frac{(a+\chi)^2}{EI_{eff}} \qquad \textbf{[4.1]}$$

$$G_{arrest} = \frac{P_{arrest}^{\ 2}}{b} \frac{(a+\chi)^2}{EI_{eff}} \qquad \textbf{[4.2]},$$

where EI_{eff} is the effective modulus of elasticity (E) and moment of inertia (I) product, found using the slope (m) of the linear portion of the $\sqrt[3]{C}$ vs. a curve for each specimen, as shown in Equations [3.4] to [3.13].

The average bondline thickness for each adhesive was measured from 5 different specimens, with 3 measurements taken from each specimen. Measurements were taken using image analysis software with a light microscope.

Analysis

The MTS software is programmed to start the test at a loading rate of 1 mm/min. Once the crack tip starts extending, the load drops. When a 5% decrease in load is detected, the test machine holds its position for 45 seconds. The operator records the crack length with the help of an LCD camera and monitor to increase magnification. The crosshead lowers to the starting position and the next cycle begins. The loading rate for the next cycle is the crosshead opening displacement per minute. This allow all cycles to have a constant time from zero displacement to the last crack initiation displacement. These cycles are repeated until the crack has propagated completely through the specimen, or until the operator stops the test. The MTS software records the load and crosshead position (Figure 4.2).

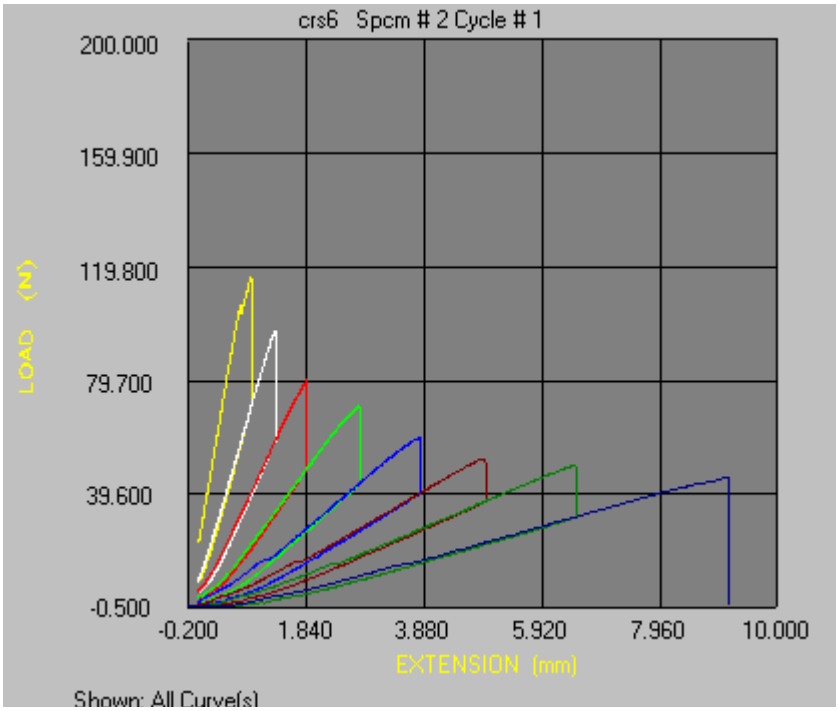

Figure 4.2 – Typical Load – Displacement Curve

Since fracture energy is a property of the individual specimen, it should be independent of crack length. See Figure 4.3 for an example of the fracture energies of a specimen that was used in this study. The correction factor χ (Equation 3.5) takes into account the compliance for each specimen and crack length, and calibrates the fracture energy for each compliance.

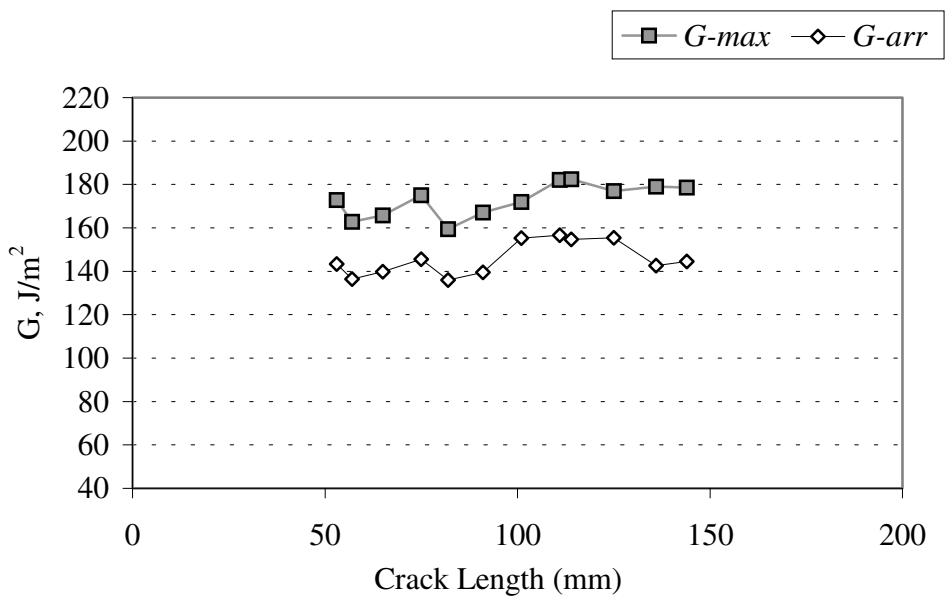

Figure 4.3 – Example Critical and Arrest Energies

Figure 4.4 is an example of the effect of the correction factor on the fracture energy of the same specimen as in Figure 4.3. The analysis using the correction factor correctly shows the fracture energy values in a horizontal trend as crack length increases. If the correction factor is not used, the trend increases as crack length increases.

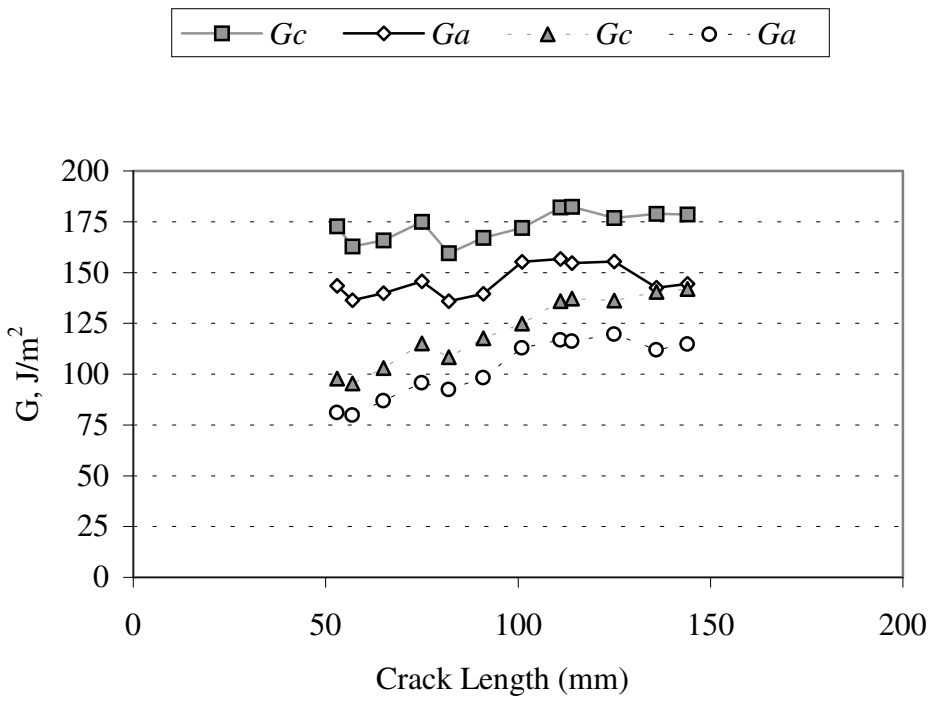

Figure 4.4 – Effect of Correction Factor

SUMMARY

The DCB fracture method using the correction factor is an effective tool for analyzing the fracture energy of the bondline. This method allows focusing on surface activity because it is not affected by variables that may change the results of other tests.

ACCELERATED AGING

Two accelerated aging schedules were followed: 2-hour boil cycles and a variation of ASTM D-1037 cycles. The boil cycle consists of immersion in water at 98.5° C for 2 hours, and oven drying at 102° C for 22 hours. The environmental cycle (variation of ASTM D-1037) starts at 50° C and 100% RH for two hours, then 102° C and 0% RH for 4 hours, then 2 hours at the first condition, and then 16 hours at the second condition (see Figure 4.5 – one cycle is shown). Control specimens were placed in the environmental conditioning chamber (20° C, 65% RH) until testing at the same time as the specimens that were exposed to the accelerated aging cycles. After the accelerated aging cycles were completed, the specimens were equilibrated for 4 days to 12% MC before testing.

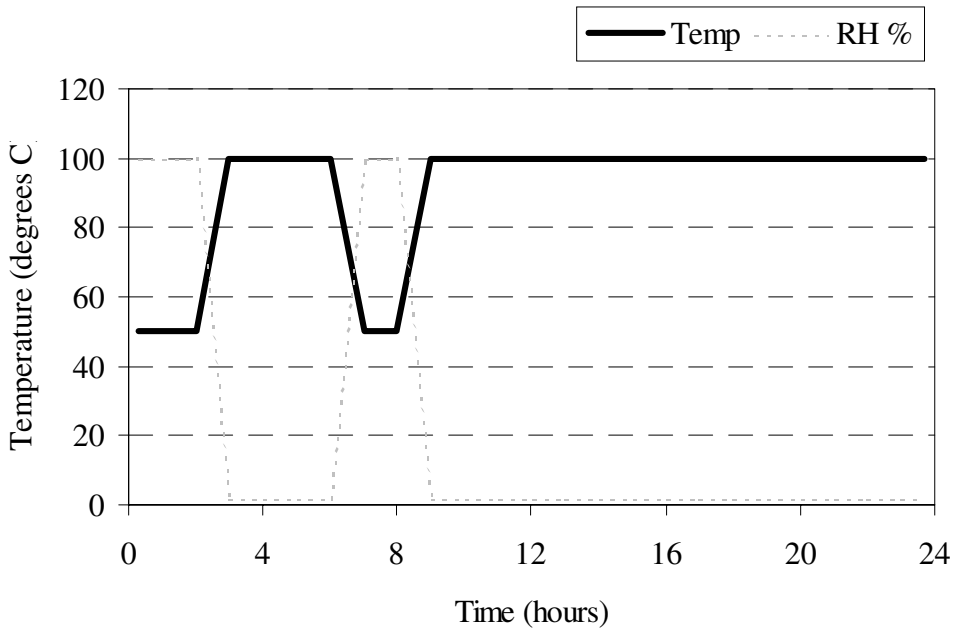

Figure 4.5 – Environmental Aging Cycles

Results and Discussion

DIELECTRIC ANALYSIS

Figure 4.6 is a graph of the conductivity of the PF and pMDI adhesives in the laminate during a 20 minute hot press. The data is from conductivity measurements at 10 Hz.

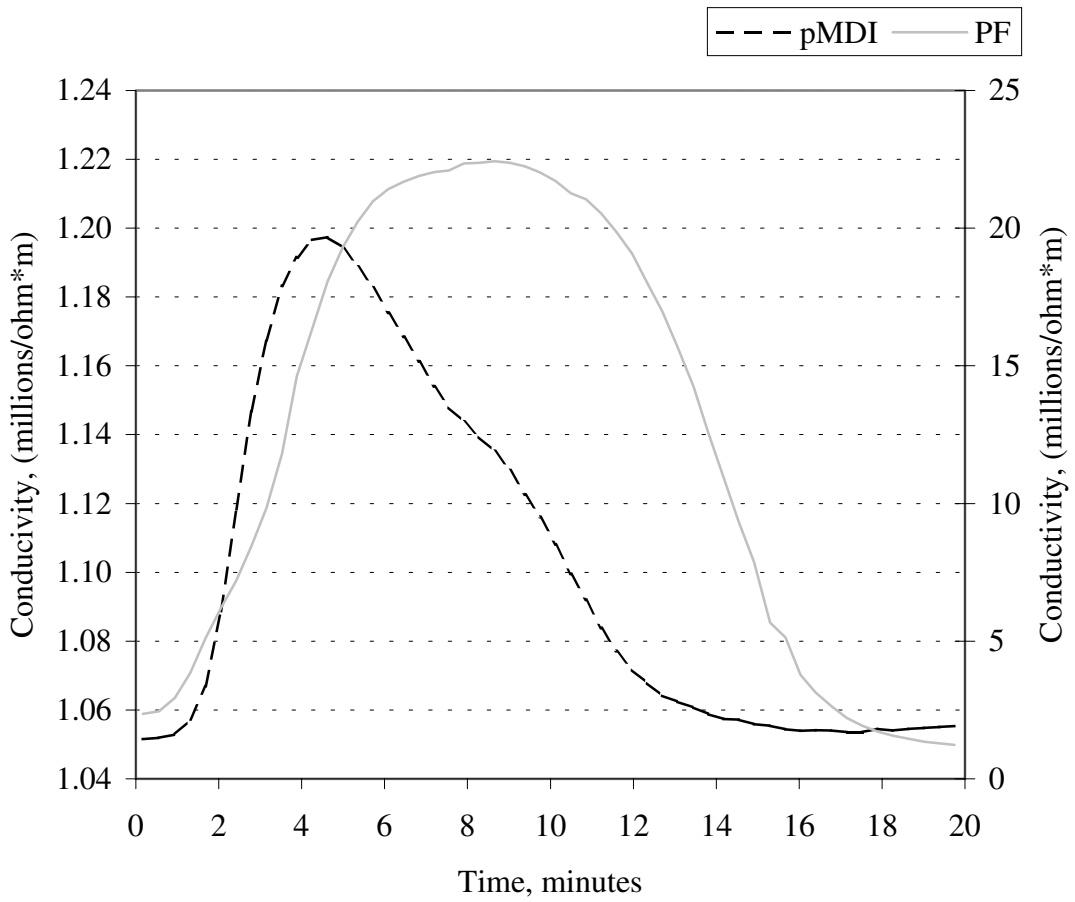

Figure 4.6 – Dielectric Analysis Data for PF and pMDI Adhesives

At the beginning of the hotpress time, the conductivity increases as the adhesive flows. The heat increase also causes faster cross-linking, which will tend to solidify the adhesive. After 20 minutes, the conductivity levels off. When the conductivity of an adhesive stops decreasing, it is an indication of a significant amount of cure. Further cure is possible, but it is not necessary for an adequate adhesive bond.

In Figure 4.6, the conductivity scale on the left is for pMDI and the scale on the right is for PF. The difference in the two scales is not important, since we are looking at the shape of the curves. The important information is how the conductivity levels off, or where the slope is nearly zero. The conductivity of pMDI levels off around 16-17 minutes. From Figure 4.6 we can assume that the pMDI is fully cured, and may have some degree of embrittlement. The conductivity of

PF adhesive does not quite have a zero slope at 20 minutes. This indicates that the PF may be not fully cured by 20 minutes, but the adhesive should be very close to cure when the laminate is removed from the hotpress.

The difference in the degree of cure, as shown by the dielectric analysis curves, may have an effect on the performance of the cured resins. If there is an effect, it might tend to weaken the pMDI due to overcure, and the PF might have a slightly higher fracture toughness than if the two adhesives were cured to the same degree.

The average bondline thickness for PF specimens was 77.6 µm (12.0 µm standard deviation), and the average for pMDI was 22.4 µm (8.76 µm SD). Measurements were taken using a light microscope. Generally for wood laminating adhesives, the solids content of pMDI adhesive is half that of PF. In this study, the solids content of pMDI adhesive was roughly twice that of the PF adhesive. pMDI specimens had 0.0116 g/cm^2 of solids applied to each face, and the PF specimens had 0.00626 g/cm^2 applied to each face. This amount of pMDI resins solids contributes to the pMDI bondline being thicker than normal, in this case about one-third of the PF bondline thickness. The thicker than normal pMDI bondline contributes to its fracture toughness and comparable performance to the PF fracture specimens.

FRACTURE ANALYSIS - BOIL TEST

For the first part of the analysis, the effect of the 2 hour boil cycles on fracture energy is addressed. In most of the discussion, the critical energy of crack formation (G_{max}) and the crack arrest energy (G_a) will be discussed together.

The experimental design of this study is a split-plot design (SPD) with the a randomized complete block design (RCBD) using repeated measurements for the whole plot. The *adhesive* factor is the main replication, and the two treatment factors are the *laminate* and the *number of cycles*. The fracture specimen is the experimental unit, and the surface of the bondline at one of the loading cycles that corresponds to a crack length is the observational unit. Each specimen had between 5 and 15 repeated measurements taken, which correspond to the crack lengths. On average, PF specimens had fewer crack cycles than the pMDI specimens.

The overall difference between controls PF and pMDI adhesives was significant. For the null hypothesis (H_o) that the adhesives are the same, the p-value is less than 0.0001 for G_{max} and is 0.1222 for G_a (see Table 4.3). At a significance level of $\alpha = 0.10$, we reject the null hypothesis and conclude that the adhesives are different. Although G_a values were not significantly different, the difference in G_{max} is sufficient to show that the adhesives are significantly different. An α level of 0.10 was used in all the significance tests for statistical comparisons in this chapter and in Chapter 5.

Table 4.3 – Overall average fracture energy values, standard error, and p-values for controls (SE is one standard error).

Adhesive	Average G_{max}	SE	p-value	Average G_a	SE	p-value
pMDI	167.5	9.32	<0.0001	144.8	5.91	0.1222
PF	229.1	10.29		151.7	6.97	

Bondline thickness has a great affect on the fracture energy. PF bondlines were thicker than pMDI bondlines, even though the amount of PF solids spread on each side was about half that of pMDI solids. pMDI generally has thinner bondlines than PF due to the amount of adhesive that penetrates further away from the bondline.

For this study, the performance of the adhesive after exposure to boil cycles is more important than the overall differences in pMDI and PF. The effects of the boil cycles on the pMDI specimens was different than the effects on the PF specimens. The average values and SE of the fracture energy data are found in Table 4.4.

Table 4.4 – Average fracture energy for boil cycles.

	# of cycles	Average G_{max}	SE	Average G_a	SE	N
pMDI	0	141.08	12.58	121.14	11.05	63
	1	173.03	12.46	152.18	10.95	62
	2	181.29	12.85	155.84	11.31	55
	3	208.48	12.98	181.29	11.44	55
	4	165.18	12.49	144.34	10.97	65
PF	0	213.16	13.29	142.65	11.73	36
	1	88.50	13.57	81.79	11.99	34
	2	104.47	13.40	97.26	11.83	38
	3	118.11	13.49	106.42	11.91	41
	4	117.46	13.47	105.97	11.90	40

In the above table, N is the number of fracture energy measurements of all specimens combined for that cycle. The pMDI specimens showed a slight increase in fracture energy over the first three cycles, and then decreased after the fourth. The average critical (maximum) and arrest fracture energies are shown in Figure 4.7. One standard error is shown in the error bars. The PF specimens decreased in fracture energy after one boil cycle, then stayed the same, as in Figure 4.8.

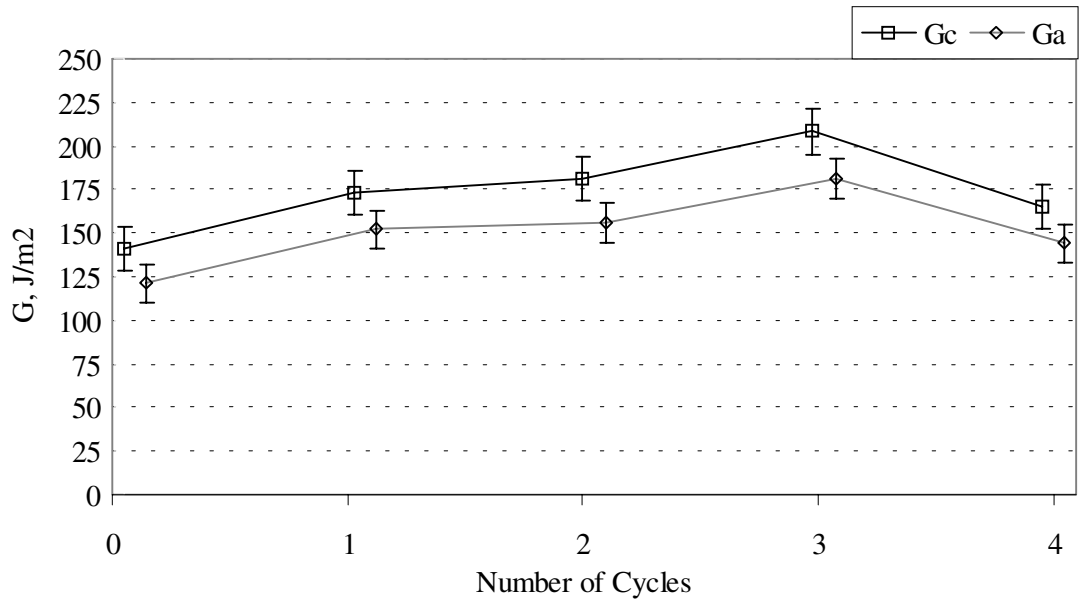

Figure 4.7 – pMDI Fracture Energy for Boil Cycles

A significant increase was found in average fracture energy from the controls to 3 cycles, at the $\alpha = 0.10$ level. Recall that one standard error is represented by the error bars in Figure 4.7. Significance was verified using *Ho*: given the pMDI adhesive, all cycles are equal.

The fracture energies for the pMDI adhesive were very resistant to the boil cycles. The fracture energy of the specimen was not reduced by boiling, but increased slightly. This could be due to the relaxation of internal stresses in the bondline as the wood is plasticized in the boil process. The reduction of internal stresses may increase the fracture energy of a bonded specimen.

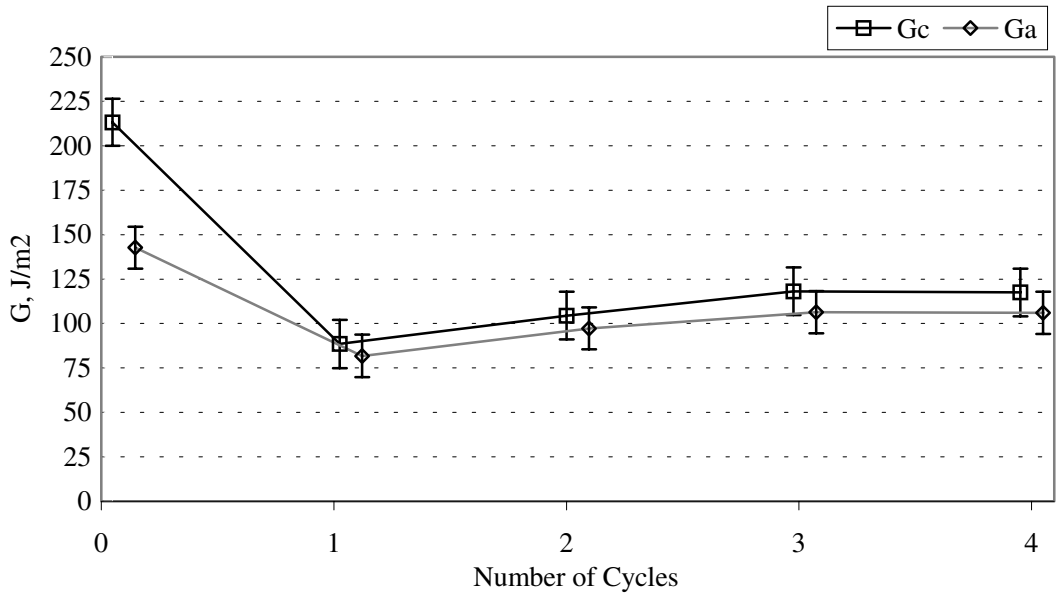

Figure 4.8 – PF Fracture Energy for Boil Cycles

The specimens made with PF adhesive had a substantial decrease in the fracture energy after one boil cycle (Figure 4.8). The fracture energy for the PF specimens dropped considerably after the first boil cycle. Although the average values actually increased after 1 cycle, there were no significant differences between cycles 1, 2, 3, and 4. The null hypothesis for this test was that all cycles have the same fracture energy. For *Ho*: all cycles equal, the *p*-value is 0.0014. At the $\alpha = 0.10$ level, we reject *Ho* and conclude that there is a significant difference between at least one pair of means. The p-values for a multiple comparison between cycles are shown in Table 4.5. All differences are significant if the *p*-value is less than α (= 0.10). Note that there are no significant differences in specimens made with pMDI at this significance level.

Table 4.5 – Averages and p-values for differences in number of boil cycles.

	Comparison of -		Difference in G_{max}	SE	*p*-value	Difference in G_a	SE	*p*-value
	# cycles	# cycles						
pMDI	0	1	-32.15	25.66	0.7217	-31.01	19.56	0.5057
	0	2	-41.05	25.88	0.5261	-34.29	19.81	0.4442
	0	3	-67.22	25.95	0.1193	-58.21	19.89	0.0648
	0	4	-27.31	25.66	0.8217	-25.30	19.56	0.6990
	1	2	-8.90	25.76	0.9966	-2.68	19.68	0.9999
	1	3	-35.07	25.82	0.6610	-26.60	19.75	0.6678
	1	4	4.84	25.57	0.9997	6.31	19.49	0.9973
	2	3	-26.17	25.99	0.8484	-23.92	19.94	0.7516
	2	4	13.74	25.81	0.9826	8.99	19.73	0.9902
	3	4	39.91	25.87	0.5510	32.92	19.81	0.4826
PF	0	1	125.68	25.6	0.0013	63.26	23.87	0.1075
	0	2	109.80	25.5	0.0043	47.69	23.80	0.3079
	0	3	94.99	25.7	0.0146	39.33	23.94	0.4935
	0	4	93.81	25.6	0.0155	37.30	23.86	0.5396
	1	2	-15.88	25.7	0.9701	-15.57	23.91	0.9639
	1	3	-30.70	25.9	0.7601	-23.93	24.10	0.8546
	1	4	-31.87	25.8	0.7318	-25.96	23.99	0.8131
	2	3	-14.82	25.8	0.9770	-8.36	23.99	0.9965
	2	4	-15.99	25.7	0.9692	-10.39	23.89	0.9918
	3	4	-1.17	25.9	1.0	-2.03	24.09	1.0

The drop in fracture energy after boiling may be caused by cleavage of bonds in the PF adhesive or in the adhesive-wood interface. Plasticizing the wood may also decrease bond integrity. There was a greater difference in G_{max} and G_a for controls than for other cycles. The difference in stress intensity factor (K) of the PF and pMDI may have some influence in the G_{max} and G_a differences. An estimated K value for the PF control specimens was 1.56 MJ/m^2, while the

estimated K for pMDI control specimens was 1.18 MJ/m^2. K values were estimated using Equation 3.1 and a very small Poisson's ratio was assumed. The higher K of PF adhesives indicates that the stresses and strains at the crack tip would have to be higher in order for the crack to propagate. This is related to a higher fracture energy and is shown by the difference in G_{max} and G_a.

FRACTURE ANALYSIS - ENVIRONMENTAL TEST

The environmental cycle study was performed using the same analysis as the boil cycle study. The overall difference between G_{max} values from PF and pMDI specimens was not significant for the environmental cycles; but the overall difference between G_a values was significant. The null hypothesis that the adhesives have equal means was not rejected for G_{max} (p-value = 0.4247); and the null hypothesis was rejected for G_a values (p-value = 0.0058). A summary of the overall differences is presented in Table 4.6. Table 4.7 contains the fracture energy data for each environmental cycle.

Table 4.6 – Overall average fracture energy values for environmental cycles.

Adhesive	Average G_{max}	SE	p-value	Average G_a	SE	p-value
pMDI	169.02	9.32	0.4247	144.82	5.91	0.0058
PF	180.65	10.29		108.23	6.97	

Table 4.7 – Average fracture energy for environmental cycles.

	# of cycles	Average G_{max}	SE	Average G_a	SE	N
pMDI	0	184.61	15.30	168.28	6.44	52
	1	145.03	15.96	121.78	6.94	48
	2	173.69	16.29	143.72	7.14	51
PF	0	247.25	16.72	155.10	15.13	18
	1	186.45	14.27	104.31	12.59	28
	2	141.05	13.35	85.92	11.51	37

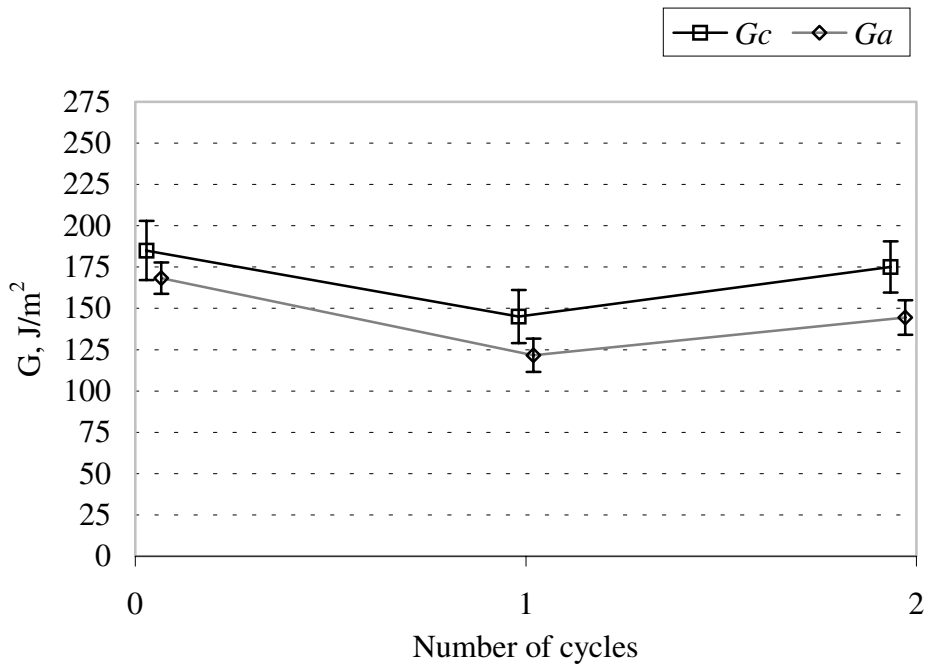

Figure 4.9 – pMDI Fracture Energy for Environmental Cycles

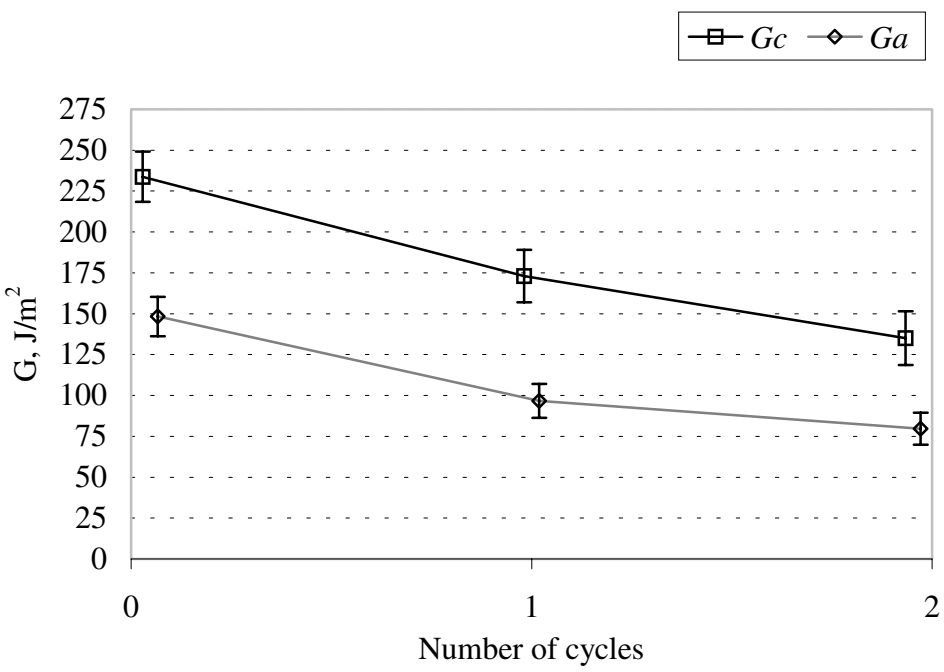

Figure 4.10 – PF Fracture Energy for Environmental Cycles

The fracture energy of pMDI adhesives decreased slightly after the first environmental cycle, then increased after the second. There was no significant difference between any values of G_{max} for the environmental cycles of pMDI, but the difference in G_a was significant between the controls and the first cycle, and between the first and second cycle. The average critical (maximum) and arrest fracture energies are shown in Figure 4.9. The error bars represent one standard error.

The fracture energy of PF decreased significantly after each environmental cycle (see Figure 4.10). The differences are significant if the *p*-value is less than 0.05, as shown in Table 4.8.

Table 4.8 – Comparison between average fracture energies and p-value for environmental cycles.

	# of cycles	# of cycles	G_{max} difference	SE	*p*-value	G_a difference	SE	*p*-value
pMDI	0	1	39.98	22.19	0.1216	46.75	13.79	0.0060
	1	2	-28.65	22.55	0.2471	-22.88	14.29	0.1387
	0	2	10.78	22.41	0.6474	23.87	13.98	0.0910
PF	0	1	60.61	20.67	0.0386	50.79	14.65	0.0125
	1	2	38.17	19.68	0.1280	18.40	13.42	0.2529
	0	2	98.78	21.57	0.0051	69.19	14.72	0.0035

SUMMARY

The effects of 2-hour boil cycles and the 4-day environmental cycle on fracture energy of pMDI and PF bonded wood specimens were found and discussed. The fracture energy of the PF specimens decreased after exposure to the accelerated aging cycles, and the fracture energy of the pMDI specimens was mostly unaffected by the accelerated aging cycle.

Conclusions

Based on testing of bonded wood DCB fracture specimens, the following conclusions are made:

-Up to four 2-hour boil cycles do not decrease the fracture energy of pMDI specimens.

-The fracture energy of specimens made using PF adhesive decreases after one boil cycle.

-The environmental cycle used in this study slightly lowered the fracture resistance of pMDI specimens after one 4-day cycle, but fracture energy values increased after the next 4-day cycle.

-The environmental exposure cycle used in this study reduced the fracture energy of PF specimens.

-pMDI is more resistant to the accelerated aging cycles of this study than is PF adhesive, when fracture energy is used as the durability criterion.

Recommendations for Future Work

The DCB fracture test method should be considered for use in assessing the quality of different types of adhesives and substrates. Some examples of possible applications include investigating the fracture energy of construction adhesives between sub-floor panels and joists, gluing two wood composites together, or investigating the effects of other surface treatments on the fracture of bonded composites.

CHAPTER 5 - FEASIBILITY OF FRACTURE TEST METHOD WITH WOOD COMPOSITES

Introduction

The application of the DCB fracture method to wood composite materials such as OSB and engineered lumber products is the purpose of this part of the investigation. One objective is to determine the feasibility of the DCB fracture test method for various composite specimens. Another objective is to compare the effects of a 2-hour boil test on fracture energy and internal bond (IB) strength.

Common DCB fracture specimens are made of two solid pieces of wood that are bonded together. For fracture testing, the crack direction is controlled by the grain of the wood material. The 5 degree converging grain angle forces the crack to follow the adhesive layer. In contrast, in the case of wood composite materials, a few difficulties must be overcome to foster a predictable crack extension.

The orientation of the strands, veneers, or other constituents of composite wood materials have some random component. There is a degree of variability in the size of furnish going into the material, and the flakes, veneers, or particles are aligned; however, this alignment cannot always be controlled exactly and a slight amount of variation is present in the alignment of the wood fibers in the final product. In some materials such as OSB, LSL, and PSL, there is some degree of overall orientation to the grain of the wood fibers, but such fibers may be in the range of 15 to 90 degrees from the principal material direction of the final product.

As in fracture of solid wood specimens, cracks propagating through wood material tend to follow the grain. Furthermore, it is difficult to control the location of crack initiation and location of crack propagation with respect to the bondlines. Bonded solid wood fracture specimens use a pre-crack that focuses the stress at the bondline between the two sides of the laminate. In a composite wood specimen, the interface between the adhesive and the wood is not a single plane, but a network of bondlines that follow the compressed and deformed surfaces of the wood fibers, either in flakes or veneers. At any point along a line in a composite

specimen, the line may be in the middle of wood fibers, in the middle of bulk adhesive, or at the interface between adhesive and fiber.

Since the grain angle in composites is too variable to be affected by a 5 or even 10 degree grain angle, a different approach must be used to try to direct the crack propagation. In the analysis of fracture mechanics, many tests have been developed that investigate the effect of a v-shaped notch to initiate a crack. The sharp angle of v-notches acts as a stress concentrator, producing almost infinite stresses with little energy input, or global stress. This chapter focuses on the value of notches or grooves in controlling the direction of crack propagation in fracture specimens.

Crack Propagation Test

In order to assess the usefulness of a composite specimen for use with the DCB method, a crack propagation test was conducted to see if the crack would follow the central plane of the specimen. If the crack splits the composite specimen into two cantilever beams, the dual cantilever beam (DCB) method could be an appropriate analysis of the fracture toughness of the composite. The crack propagation test discussed below is not a fracture analysis, rather it is a simple exercise using only a pass or fail criteria. The load and displacement of the crosshead were not recorded in this test.

Materials

Three types of wood composites were used in this study: ¾" Huber Advantec® Oriented strandboard (OSB); 2 x 4 Timberstrand® laminated strand lumber (LSL); and 2 x 4 Parallam® Parallel strand lumber (PSL).

Specimen Machining

LSL and PSL specimens were planed to 20mm thickness, and OSB specimens were left at their original 19.1 mm thickness. Specimens were machined to 20mm x 200mm using a table saw. Preliminary experiments showed that the specimen geometry would have to be modified to direct the crack propagation direction along the fracture plane, which is the central plane of the

specimen. Attempts to use solid specimens with no modifications resulted in the crack leaving the center plane and exiting through the top or bottom of the beam specimen. Initially, solid blocks of wood were bonded to the outside of the fracture specimen in order to avoid breaking the composite material with the loading pin (Figure 5.1). Subsequent trials showed that physical reinforcement of the holes was not necessary.

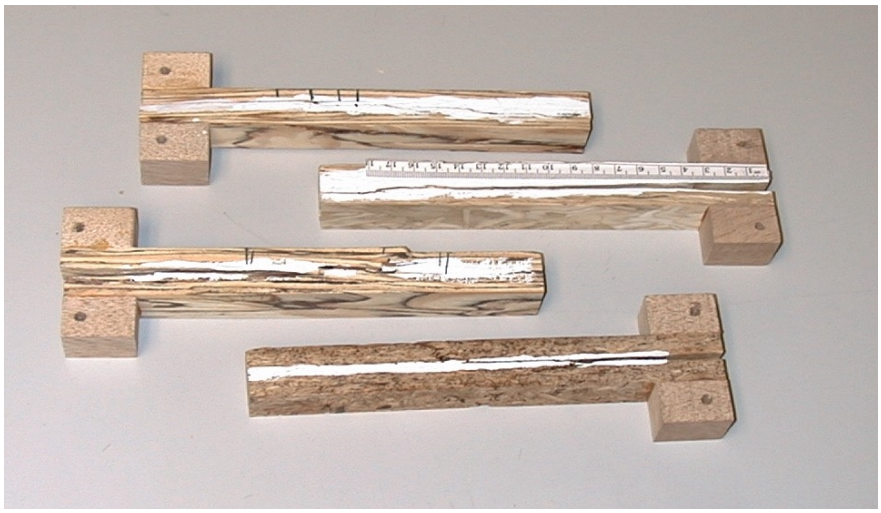

Figure 5.1 – Solid wood blocks for pin holes

Two specimen configurations were prepared and tested to determine if the crack propagates in the desired fracture plane (see Table 5.1). A V-shaped groove or a square groove was cut in the sides of the specimens. The 90° V-shaped groove was cut in each side of each of six specimens along the desired fracture plane using a 90° bit and router table. Also, square grooves were cut along the fracture plane using a table saw with a 3.5 mm thick blade. Both types of grooves were 5 mm deep on each side, leaving the specimen 10 mm wide at the fracture plane (Figure 5.2).

Table 5.1 – Number of specimens tested for each material.

	Square-groove	V-groove
Material	# of Specimens	# of Specimens
OSB	26	6
LSL	19	7
PSL	19	6

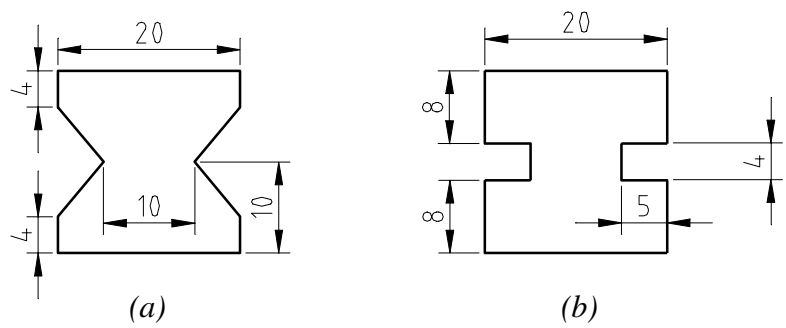

Figure 5.2 – Cross-section dimensions, mm, for V-groove (a) and square groove (b).

A crack propagation test was conducted to determine if the geometry of the specimen is such that the crack will stay reasonably within the desired fracture plane during testing. Specimen geometry that shows a high propensity for successful crack guidance should work well with the DCB fracture test setup. Initially, six V-groove specimens and six square-groove specimens were made from each of the three composite materials. Afterwards, 20 more square-groove specimens for each material were made and tested. Specimens were placed in the same grips that are used for solid wood fracture specimens, but instead of following the fracture test method, the specimens were loaded at a constant rate of 20 mm/minute until the crack had either propagated through the length of the specimen or until the crack left the desired fracture plane.

RESULTS AND DISCUSSION

The percentage of failed specimens from the crack propagation test is shown in Table 5.2. For the purpose of this test, a specimen was considered acceptable if the crack remained in the fracture plane for at least 170 mm. The specimens that failed were the ones in which the crack left the fracture plane before 170mm. The desired fracture plane was considered as any distance less than 2mm away from the central plane of the specimen.

Table 5.2 - Crack propagation failure rate for square and V-notch specimens.

	Square-groove		V-groove	
Material	% Failure	# of Specimens	% Failure	# of Specimens
OSB	27	26	67	6
LSL	21	19	57	7
PSL	47	19	100	6

If the crack left the desired fracture plane it either continued propagating through the thicker portion of the specimen, or one of the cantilever beams failed in bending, as in Figure 5.3.

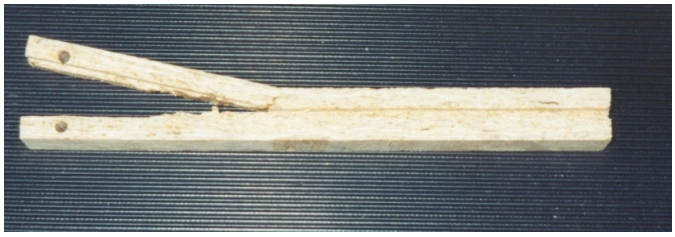

Figure 5.3 – Failed specimen, crack propagation test.

The square-groove was more effective in controlling the crack direction than was the V-groove. In the V-groove specimen, the crack follows a very gradual increase in effective width of the fracture surface as it strays from the centroidal plane of the specimen. In a square-groove specimen, there is no change in the width until the crack reaches the top or bottom of the groove.

As the crack reaches the top or bottom of the groove, there is a very abrupt change in the width of the fracture surface, from 10 mm to 20 mm. This abrupt change is a reason the square-groove works better for composite fracture specimens than the V-groove.

OSB and LSL square-groove specimens had the lowest crack propagation failure rate. Approximately one-fourth of the specimens tested would not have performed satisfactorily as a fracture specimen.

PSL specimens had the lowest success rate for the crack propagation test. Since PSL is made of veneers 2-3 mm thick and approximately 120 cm long, it has much larger solid wood components than both LSL and OSB. The difficulty in controlling crack propagation direction relates to the grain direction of the wood. Since the veneers have a random angle of orientation to the direction of the material in PSL manufacture, adjacent veneers in the final product may produce very different grain angles.

The crack propagation in PSL is not likely to be controlled by either a v-groove or a square-groove. This is likely due to the large percent volume of uninterrupted wood fibers in the PSL material.

CONCLUSIONS

Based on this investigation, the following was found:

-The square-groove specimen worked well for crack propagation tests with OSB and LSL fracture specimens.

-PSL is not suitable for use in the DCB fracture tests using V-groove or square-groove geometries.

Comparison of OSB Fracture Test Method and Internal Bond Test

The ASTM D-1037 standard give methods to assess the performance of composite wood products. The internal bond (IB) test portion of D-1037 is commonly used in industry. Many

production facilities use the IB test as a quality control test to check the IB strength of the panels as they come off the production line. This test allows the manufacturing plant to monitor the quality of the product and make necessary adjustments in case the panel has poor mechanical properties. Ease of use, prompt result capability, and low cost are some advantages of the IB test in the manufacturing plant.

There may be some advantages of using an energy-based evaluation of the integrity of wood composite materials, both in development of new products and in continual process verification. Although strength assessments don't compare directly to energy evaluations, a comparison of the IB test to the DCB fracture method was made in order to assess the potential use of the DCB test in a manufacturing environment.

Specimen Preparation

IB test specimens were prepared according to ASTM D-1037: Tensile Strength Perpendicular to Surface. Thirty blocks 51 mm square were cut from the Huber Advantec® OSB. The OSB blocks were bonded to the aluminum grip blocks using a hot-melt adhesive.

Half of the specimens were randomly chosen to be in a 2-hour boil, 22 hour dry cycle. IB tests were done on the MTS testing machine at a loading rate of 8% of the panel thickness per minute. The control specimens had a thickness of 19.1 mm, and the boil specimens had a thickness of 23.4 mm after boiling.

Experimental Design

15 internal bond specimens were controls, and 15 were tested after a 2-hour boil cycle. These were compared to 4 control fracture specimens of OSB, and 4 specimens tested after the same 2 hour boil cycle. The OSB fracture specimens were square-groove DCB fracture test specimens. Specimens were prepared according to section 5.2, and fracture analysis followed the methods described in section 4.2.

Results and Discussion

The average maximum stress for control and boil specimens was 58 and 16.4, respectively (see Figure 5.4). The IB strength of this OSB was reduced 72% by the 2-hour boil.

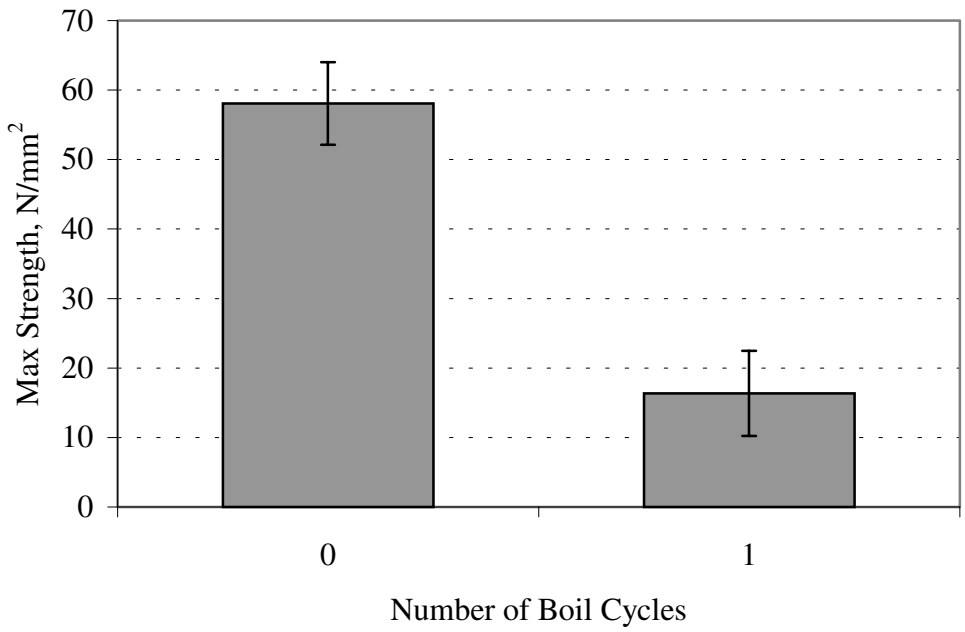

Figure 5.4 – Internal bond strength of OSB before and after 2-hour boil cycle

Error bars in the figures show one standard error. The boil cycle for the fracture specimens reduced G_{max} and G_a by 38% and 54%, respectively (see Figure 5.5).

Standard error was lower for fracture tests. Part of the lower variability is due to a greater number of measurements on the fracture specimens. The IB test only has one measurement taken per specimen (at maximum load), and the fracture tests had an average of 6 measurements per specimen.

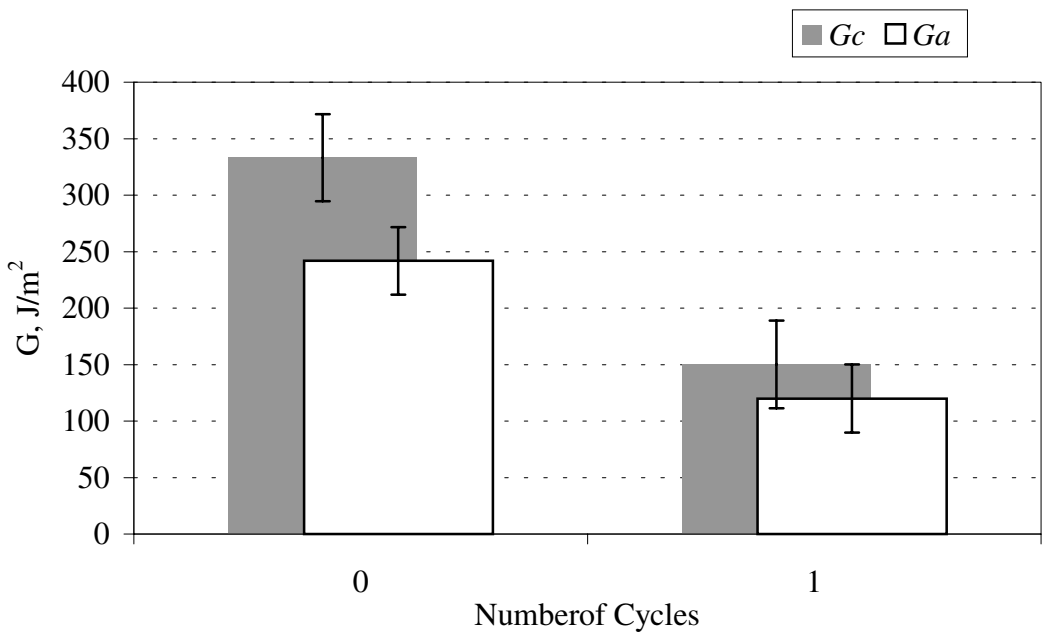

Figure 5.5 – Fracture energy for OSB before and after 2-hour boil cycle.

The break in the IB specimens occurred at the center of the thickness of the panel for both the controls and the boiled specimens. The most common fracture for the DCB specimens was characterized by the crack remaining in the thickness of the groove for the length of the specimen. The fracture description did not change for the controls or the boiled specimens. In the fracture tests, 25% of the specimens failed before testing was completed and had to be rejected.

SUMMARY

Composite fracture specimens were evaluated using a crack propagation test and square notches were found to be successful with OSB and LSL specimens. Also, the IB test was compared to the DCB fracture test using a 2-hour boil exposure and OSB material.

Conclusions

The following conclusions were based on the testing from this chapter:

-The 2-hour boil test had a greater affect on the IB test than on the fracture test.

-The fracture test showed less variability than the IB test.

Recommendations for Future Work

There is a great deal of work to be performed in the area of fracture testing of wood composites. A thorough study of the differences in the DCB fracture test method and the IB test is necessary to further investigate the potential of the fracture method in industry. Market analyses of the use of the fracture test, advantages, and disadvantages should be considered. Correlations between results from the IB test and the fracture test should be addressed as well.

CHAPTER 6 - CONCLUSIONS

SUMMARY

The effects of 2-hour boil cycles and the 4-day environmental cycle on fracture energy of pMDI and PF bonded wood specimens were found and discussed. The fracture energy of the PF specimens decreased after exposure to the accelerated aging cycles, and the fracture energy of the pMDI specimens was mostly unaffected by the accelerated aging cycle.

Composite fracture specimens were evaluated using a crack propagation test and square notches were found to be successful with OSB and LSL specimens. Also, the IB test was compared to the DCB fracture test using a 2-hour boil exposure and OSB material. The IB test is a strength-based test and the fracture test is energy-based. The fracture energy test method has less variability than the IB test method.

CONCLUSIONS

Based on bonded wood DCB fracture specimens, the following conclusions were made:

-Up to four 2-hour boil cycles do not decrease the fracture energy of pMDI specimens.

-The fracture energy of specimens made using PF adhesive decreases after one boil cycle.

-pMDI is more resistant to the accelerated aging cycles of this study than is PF adhesive, when fracture energy is used as the durability criterion.

-Square-groove specimen worked well for crack propagation tests with OSB and LSL fracture specimens.

-PSL is not suitable for use in the DCB fracture tests using V-groove or square-groove geometries.

-The 2-hour boil test had a greater affect on the IB test than on the fracture test.

-The fracture test showed less variability than the IB test.

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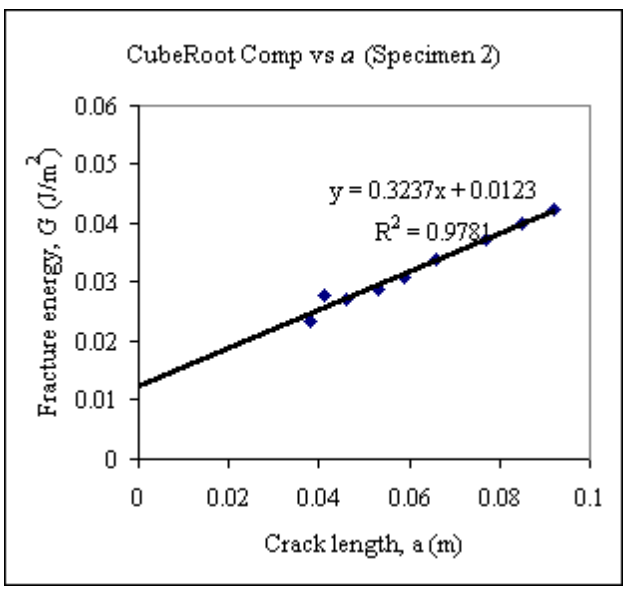
APPENDIX A – FRACTURE TEST DATA

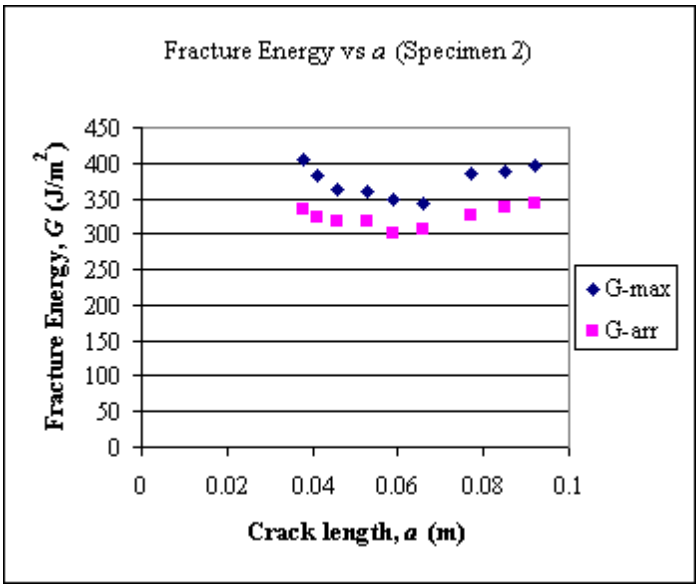
Sample Data

Specimen2	Ext	Ext	Slope	Comp	Crit Ld	Arr Ld	C^1/3	G-max	G-arr	E
Cycle End	mm	m	N/mm	m/N	N	N	(m/N)^1/3	J/m^2	J/m^2	N/m^2
1	38	0.038	78.1	1.28E-05	165.6	150.6	0.0234	404.33	334.40	1.38E+10
2	41	0.041	46.4	2.16E-05	154.8	142.2	0.0278	381.71	322.10	9.19E+09
3	46	0.046	50.6	1.98E-05	141.9	132.7	0.0270	362.57	317.08	1.20E+10
4	53	0.053	41.2	2.43E-05	130.7	122.9	0.0290	360.92	319.12	1.25E+10
5	59	0.059	33.6	2.98E-05	120.5	112.2	0.0310	348.52	302.16	1.23E+10
6	66	0.066	26	3.85E-05	111.5	105.5	0.0338	342.97	307.05	1.17E+10
7	77	0.077	19.5	5.13E-05	106.9	98.2	0.0372	385.38	325.21	1.19E+10
8	85	0.085	15.8	6.33E-05	100.2	93.7	0.0399	387.29	338.67	1.18E+10
9	92	0.092	13.1	7.63E-05	95.9	89.1	0.0424	396.25	342.05	1.15E+10

m	b	x	Eleff	E
0.324	0.012	0.038	19.647	1E+10

Average	374.44	323.09	1.19E+10
Std Dev	21.49	13.61	1.2E+09
COV	17.43	23.73	9.884775
count	9	9	9





VITAE

Christopher Russell Scoville was born in Alexandria, Virginia in 1973. Even before high school, Chris was making tree-houses and furniture with wood scraps around the garage. He graduated from West Potomac High School in 1991, having participated in Band and Crew. Chris started his college studies in Mechanical Engineering at Northeastern University, but found his way to the Wood Science Department at Virginia Polytechnic Institute and State Univesity and graduated with his Bachelor's of Science in 1997. Chris served a full-time mission in Venezuela for two years and returned for his Master's degree in Wood Science at Virginia Tech. Having received a research fellowship from the Wood-Based Composites Center, Chris finished his degree in 2001. He has since taken a Research Scientist position at J. M. Huber Corporation in Commerce, Ga.