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Factors that Lead to Failure with Wood Adhesive Bonds

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ABSTRACT: Understanding what makes a good wood adhesive is difficult since the type of adhesive, wood species, bonding process, and resultant products vary considerably. Wood bonds are subjected to a variety of tests that reflect the different product performance criteria in diverse countries. The most common tests involve some type of moisture resistance; both wood and adhesive factors influence this moisture related failure. Models have been developed to explain the adhesive performance. The proper microscopic and spectroscopic techniques can lead to a better understanding of the failure mechanism so that improved adhesives can be designed and bonding processes can be optimized.

KEYWORDS: wood, adhesive, failure, microscopy, spectroscopy, moisture, epoxy

1 INTRODUCTION

Adhesives have been used for making bonded wood products for millennia. However, for much of this time bonding was limited to furniture and other interior products. It has only been in the past century that wood products, bonded with adhesives, have been used structurally with partial or full exterior exposure. These applications place much greater demand on the adhesive and have required stronger and more durable bonds.

Like many product areas, wood bonding was a technology application, long before it was studied as a scientific area. The first publications on how adhesives bond wood proposed either mechanical interlock [1] or specific adhesion [2]. Marra presented an in-depth discussion of wood adhesives and bonded wood products [3], with an important new contribution being the consideration of the adhesive bondline as being a series of links in a chain. Although in reality the bondline is a continuum of changes from the bulk wood on one side through the bulk adhesive to the bulk wood on the other side of the bond, the model is still useful for describing failure zones in the bondline. The literature can be confusing on terminology, and thus it is important to be clear on what the terms mean. Adhesion is the force necessary to separate two materials at their common interface. On the other hand, adhesive strength involves the energy to break a bonded assembly at its weakest point whether that is at the interface, or elsewhere in the bondline or wood. While the interface is defined as the discreet line separating the adhesive and wood, the interphase involves the transition area between the bulk property of a substrate and the substrate/adhesive interface. Given the critical nature of understanding this difference, we have illustrated this concept using a slightly modified version of Marra's chain link model in Figure 1, and a photograph of an actual bondline in Figure 2.

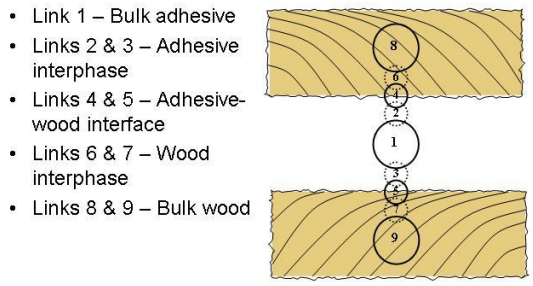

***Figure 1**: Modified Marra model for the adhesive bondline [3].*

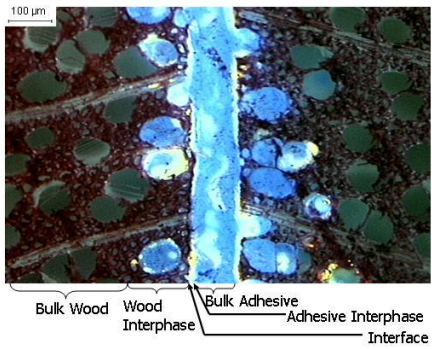

***Figure 2:** Cross-section of an epoxy bondline*

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As Marra noted, failure in wood bonding is seldom at the interface once a good bond is formed [3]. It is true that some wood surfaces are hard to wet for good bonding due to surface inactivation, and extractive build up on the surface [4]. Low density wood species are obviously likely to fail in the wood. However, even strong wood species can fail in the wood if the cured adhesive is very strong. The wood interphase (bulk wood to interface involving adhesive penetration of the wood) can be weak due to crushing of the cells or other surface defects. Although adhesive *interphase* failure is common, it is often attributed to *interface* failure because it can be difficult to detect a thin adhesive film on the failure surface. An example is given later when discussing epoxy bonding.

In any case, it is important to understand why some wood bonds provide good service and others fail. Most testing data only gives performance values, although some tests require the use of ASTM D5266 to quantify the percent wood failure [5a]. Though this method tries to specify where failure occurs, it can be difficult to see some types of failure and the method is only for laminated products.

2 TESTING OF WOOD BONDS

Bonded wood products need to meet many requirements [3,4,6]. A few critical ones are listed here since they apply to most bonded wood products. In general, the properties of the adhesive cannot be determined separately from those obtained in a bonded wood product, so most of the adhesive tests are based upon the premise of testing the bonded item to determine the adhesive strength properties. However, there are several key factors that must be kept in mind when examining the available data. The first is that the data needs to be obtained on the wood used in the final application. It is important to understand the effect of wood variability when interpreting the data since there is a wide variability between species and even within species. While some of the adhesives have a consistent formulation, many of those used for commercial board production are tailored to be most efficient for a particular mill's equipment, operation and wood source. There can even be seasonal changes due to differences in temperature and humidity that can influence adhesive performance.

2.1 CREEP

It is desirable that wood adhesives not distort (creep) over time due to static loads on the product. The sagging of a glulam beam or bookshelf is not acceptable because it usually leads to the need for repair or replacement. Solid wood itself creeps, especially under higher humidity and temperature conditions, but it is worse when the adhesive adds to this tendency. A new ASTM method D7966 involving new specimens and test apparatus addresses wood swelling issues and excessive wood failure in the ASTM D4680 standard meant for more moderate conditions [5e,f].

Avoiding creep in bonded wood products requires that the adhesive be a thermoset instead of a thermoplastic. By definition thermoplastics can flow at elevated temperature conditions without breaking covalent chemical bonds. Thermosets involve polymers that are cured by heat or added chemicals, including water for polyurethane and isocyanate adhesives. These cured polymers can be rigid or flexible, but in all cases return to their original shape when the force is removed unless some covalent chemical bonds are broken. Although creep is a controllable issue with adhesives used in laminated products, it is still an important issue with composites, such as particleboard and fiberboard. In any case tests have been established for either the adhesive in structural products [5b,c] or bonded product in the case of composites [7e,f]. For composites, the issue is more complex since the creep of the wood is included as well as the spot adhesion of the adhesives. In contrast to structural adhesives where no creep is allowed for the adhesive, creep exists for composites and the adhesive needs to minimize this. Better creep resistance without the use of more adhesive is desirable and can lead to higher performing bonded products.

2.2 HEAT RESISTANCE

Two types of heat conditions exist for bonded wood products, ambient heat and fire. The ambient heat tests are generally met by most thermoset adhesives. The ASTM standard involves putting the specimen under a static load and then subjecting the loaded specimen to heat [5e].

However the fire tests are a more complex issue. While fire departments understand the risk of a roof or floor collapse with solid wood, there is concern that bonded wood structures may fail sooner. The problem is that an adhesive may either thermally soften or chemically degrade causing the member to lose its strength, leading to structural collapse. In North America, the fire test involves heating solid and bonded wood (side-matched) up to the temperature where wood begins to lose its strength, usually about 210 °C [5f]. Then the heated bonded specimen is subjected to a compression shear test and needs to retain about as much strength as does the side-matched solid wood after heating. This test does not sort out the mechanism of failure. ASTM 7998 [5e] method provides for a hot test using an Automated Bonding Evaluation System (ABES) that has been shown to be able to separate adhesive thermal softening using heating times of minutes from adhesive degradation using heating times of hours [8]. Using the ABES test provides useful information on why failure takes place, and allows for designed improvement in the adhesive. In addition the test is easy to run at different temperatures to elucidate the temperatures at which the loss of strength is occurring, especially given that the properties of adhesives in wood bonds may be different than to the adhesive by itself.

2.3 MOISTURE

A key test for most bonded wood products involves moisture resistance. Whether the test involves soaking followed by a wet strength test, soaking and drying followed by a dry strength test, a delamination test after soaking and drying, boiling water exposure, thickness or length swell tests after soaking for composites, all are accelerated tests to substitute for long term exposure. The accelerated tests assume that a few cycles of more extreme

conditions can predict the failure resistance of a bondline after many cycles of less drastic changes that would occur in a fatigue test. There is some support for this with a structural bonded wood product test [9,10]. Moisture is an important issue for bondline delamination and care must be taken that the wood being bonded is close in moisture to what it experiences in its end-use environment to minimize the effect of moisture changes [11].

3 WOOD AND ADHESIVES

3.1 WOOD SWELLING

Wood will change in all three orthogonal dimensions with changes in moisture, but the changes are not even. For example, the wood swells more tangentially than radially, which swells more than longitudinally [12]. Furthermore, swelling of the wood is greater as the wood density increases. This not only means that some species swell more because of their higher density, but also wood of non-uniform density displays non-uniform swelling. This can lead to uneven swelling between earlywood and latewood, which can be hard for the adhesive to accommodate.

3.2 ADHESIVE RESPONSE TO SWELLING

As wood swells and shrinks, adhesives do not follow with the same volumetric expansion; thus, there is significant strain between the wood and adhesive. Figure 3 illustrates three cases: a) where the interfacial strain is not relieved, b) where the adhesive modifies the wood to reduce the interfacial strain, c) where the adhesive has enough flexibility to reduce the interfacial strain. In case (a), bond failure is most likely to occur with considerable changes in the wood moisture content, while in cases (b) and (c), the bondline is much more likely to stay intact. This emphasizes that the adhesive interaction with the wood is important for the strength of the bond under wet or changing moisture conditions [13]. In addition one needs to consider that different groups of adhesives may need to perform different functions in order to form a moisture durable bond.

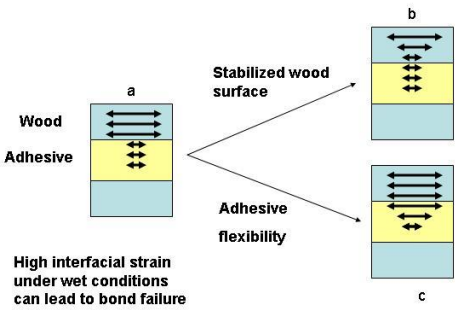

***Figure 3**: Adhesive-wood strain model*

The strain model necessitates that researchers understand whether the adhesive being tested falls into class (a), (b), or (c). Prior literature had not considered that there was more than one class of adhesive; thus, they would try to explain adhesive failure using a common model. Based upon the polymer morphology, interaction with wood cells, and mechanical properties of the adhesive, wood adhesives can be grouped as *in-situ* polymerized or pre-polymerized [14].

3.2.1 In-situ polymerized

One group of adhesives consists of mainly monomers or oligomers that polymerize during the bonding process [14]. This can be done by adding a catalyst, co-reactant or supplying some type of energy, usually heat. Catalysts include latent acids for accelerating amino resin curing, while co-reactant examples are formaldehyde to couple resorcinol oligomers and water for reacting with isocyanate and polyurethane adhesives [15]. Heating is a necessity for compressing the wood into composite boards and has been used for a long time in accelerating the bonding of plywood [3]. Radio frequency curing works in some cases, but is less prevalent.

Given the highly cross-linked nature of *in-situ* polymerized wood adhesives and their rigid backbone, these polymers will not withstand the strain induced by wood swelling if they do not infiltrate and stabilize the cell wall near the wood surface as in Figure 3, case (b). The positive effect of the reduced swelling is illustrated on a cellular level in Figure 4 [16]. In addition to reducing strain, the *in-situ* polymerized adhesives being low in molecular weight and good flow properties should also be able to repair damaged wood surface cells that could form a weak wood boundary layer [17].

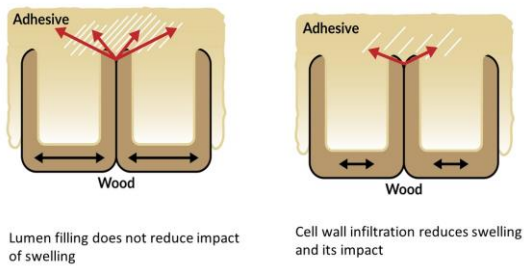

***Figure 4:** Effect of wood swelling on adhesive strain*

The *in-situ* polymerized adhesives are the most common group of wood adhesives and include the phenolics and amino resins [14]. These adhesives are not only rigid due to very limited mobility in their backbones, but also due to their high degree of cross-linking. These characteristics rule out case (c) strain relief (Figure 3). However the low molecular weight versions of these resins are known to reduce the wood swelling and shrinking when the moisture exposure changes [18]. This group of adhesives also includes the isocyanates, such as polymeric diphenyl-methane diisocyanate and epoxy resins. The isocyanates provide good water resistance, while the epoxies do not as discussed later in this paper.

3.2.2 Pre-polymerized

The other group of adhesives is polymerized prior to the bonding process and therefore the molecules are too big to infiltrate and stabilize the wood cell walls against

swelling. Thus, these adhesives need to be flexible enough so that the adhesive can absorb the strain when the wood cell walls are swelling and shrinking. In contrast to the *in-situ* polymerized adhesives, the pre-polymerized adhesives have flexible backbones to allow chain mobility. These are usually cross-linked to provide creep and moisture resistance, but the cross-linking needs to be low to avoid too much rigidity. Typical examples are cross-linked poly(vinyl acetate) and polyurethanes. Proteins, the first wood adhesives, also fall into this group.

3.3 LOCATION OF FAILURE

Because adhesives are used to hold two substrates together under a variety of conditions, bond strength of the bonded material is the most important factor. For improving an adhesive, it is valuable to understand that when the failure took place, where the failure is in the bondline and why the failure occurred. Most wood adhesives are strong enough to give failure deep in the wood under dry conditions. Deep wood failure is easy to determine, but shallow wood failure is more difficult to determine [5a].

Failure within the bondline whether measured using a shear test or a delamination test can be harder to evaluate. The failure type is usually not determined after a delamination test, except to measure the percent of the bondline length where failure has occurred. This is unfortunate because opening the bondline can allow for determining if failure was due to poor cohesive strength, starved bondline (over-penetration), lack of transfer to the non-coated side, wood factors, etc. For improving performance, it is recommended that delamination areas be opened up so the failure can be better understood using the techniques discussed below and in Marra [3].

If a shear test is used, the failure surfaces are visible. In fact, some test methods require the use of a method for quantifying percent wood failure, such as ASTM D5266 [5]. This method also calls for distinguishing between deep and shallow wood failure, but the latter can be difficult to apply in some situations in that the contrast between the wood and adhesive may not be that large. Trained analyst can distinguish the subtle difference, but untrained people are much less consistent. Distinguishing a few layers of wood fibers/tracheids on the failed surface my require techniques that enhance visibility, such as fluorescence combined with microscopy (as will be discussed below). Additional methods have been tried [19,20], but none have been well accepted. However, additional methods can be used to understand failure as illustrated using epoxy failure situation below.

4 FAILURE ANALYSIS

4.1 EPOXY BONDED WOOD EXAMPLE

Although it had been known for a long time that epoxies are not very durable adhesives for wood bonding, there has not been a good explanation as to where and why these bonds fail. Epoxies provide durable bonds to many substrates including metals and concrete, but it has been demonstrated that a variety of epoxy adhesives do not give bonds of limited moisture durability [21,22], except when a primer is used [23] or the change in moisture is less severe [24]. The role of the primer was thought to be as a coupling agent. However, Figure 2 shows that the epoxy adhesive had a very good flow into the lumens, but this was not sufficient to provide moisture durable bonds [25]. Surface coupling could not add significantly to bond strength if there was only **loss of adhesion** upon wetting. This is an important issue because loss of adhesion is often used in the literature to rationalize poor adhesive performance under wet conditions, but the surfaces are usually not analyzed in any great detail.

4.2 NORMAL VISUALIZATION

The first stage is to use the unaided eye to see where failure is taking place, such as ASTM D5266 [5a]. Although this method is good for determining deep wood failure, it becomes more subjective for failure within the bondline. The images in Figure 5 show what looks like adhesion failure for the epoxy adhesive given the wood like texture on both the adhesive side of the bond and on the wood side of the bond.

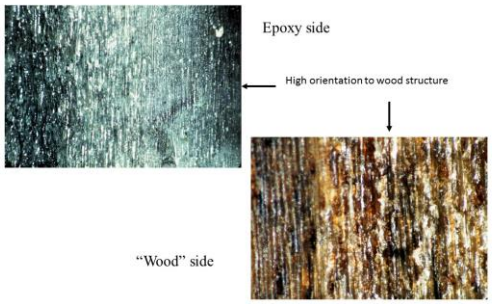

Figure 5: *Surfaces from compressive shear test after water soaking with the adhesive surface on the right and wood surface on the left.*

4.3 ADDITIONAL MICROSCOPY

However, we decided to go beyond the normal failure examination. Microscopy allows for selection of specific spots for more detailed observations of the different failure locations; this process can be enhanced by using various light sources at different orientations to the surface. Careful selection of locations for further examination and going stepwise in increasing the magnification reduces false interpretation of the general failure mechanism by selecting an unrepresentative location.

Adding fluorescent dyes/pigments or suppressing the fluorescence of lignin in the wood can be very helpful in distinguishing the adhesive from the wood compared to normal light wavelengths. An area of concern is the migration of a dye on its own rather than as part of the adhesive; this can be mitigated by chemically attaching the dye to the adhesive. In some cases using very fine, well-dispersed pigments can help overcome the problem of selective migration of added dyes into the wood cell walls.

The fluorescence of the epoxy used in Figures 2 and 5 can be used to study the failure location after the compressive shear test. Even without any magnification, what looks like a wood surface in Figure 5, shows traces of epoxy florescence under a standard ultraviolet light. The higher

magnification of a failure surface shows in the center portion of Figure 6, a portion that is normally considered to be adhesion failure, actually has a thin layer of epoxy on the wood surface. The failure from the wood swelling causes the texture of the epoxy to be oriented with the wood grain and not perpendicular to the grain, which is what would be expected for failure induced by the applied tensile load on the bondline. At the sides are thicker epoxy layers, and again the failure is more parallel to the grain rather than perpendicular.

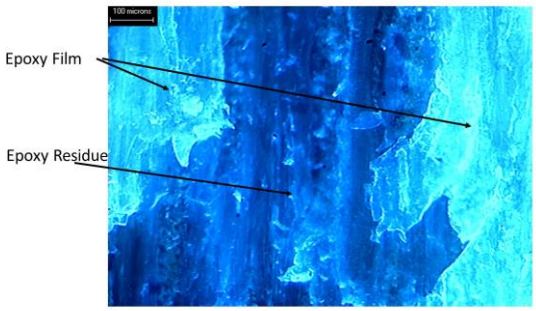

Figure 6: *Florescent microscopy magnification of failure surface of epoxy bonded wood*

Greater magnification of the failed bond surface by using scanning electron microscopy (SEM) can add additional understanding of bond failure location. Again, this technique needs to be applied to representative portions of the wood surface as well as comparing an adhesive coated surface to a sample of the wood itself. It is also helpful to have an unbonded wood surface to use for comparison because there is no color contrast. It is valuable to look for cells filled with adhesive and failure that is more adhesive like than wood like. The SEM can add supporting information, but it is unwise to use it as the main tool.
Atomic force microscopy (AFM) has been used for examining adhesive failure because it can examine both the physical response of the surface as well as the topography. The normal roughness of a wood surface is larger than AFM tip, however making the analysis difficult. In addition, caution needs to be used as these surfaces need to be representative of the main failure location.

4.4 SPECTROSCOPY

A number of spectroscopic techniques can be applied to examine bond failure surfaces. One readily available method is Fourier transform infrared spectroscopy (FTIR) that can be applied using attenuated total reflectance and diffuse reflectance to look for functional group differences between the adhesive and the wood on the failed surface. As the example for the epoxy adhesive shows in Figure 7, both sides of the failure surfaces resemble more the epoxy FTIR spectrum than they do the spectrum of the planed wood surface. Thus, using both sampling methods for the FTIR supports that the failure is in the epoxy and not an adhesion failure.

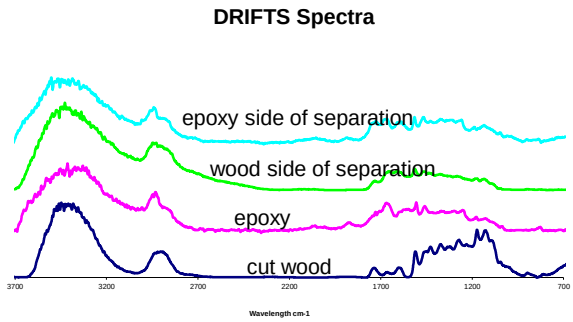

Figure 7: *FTIR of wood side and epoxy side of failed bondline, along with control wood and epoxy samples.*

X-ray photoelectron spectroscopy (XPS) can provide information on the oxidation states of the different surface atoms. However, like any small scale surface analysis great care needs to be exercised to avoid contamination, and the area examined needs to be representative of the whole. Again, we are using the epoxy adhesives as an example of this method's utility. Figure 8 shows the XPS spectra for both the wood and epoxy sides of the failed bondline adhesive which resemble more the epoxy spectrum than the wood one. This again gives additional data to confirm that it is not adhesion failure, but failure in the epoxy interphase near the wood surface.

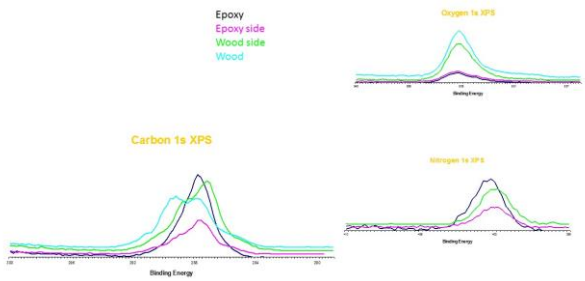

Figure 8: *XPS of wood side and epoxy side of failed bondline, along with control wood and epoxy samples.*

5 EPOXY FAILURE AND ROLE OF PRIMER

From all the available evidence, the failure of the epoxy bonded wood laminates seems to be in the epoxy interphase. This fits with Case (a) in Figure 3 and the left hand image in Figure 4. Additional data involve the fact that epoxies have less delamination to wood that swells less [26]. Although the amine curing agent of the two component epoxy should infiltrate the cell walls given its polar nature and its low molecular weight, the epoxy is less polar making it less likely to infiltrate the cell wall. The epoxy and amine would need to find each other in the cell wall and react to provide any stabilization of the cell wall. There are flexible epoxies, but these are modified for impact resistance, not as much for providing some ductility.
The ability of polyethyleneamine and HMR to provide durable epoxy bonds [9,23] led to the proposal that they are stabilizing the wood surface [13]. Despite HMR being an excellent primer, the mechanism for providing strength is still not well understood. The HMR has been shown to

work well on a number of wood species and several types of adhesives [27]. However, wood surfaces are not understood well enough to be sure of any definite mechanism for HMR [28].

6 CONCLUSIONS

Understanding why wood bonds fail involves a detailed knowledge of adhesive-wood interactions and detailed information on where failure takes place by using a variety of microscopic and spectroscopic techniques. Although deep wood failure is relatively easy to determine, and bulk adhesive failure can be determined by the unaided eye, failure in the interphase regions of the bondline are more difficult.

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   d. D7247 − 07a, Standard Test Method for Evaluating the Shear Strength of Adhesive Bonds in Laminated Wood Products at Elevated Temperatures
   e. D3535-07a Standard Test Method for Resistance to Creep Under Static Loading for Structural Wood Laminating Adhesives Used Under Exterior Conditions.
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