LOCATION OF FAILURE IN ADHESIVE-BONDED BUTT JOINTS
Abstract

The exact location of failure was determined in butt joints of slash pine (Pinus elliottii Engelm.) wood bonded with two experimental epoxy-resin adhesives. Failure in adhesion between two bonded wood samples did not occur, rather failure within the adhesive was always in cohesion. Methods are described for determining whether failure occurred in adhesion at the interface between adhesive and substrate, or within either of these.

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Succeeding U.S. F.S. Research Notes will be:

FPL-0178 Effects of Adhesive Formulation and Age on strength of Bonded Butt Joints

FPE-0179 Contributions of End-Wall and Lumen Bonding to Strength of Butt Joints
LOCATION OF FAILURE IN ADHESIVE-BONDED BUTT JOINTS

By

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Although some aspects of transverse bonding of wood with adhesives have been studied in detail, others have received only scant attention. Particularly neglected have been location of joint failure, effects on joint strength of adhesive penetration into open cell lumens, and effects of adhesive viscosity as affected by the age of the mix at the time it is applied to the surfaces to be bonded. More research is also needed on the relationship of wood structure to joint strength: for example, the relationship of wood contact area to overall adhesion, and the individual contributions of cell lumens and cell walls to joint strength. The present investigation was made to explore these aspects of bonding in butt joints fabricated from one coniferous species and two formulations of one basic adhesive.

A critical evaluation of the exact location of fracture is relevant to this study. If it is assumed that failure in adhesion, even across restricted areas of the bonding surfaces, implies non-wetting of the substrate by the adhesive, then in calculating joint strength a corresponding allowance must be made for the seduction in bonding surface. This Note is concerned with methods of establishing whether failure occurred in adhesion at the interface or in cohesion either within the adhesive or the substrate.

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Review of Literature

Most investigators agree that adhesion is due to molecular attraction. However, some authorities claim that, when a joint is broken, the resultant breaking stress is not a measure of the molecular attraction between the adhesive and substrate, because failure does not occur at the interface between them (3, 4, 23, 25, 26).\(^4\) While in many cases it may be clear that failure occurred in the adhesive or in the substrate, in others it may appear to occur at the interface between them, at least to the unaided eye. Broken test pieces of any material have not been examined carefully enough to determine positively whether or not a monolayer or thin film of one material may remain attached to the surface of the other one (indicating fracture in cohesion and not adhesion) (1, 4, 15, 25).

There is some evidence that the first molecular layer is adsorbed on a substrate more strongly than subsequent layers (9, 30). The degree of orientation of molecules in an adhesive is inversely proportional to the distance from the adhesive-substrate interface, suggesting that failure should occur in a layer other than the one first adsorbed at the interface (5, 26, 27). In a few cases in which attempts were made to study the location of joint failure using metals, plastics, or rubber as substrates, failure appeared to be in cohesion within either the adhesive or substrate, but not at the bonding interface (8, 13, 22, 23). In one study of steel joints tested in shear impact at elevated temperatures, it was reported that failure occurred in both adhesion and cohesion, but it was not clear whether molecular bonds had been established between the adhesive and the steel in the areas in which failure was apparently in adhesion (10).

Experimental Approach

To join two pieces of wood end to end and obtain a state of complete homogeneity may not be possible. Wood is strongest in tension parallel to the grain, and adhesives presently available are not capable of developing the full capacity of wood strength. However, increased joint efficiency might result if more information on interrelationships between adhesive and substrate structure were available.

\(^4\)Underlined numbers in parentheses refer to Literature Cited at the end of this Note.
The stiffness of an adhesive in a joint has not been quantitatively evaluated with success. Even though the adhesive may be intrinsically stronger than wood, decreased joint strength is generally related to stress concentrations within rigid joints. These stress concentrations arise either from incomplete wetting or stress risers as a result of volumetric shrinkage or void formation from solvent loss, as well as from geometrical factors of joint design. In addition, incomplete glue films, entrapped air bubbles, adsorbed moisture, and contamination all detract from the theoretical levels of strength attainable in joints. Since stress concentrations are intensified with increasing rigidity of the adhesive, it may be possible to minimize them by using a ductile adhesive formulation to provide some stress relief. To avoid some of the undesirable physical characteristics associated with conventional wood-bonding adhesives, it would be advantageous if the adhesive had a solvent-free base, a high solids content, a minimum of shrinkage upon cure, and versatile mechanical properties.

In evaluating strength of composite wood-glue joints, it must first be established whether or not, and to what extent, the adhesive wets the wood. In wetting the substrate the free energy required to establish molecular bonds may change with adhesive age or viscosity before the adhesive is applied to a bonding surface. Therefore, the effect of adhesive viscosity in relation to joint strength should be investigated. Functions of the adhesive are: (a) to support the walls of wood elements near the adhesive boundary, (b) provide continuity between the adhesive film and solid wood, (c) increase contact between the adhesive and the substrate by wetting the surface to be bonded and by penetration into open cell lumens. The extent of the surface contact area will be influenced by thickness of cell walls, total length of lumen perimeter per unit of cross-sectional surface area, and depth of adhesive penetration. Therefore, to evaluate the effect of substrate structure on joint strength it seemed reasonable to confine the study to a single coniferous species and obtain extremes in cell dimensions by separating annual rings into earlywood and latewood components.

To maintain continuity between adhesive and substrate, flat and parallel end-grain surfaces for bonding were smoothed with a microtome to provide a surface free of bent, torn, or frayed fibers. The presence of such fibers would have impeded adhesive wetting and penetration and created a weak boundary condition at the wood-adhesive interface. The study was restricted to butt joints because cell wall thickness and lumen diameters could be more accurately measured on transverse surfaces than on those cut at various angles to the grain, such as in scarf or finger joints.

The relative contributions to joint strength from either cell wall or lumen bonding can be obtained by blocking the open cell lumens to confine bonding to
transections of cell walls (along the plane of the microtome cut—hereafter referred to as “end-walls”) or blocking wood-adhesive contact to the exposed end-walls to confine bonding to open lumens. The application of boundary layers to isolate end-walls or lumens requires a smooth transverse surface free from disrupted fibers.

The strength of a joint and location of failure with a given adhesive can be controlled by standardizing bonding techniques. For a given species, joint strength may be affected by such wood characteristics as moisture content, temperature, structure and density on either side of the glueline, and grain angle. It may also be affected by some properties of the adhesive including duration and temperature of cure and glueline thickness.

Materials and Methods

Selection of Materials

The wood of a conifer, slash pine (Pinus elliottii Engelm), was selected because of the homogeneity of its xylem elements. In addition, its straight grain facilitates splitting of annual rings into earlywood and latewood. Moreover, when properly selected, its wide annual rings have a high proportion of late-wood, required for the preparation of specimens of adequate size from both earlywood and latewood.

A single 4-foot bolt, containing 25 annual rings, was obtained from the butt portion of a slash pine log. The bolt was split into several pieces to obtain vertical orientation of wood elements; it was cut into flitches, kiln-dried to 12 percent moisture content, and then separated into 1/8- by 1/8- by 3-inch specimens of either low-density earlywood (average specific gravity 0.27) or high-density latewood (average specific gravity 0.61). Compression wood was not present in samples tested. Specimens were taken only from the outer five annual rings where latewood width was adequate to obtain the desired sample size. To simulate conditions used in gluing wood commercially, moisture content of specimens was controlled by conditioning them to constant weight at 80° F. and 65 percent relative humidity (equilibrium moisture content, 11.7 percent).

Epoxy resins were used. Although typical epoxy resins are rigid when cured, they can be modified to various degrees of ductility and strength by addition of compatible substances (14). Epoxy resins contain nearly 100 percent solids (24).

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5 Specific gravity determined on ovendry weight and green volume basis (2).
resulting in shrinkage of less than 1 percent during cure (6). No by-products of condensation are released during cure, thus minimizing void formation in the glue line (12, 14, 24, 28), and little or no gluing pressure is necessary during the cure cycle. Since epoxy resins can be cured at room temperature, the absence of heat and their low volumetric shrinkage minimize adverse effects from internal drying and thermal stresses (14, 29). Furthermore, epoxy resins exhibit a high degree of adhesion to cellulose (16, 17, 18, 19, 20).

Two formulations were used for bonding. The unmodified epoxy resin (Epon 828, Shell Chemical Co.) simulated a conventional rigid adhesive system. A modified ductile form which provided some degree of stress relief was obtained by adding 30 parts by weight of a compatible and reactive polysulfide flexibilizer (LP-33, Thiokol Corp.) to 70 parts of the base resin. Each formulation was cured with 10 percent diethylene triamine (DETA) by weight of epoxy resin in the mix. The modified resin exhibited a ductile mode of failure rather than a brittle one that was obtained with the unmodified form. The modified resin had an average tensile stress of 5,900 p.s.i. (pounds per square inch), the unmodified resin 12,300 p.s.i. (based on 15 cast solid specimens of each). Pot life for both formulations was about 30 minutes.

The effect of adhesive age, and hence of adhesive viscosity, on joint strength was investigated by allowing the fresh adhesive mix, with curing agent, to polymerize for intervals of 0, 10, and 20 minutes before application to the bonding surface. These intervals were arbitrarily selected from the measured pot life of the two formulations. Viscosities of the adhesives were measured at these intervals by the falling sphere method of Gibson (7) as modified by Merrington (21).

**Special Treatments**

To evaluate the respective contributions of lumen or end-wall bonding to joint strength with the ductile adhesive, vaseline or zinc stearate powder was applied to the wood surface before bonding. Neither vaseline nor zinc stearate contamination of the ductile adhesive affected its average bulk tensile stress. Efficient boundary layers were established by fabricating 20 joints of aluminum substrates, with the ductile adhesive on one bonding surface and the contaminating material on the opposing surface. There was no evidence that wetting of the aluminum substrate occurred through the boundary layers.

The boundary layer of vaseline was applied to the wood surface by squeezing "dry" a lint-free gauze that had been dipped in heated vaseline (120° F.) and
Figure I.—Aluminum assembly jigs used for joint preparation:

Top, Jig used for sawing wood specimens in half and smoothing the matching surfaces to be bonded. The A and B portion of the assembly is detachable from the C portion for mounting in a microtome chuck.

Bottom, Jig for holding five joint specimens during cure. The slot permits insertion of a 15-mil wire gage to adjust glue-line thickness and accommodates a silicone gasket to prevent adhesive from migrating from the joint under the influence of gravity.

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touching it several times to the wood surface. When the entire surface appeared “glossy” under a microscope with oblique lighting, the boundary layer was considered established. Microscopic examination verified that this method of application prevented filling or plugging of the cell lumens with vaseline.

The zinc stearate boundary layer was applied by tamping the powder into open cell lumens with a glass rod. Excess powder was removed by first blowing off the surface and then making a skimming cut with a microtome knife to clean residual powder from exposed cell walls.

**Specimen Preparation**

Three types of specimens were required for testing. The first two provided information on tensile strength of the basic materials and the third provided similar information on the composite joint.

Adhesive specimens were cast of both formulations (15 each) to provide semi-qualitative information on tensile stress and mode of failure, attainable by either adhesive mix. The procedure for casting solid specimens was as follows: The adhesive was mixed with the curing agent, blended for 1 minute, poured into a mold assembly, evacuated for 2 hours at 27° C. to remove entrapped air, and cured for 10 days at 80° F. and 65 percent relative humidity. Specimens were then removed from the assembly and shaped to concentrate stresses in the narrowed portion of the specimen. The cross sectional area of the narrowed portion (1/8 by 1/8 inches) was equal to that of a glue film in a bonded joint.

Fifteen specimens each of earlywood and latewood were shaped into miniature tension specimens to obtain basic wood strength.

Because it is important to maximize end-to-end grain matching on either side of the glueline in the bonded specimens, the wood pieces to be used for joint fabrication were placed in a specially prepared jig (fig. 1) and sawn in half with a jeweler's saw to minimize kerf. The matching ends of a specimen, at the cut, were frozen with liquid carbon dioxide to increase rigidity of cell walls, and smoothed with a microtome knife. After surfacing (fig. 2) the specimens were reconditioned to constant weight before bonding of receiving special surface treatments prior to bonding.

**Joint Fabrication**

Joints were fabricated by dipping matched untreated or treated wood surfaces into the adhesive at the preselected viscosity intervals (0, 10, or 20 minutes
after mixing). Each matching specimen half, with adhesive on its surface, was placed in a holder (fig. 1), with care taken to orient cut tissues as in the original uncut specimen. All gluelines were assembled immediately, and adjusted to 15 mils in thickness with a suitable gage, and cured for 240 hours at 80° F. and 65 percent relative humidity. After cure, bonded specimens were removed from the holders, trimmed of excess glue, and tested in tension.

Testing

Tension tests were made in an Instron machine using a head speed of 0.05 inch per minute. A free span of 1.5 inches between tensile grips was maintained for loading specimens in the machine.

Photomicrographs were made at a magnification of 35X for all fracture surfaces after test to provide a base to measure actual fracture areas planimetrically. Fracture areas were measured from negatives rather than from prints to eliminate possible variability due to shrinkage of the paper.

Experimental Design

The study was conducted in two phases. The first included a comparison of the efficiency between joints bonded with the rigid and the ductile adhesive. The second was concerned with the special surface treatments applied prior to bonding. Because of the vast number of anatomical measurements that would be required, this phase was concerned with the adhesive system that provided the strongest joints as determined from phase one.

To compare the effect of adhesive formulation (rigid or ductile) on joint strength, three replications were made of five joints within each of the three viscosity-age classes and each of the wood types (earlywood and latewood). Hence, 180 specimens were involved.

For the special surface treatments, three replications were made of five joints in each of three viscosity-age classes in each wood type (earlywood and latewood) for each of the surface treatments (applied boundary layers of vaseline and zinc stearate) and with only the ductile adhesive, to give a total of 180 bonded specimens.

In all cases each replicate of five specimens within the three viscosity-age classes was made from a single 50-gram batch of adhesive mix.
Figure 2.--Appearance of wood surface of slash pine after smoothing with a microtome knife in preparation for bonding (90X).

Figure 3.--Use of fluorescence microscopy to determine presence of adhesive. Here the surface was prepared by cutting a bonded area with the microtome knife at a slight angle to the wood surface, revealing the adhesive (top), wood-adhesive interface, and underlying solid wood (bottom). The adhesive in the upper portion masks the natural fluorescence of wood, as compared to the solid wood where the film has been removed.
Microscopic Techniques

A Leitz Ortholux microscope equipped with an Ultrapak incident light attachment and a Xenon-150 lamp was used. For white light illumination the lamp was fitted with a diffusion disk and neutral density filters to control light intensity. Additional filters such as green, or blue (daylight) were used where necessary to provide contrast for photography.

Photographing the transverse section (plane of fracture through the glue film) of all fracture surfaces at 35X provided the base negatives from which the fracture areas were measured planimetrically; in addition, they provided the initial survey from which the obvious fracture locations were readily determined. Where fracture appeared to be at the interface between adhesive and wood even at increased magnification (up to 625X), other procedures were used to determine whether failure was in cohesion or adhesion.

Fluorescence Microscopy

Observations of fracture surfaces under ultraviolet illumination made use of the natural fluorescence of wood. Where adhesive was present on the cut end-walls of cells, natural fluorescence of the wood was masked (fig. 3). This technique was used when the epoxy film on the cut end-wall was so thin and transparent that under white light its presence could not be detected. Under ultraviolet light such films were easily detected because, for the same optics and optical system, ultraviolet illumination increases resolution over that for white light.

To obtain ultraviolet illumination, a Corning glass filter No. 5840 (peak transmitted wavelength, 365mµ) and a Leitz BG-38 (far-red adsorbing filter) were inserted into the light train. Ultraviolet adsorbing filters (euphos, 2.5mm.) were fitted to the eyepieces to protect the eyes and prevent fogging of the film from stray radiation. Most of the observations were made with ultraviolet illumination. However, most of the photomicrographs were made with white light because exposure time with it averaged only 1/50 second whereas with ultraviolet light exposure times of 2 to 5 minutes would be required.

Surface Staining

Neither white nor ultraviolet illumination at any magnification was suitable for viewing specimens which had an applied boundary layer of vaseline because
Figure 4. --Diagrammatic representations of the location of failure.

A. Normal (untreated) wood surfaces. Type I failure has occurred in the bulk of the adhesive (heavily stippled area). Type II failure has occurred adjacent to the wood-adhesive interface. The arrow marked "Photograph" indicates the direct ion the photomicrograph shown as Figure 5 was taken.

B. Surfaces receiving special treatment of vaseline or zinc stearate prior to bonding. Failure occurred as Type II within the adhesive and within the applied boundary layers.
the Vaseline could not be distinguished from the adhesive. However, after testing various staining procedures as outlined by Johansen (11) a saturated solution of safranin in alcohol was selected to stain the specimens. Before applying stain to the fracture surface the inhibiting vaseline was first removed with benzene. Where adhesive was present on the end-walls of cells, no staining of the wood occurred. This staining procedure could have been used with all specimens but it was not used extensively because of the more rapid survey procedure available with the incident fluorescence.

Results and Discussion

Microscopic examination of fracture surfaces in the different specimens showed conclusively that both adhesives, at each of the three levels of adhesive age investigated, wet the exposed wood surface, and no bonding occurred through the applied boundary layers of vaseline or zinc stearate. All joint failures were in cohesion within the adhesive film, Where vaseline or zinc stearate were applied to a bonding surface, cohesive failure of the adhesive was accompanied by cohesive separation within the boundary layers.

The location of failure did not differ among viscosity classes or between wood types within a single adhesive formulation. The plane of failure was perpendicular to the direction of the applied tensile load, and fracture initiated either at a small entrapped air bubble or surface crack at or near the edge of the gluefilm. However, the location of the fracture plane differed in joints made with the rigid and ductile adhesives. The fracture plane in all joints bonded with the rigid adhesive occurred fairly deep within the bulk of the adhesive, whereas in all joints bonded with the ductile adhesive it was near the wood-adhesive interface. These two locations of failure were designated Types I and II, respectively and are illustrated in figure 4.

There was no difficulty in identifying Type I failures because they were apparent during the initial survey conducted at low magnification (35X). Seventy percent of Type II failures were identified at magnifications of 100X or less. The remainder were identified at magnifications of less than 300X since fracture lines could be seen in the adhesive extending across the end-walls of the adhesive-filled lumens (fig. 5). Fracture lines were taken as indicative of cohesive failure within the adhesive. These fracture lines were confined to end-walls only in specimens with the zinc stearate-plugged lumens, and to lumens only in specimens having an applied boundary layer of vaseline on the cut end-walls.
Figure 5.--Typical appearance of surface view of Type II failure of joints fabricated from untreated surfaces. Both photomicrographs show that failure was in cohesion. In A, appreciable amounts of adhesive obscure the underlying wood structure. In B, the film of adhesive is very thin but can still be detected by fracture lines extending from the adhesive-filled lumens across the cell end-walls.
At the three viscosity levels investigated, both adhesives were effective in wetting the wood. Since wetting occurred and the adhesive failed in cohesion, the load at failure apparently measured some bulk property of the adhesive rather than the forces of adhesion between the adhesive and substrate. Therefore, the load-carrying capacity of joints could be calculated from the actual external dimensions of the cross section, without a concomitant reduction in effective bonding areas. In addition, the total wood-adhesive contact area and depth of adhesive penetration could be determined, and the influence of wood structure on joint strength could be investigated directly. These interrelationships will be discussed in future papers.

**Literature Cited**


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