STRUCTURALLY DURABLE EPOXY BONDS TO AIRCRAFT WOODS

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ABSTRACT

The lack of structural durability of epoxy bonds to wood has always been a problem for fabricators of adhesive-bonded wood products intended for service in exterior environments. Although epoxy adhesives develop dry shear strengths that exceed the strength of wood itself, the epoxy bonds fail in delamination once exposed to the severe stresses of water soaking and drying. Recent research at the Forest Products Laboratory (FPL) demonstrated that a hydroxymethylated resorcinol (HMR) coupling agent physicochemically couples to both epoxy adhesive and lignocellulosics of wood to produce bonds of extraordinary structural durability. The purpose of this report is to demonstrate how HMR enhanced adhesion of three epoxy adhesive formulations (based on diglycidylether of bisphenol-A resin) on two softwood and two hardwood species commonly used to construct aircraft components. A new FPL formulation met requirements for resistance to delamination, shear, and deformation in HMR-primed lumber joints on all four species of wood, in accordance with ASTM Specification D 2559. Two commercial formulations performed well in tests of resistance to delamination and shear on three woods of moderate density, but failed requirements on high-density yellow birch. Without an HMR coupling agent, none of the epoxy formulations had sufficient resistance to delamination to meet ASTM requirements on any of the species tested.

The. Forest Products Laboratory (FPL) has received many inquiries over the years about the structural durability of epoxy bonds to wood, usually from builders of wood aircraft and boats, and manufacturers of specialty wood products such as architectural posts and railings. When reporting failures, users invariably have described bonds that delaminated on exposure to water in an exterior environment. Epoxy adhesives do not equal resorcinols in durability of bonds to wood. They develop dry shear strengths that may exceed the strength of the wood itself, but the bonds fail in delamination once exposed to the severe stresses of water soaking and drying.

In the early 1960s, Olson and Blomquist (7) developed more durable epoxy formulations. One formulation in particular, FPL 16, was capable of exposure to 120-hour boil and dry tests. This formulation was modified, marketed privately as FPL 16A, and is still a popular adhesive with builders of wood aircraft. In *Sport Aviation* magazine, Myal (6) described FPL 16A as the ultimate glue. Caster (5) of the Weyerhaeuser Company, in cooperation with the Dow Chemical Company, made further progress toward more durable epoxy bonds by priming wood surfaces with a 2 percent aqueous solution of polyethylenimine. The surface treatment enabled two adhesive-bonded assemblies to perform comparably to small, solid-wood specimens in accelerated aging and exterior exposure tests.

A continuing need for structural epoxy bonds with greater resistance to stresses from repeated water soaking and drying led to further exploration of chemical surface treatments at FPL. This work led to the discovery that hydroxymethylated resorcinol (HMR) physiochemically bonds to both epoxy adhesive and wood lignocellulosics to produce lumber joints that are extraordinarily resistant to delamination (9,10). Although molecular structures and size distributions are still being determined, the structure of trihydroxymethylated resorcinol and its trimer are suggested in **Figure 1.** The possible coupling reactions of HMR with the wood cellulosics and an epoxy adhesive based on diglycidylether of bisphenol-A epoxy adhesive (DGEBA) are shown in **Figure 2.** HMR may covalently bond with the epoxy adhesive by forming ether linkages through condensation reactions between hydroxyls of epoxy and hydroxymethyl groups of HMR (position 5, **Fig. 2).** Other available hydroxymethyl groups of the coupling agent may form ether linkages with the primary alcohols of

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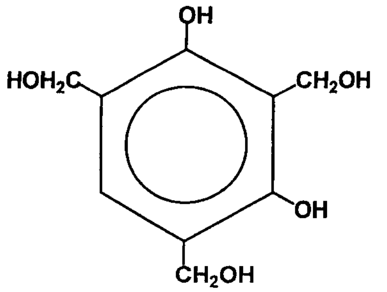
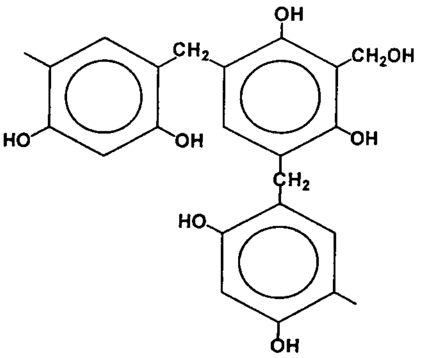


Trihydroxymethyl resorcinol



Hydroxymethylated resorcinol trimer

Figure 1. — Structure of trihydroxymethylated resorcinol and its trimer.

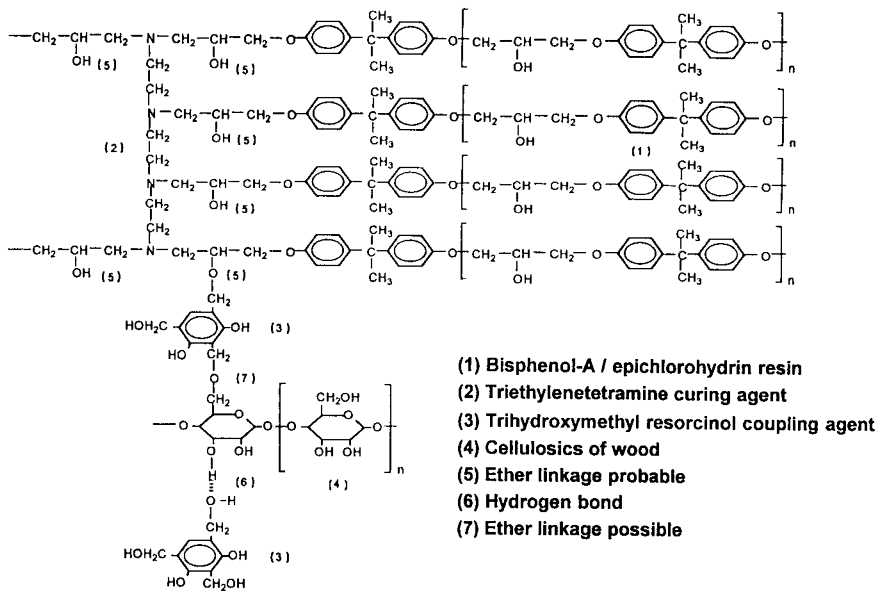


Figure 2. — Covalent and hydrogen bonding of the HMR coupling agent between diglycidylether of bisphenol-A (DGEBA) epoxy adhesive and cellulosics of wood.

wood cellulosics (position 7, **Fig. 2**). If conditions and sites are not conducive to covalent bonding, then hydrogen bonding is more likely to occur (position **6, Fig. 2**). When cell walls are thoroughly covered and penetrated by a multimolecular layer of highly reactive HMR of relatively small molecular size, opportunities abound for high-density hydrogen bonding with primary and secondary hydroxyls of wood lignocellulosics. The U.S. Department of Agriculture has been granted a patent for this invention (9).

The purpose of this report is to demonstrate that HMR effectively increases the structural durability of epoxy adhesive bonds to softwood and hardwood species commonly used to construct wood aircraft components. The effectiveness of adhesion was evaluated by measuring resistance to delamination, shear, and deformation of epoxy-bonded lumber joints in accordance with ASTM Specification D 2559 (3). This specification is used to qualify adhesives for structural glued-laminated timbers intended for wet-use exposures under industry standard ANSI/AITC A190.1-1992(1).

EXPERIMENTAL MATERIALS AND METHODS

HMR COUPLING AGENT

The HMR coupling agent was prepared by reacting formaldehyde with resorcinol in a 1.5 mole ratio at mildly alkaline conditions. The mixture was reacted for 4 hours at room temperature before application to the wood surfaces. Since the length of reaction time determines molecular-weight distribution and reactivity of HMR, the reaction time has a strong effect on adhesion. The following proportion of ingredients yields 5.0 percent dry solids in aqueous solution:

HMR ingredient	Parts by weight
Water, deionized	90.43
Resorcinol, crystalline	3.34
Formaldehyde, 37 percent	3.79
Sodium hydroxide, 3 molar	2.44
Total	100.00

Dodecyl sulfate sodium salt (0.5% by weight) was added to these mixtures at the end of the reaction time to aid wetting of the wood surfaces.

Water interferes with adhesion of epoxies to wood. For most effective adhesion, water must be evaporated from the HMR-primed wood surfaces before the adhesive is spread. Heat must not be used to accelerate evaporation of water, otherwise reaction of HMR will be accelerated and render the coupling agent useless.

TABLE 1. — Composition of FPL 16A, FPL 1A, and COM A adhesives.

Formulation and ingredients	Parts by weight
FPL 16A	
EPON 828 epoxy resin[a]	100.0
Blended lacquer thinner	18.0
Titanium dioxide	30.0
Diethylenetriamine hardener	13.0
FPL 1A	
D.E.R. 331 epoxy resin[b]	100.0
Benzyl alcohol	12.5
Hydrophobic fumed silica	2.5
Triethylenetetramine hardener	11.1
COM A	
Bisphenol-A epoxy resin	100.0
Blended hardeners	20.0

[a] Tradename of Shell Chemical Company.
[b] Tradename of The DOW Chemical Company.

ADHESIVES

All three epoxy adhesives were derived from reaction of bisphenol-A with epichlorohydrin to form the resin commonly called diglycidylether of bisphenol A. The remaining ingredients in each adhesive formulation varied **(Table 1)**. For proprietary reasons, the formulation of the commercial adhesive COM A cannot be shown. The FPL 16A formulation is also a commercial product (Star Technology, Inc.); however, it is essentially the same as the original FPL 16 formulation (7), with the exception that FPL 16A includes three more parts of lacquer thinner.

WOOD SPECIES

Two softwood species (Sitka spruce and Douglas-fir) and two hardwood species (yellow-poplar and yellow birch) were selected for adhesion tests. These species represent wood species and density ranges commonly used to construct structural components in wood aircraft. Generally, all pieces of wood in the test laminates were heartwood, straight-grain, free of defects, and flat-sawn. By sampling 30 pieces of lumber, estimates of the average and range of annual rings per inch (25.4 mm) were obtained for each species: Sitka spruce, 40 (16 to 86); Douglas-fir, 19 (8 to 42); yellow-poplar, 7 (4 to 16); and yellow birch, 24 (14 to 50).

The wood was conditioned at 22.8°C (73°F) and 50 percent relative humidity (RH) to approximately 9-l/2 percent equilibrium moisture content (EMC). Laminates were knife-planed to 1.9-cm (3/4 -in.) thickness 24 hours before bonding.

EXPERIMENTAL DESIGN

The experiment was designed to determine the effectiveness of the HMR coupling agent in enhancing the durability of adhesion of three formulations of epoxy adhesive in lumber laminates of four commonly used aircraft woods. The durability of bonding was evaluated by measuring delamination as lumber joints were subjected to a severe cyclic delamination test, shear strength and wood failure in a dry condition, and deformation under static loading, as required in ASTM Specification D 2559 (3). Only the most durable epoxy adhesive among the three adhesives was evaluated for deformation on the highest-density species (yellow birch).

Statistical experiments were conducted for delamination resistance, dry shear strength, and wood failure. Each experiment was a completely randomized model with a factorial arrangement (8) of 3 epoxy adhesives, 4 wood species, and 2 levels of surface priming (primed, unprimed), yielding 24 treatment combinations. Each treatment combination was replicated four times. For the delamination test, a replicate was a six-ply lumber laminate, from which three sections were cut. Delamination was measured from five bondlines on each end of the three sections in each laminate. Approximately 853 lineal cm (336 in.) of bondlines were measured for delamination for each treatment. For the dry shear strength and wood failure tests, a replicate was a two-ply lumber laminate. Five block-shear specimens were cut from each of 4 replicates, yielding 20 specimens for determining dry shear strength and wood failure for each treatment.

Parametric and nonparametric analyses of variance were conducted for each tested property. The Ryan-Einot-Gabriel-Welch multiple comparison *F* test was used to detect significant differences between treatment combinations (11).

PREPARATION OF SPECIMENS

The delamination specimens were 7.6-cm- (3-in. -) long cross sections cut from a six-ply lumber laminate (replicate). The laminate was prepared by bonding six pieces of lumber, each measuring 1.9 cm (3/4 in.) thick, 7.6 cm (3 in.) wide, and 30.5 cm (12 in.) long.

The shear strength and wood failure specimens were compression-loaded block-shear specimens with 19.4-cm^2 (3.0-in.^{2}) shear area, prepared and cut as described in ASTM Method D 905 (2). Block-shear specimens prepared from two-ply lumber laminates (replicate) were used for tests rather than stair-step shear specimens prepared from laminated beam sections. Each piece of lumber measured 1.9 cm (3/4 in.) thick, 6.4 cm (2-1/2 in.) wide, and 30.5 cm (12 in.) long.

The deformation specimens consisted of a series of 15 pairs of compression-loaded shear joints with each pair having shear areas measuring 5.1 by 1.3 cm (2.0 by 0.5 in.), for a total of 6.63 cm^2 (2.0 in.^{2}) in a pair. The complex specimen preparation and cutting procedures are described in ASTM Method D 3535 (4).

Two-, three-, and six-ply lumber laminates were prepared in the same

TABLE 2. — Delamination, shear strength, and wood failure of epoxy adhesives in lumber laminates of HMR-primed and unprimed softwoods and hardwoods.[a]

Adhesive	Species	Delamination		Shear strength		Wood failure	
		Unprimed	Primed	Unprimed	Primed	Unprimed	Primed
		(%)		(N/cm^2 (psi))		(%)	
FPL 1A	Sitka spruce	30.4	4.0	951 (1,379)	860 (1,247)	97	100
	Douglas-fir	49.6	3.7	851 (1,235)	753 (1,092)	95	97
	Yellow-poplar	10.6	0.0	1,131 (1,641)	1,048 (1,520)	93	97
	Yellow birch	27.7	0.7	1,047 (1,518)	1,488 (2,157)	12	86
FPL 16A	Sitka spruce	19.7	4.3	971 (1,408)	808 (1,172)	98	100
	Douglas-fir	59.3	4.2	811 (1,177)	879 (1,275)	99	100
	Yellow-poplar	30.9	0.5	1,091 (1,582)	1,113 (1,614)	100	100
	Yellow birch	96.0	10.8	1,581 (2,293)	1,693 (2,456)	86	77
COM A	Sitka spruce	72.8	6.0	956 (1,386)	854 (1,239)	94	100
	Douglas-fir	56.4	3.4	851 (1,234)	795 (1,154)	88	98
	Yellow-poplar	76.2	7.0	1,029 (1,492)	1,138 (1,651)	70	93
	Yellow birch	99.2	22.9	943 (1,368)	1,581 (2,293)	29	89

[a] Requirements of ASTM Specification D 2559:
Delamination: maximum 5 percent softwoods, 8 percent hardwoods
Wood failure: minimum 75 percent
Shear strength:

Sitka spruce	841 N/cm^2 (1,219 psi)
Douglas-fir	776 N/cm^2 (1,125 psi)
Yellow-poplar	869 N/cm^2 (1,261 psi)
Yellow birch	1,374 N/cm^2 (1,993 psi)

manner. If lumber surfaces were to be primed before bonding, 5 percent HMR solution was spread on both surfaces with a brush at approximately 0.15 kg/m^2 (0.03 pcf). The primed surfaces were dried 24 hours at 22.8°C (73°F) and 50 percent RH before bonding. Adhesive was spread with a roller on both bonding surfaces to total 0.35 kg/m^2 (0.07 pcf). Closed assembly time ranged from 60 minutes after the first bondline was spread to 50 minutes after the last bondline was spread. The initial pressure was about 69 kPa (10 psi), or enough to ensure a small amount of squeeze-out of adhesive full-length of every bondline. The lumber laminates were kept under pressure about 15 hours at room temperature. To ensure that all bondlines were cured to the same degree, the laminates were heated at 71°C (160°F) for 5 hours. The RH of the heating air was increased to equal the EMC of the wood so that bondlines would not be stressed by shrinkage of the wood while curing.

DELAMINATION TEST

Delamination specimens were subjected to the following three cycles of the delamination test in ASTM Specification D 2559 (3):

Cycle 1

1. Vacuum-soak in water at 84.4 kPa (25 in.-Hg) for 5 minutes.
2. Pressure-soak in water at 517 kPa (75 psi) for 1 hour.
3. Repeat events 1 and 2.
4. Dry at 65.5°C (150°F) for 21 to 22 hours.

Cycle 2

1. Steam at 100°C (212°F) for 1-1/2 hours.
2. Pressure-soak in water at 517 kPa (75 psi) for 40 minutes.
3. Repeat event 4 from Cycle 1.

Cycle 3

Repeat events in Cycle 1.

Immediately after the final cycle, delamination was measured along all end-grain surfaces to the nearest 0.25 mm (0.01 in.) with a machinist's scale under a stereomicroscope. This technique was more accurate than using the unaided eye and a 0.127-mm- (0.005 -in.-) thick feeler gauge, as recommended in the ASTM specification. Delamination was expressed as a percentage of total end-grain bondline length for each specimen. Statistical analyses were based on delamination measured after all three cycles were completed.

SHEAR STRENGTH AND WOOD FAILURE TESTS

Block-shear specimens were tested for dry shear strength and wood failure according to ASTM Method D 905 (2). At the time of testing, specimens were conditioned to 9-1/2 percent EMC, which is the same EMC used during specimen preparation and adhesive curing. Shear strength at failure was calculated as Newtons per square centimeter (pounds per square inch) based on 19.4-cm^2 (3.0-in.^{2}) shear area. Wood failure in the shear area was estimated to the nearest 5 percent.

DEFORMATION TESTS

Three-ply deformation specimens were loaded into compression-type deformation testers, then tested as described in ASTM Method D 3535 (4). Two specimens were statically loaded to 165 N/cm^2 (240 psi), then subjected to each of the following conditions for the required 7 days:

1. 71°C (160°F) at ambient RH.
2. 26.7°C (80°F) at 90 percent RH.

Deformation was measured after the 7-day loading period. However, the test was continued at each condition for an indefinite period.

RESULTS AND DISCUSSION

COMPLIANCE WITH ASTM SPECIFICATION

Resistance to delamination and shear of FPL 1A, FPL 16A, and COM A epoxy formulations in lumber laminates of two softwood and two hardwood species, with and without priming with HMR coupling agent, are shown in **Table 2.** The data show that none of the epoxy adhesives had sufficient delamination resistance to meet ASTM requirements **(Table 2)** on any species of wood that

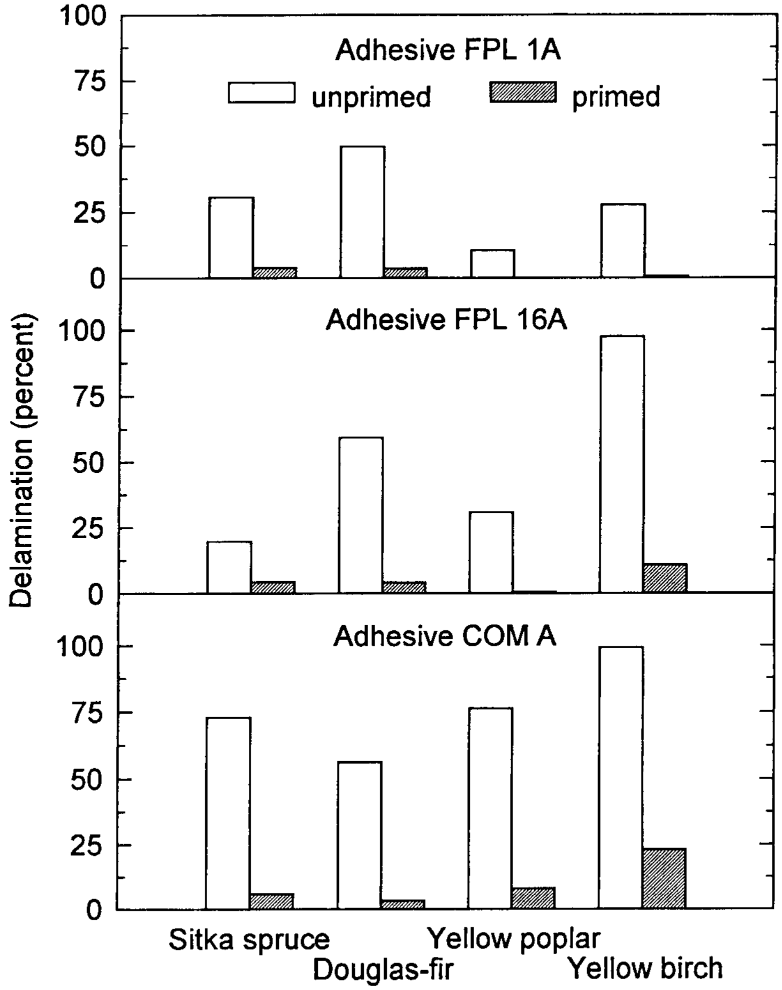


Figure 3. — Effects of epoxy adhesive, HMR priming, and wood species on delamination of lumber joints after cyclic delamination test.

was unprimed. However, priming with HMR allowed FPL 1A to exceed delamination requirements on all four species. FPL 16A met requirements on all species except yellow birch, where the 8 percent maximum was slightly exceeded, and COM A met requirements on Douglas-fir and yellow-poplar, barely exceeded 5 percent on Sitka spruce, and did not perform well on yellow birch. The data show that the HMR coupling agent was highly effective in helping all adhesives to meet the delamination requirements of ASTM Specification D 2559 (3).

Only FPL 16A exceeded dry shear strength and wood failure requirements on all unprimed wood species **(Table 2),** as well as on HMR-primed species, except as follows. Shear strength on primed Sitka spruce was 808 N/cm^2 (1,172 psi), just below the required 841 N/cm^2 (1,219 psi). However, wood failure was 100 percent, indicating that shear strength of the wood itself did not meet the requirement.

FPL 16A owes its popularity, particularly among builders of wood aircraft, to its ease of use, minimum clamping pressures, and ability to produce high, dry shear strength and wood failure on a wide variety of wood species. This adhesive is highly diluted with a blended lacquer thinner so that it penetrates and mechanically interlocks deeply into the structure of the wood, even a high-density species such as yellow birch. Yet, despite deep mechanical interlocking, FPL 16A delaminated severely when wood surfaces were not primed with HMR, as shown in **Table 2.**

FPL 1A was thixotropic and of much higher viscosity than FPL 16A, yet without priming, it exceeded the required dry shear strengths and wood failures on all species except yellow birch **(Table 2).** With HMR-primed yellow birch, however, FPL 1A met strength requirements. Even though shear strength on primed Douglas-fir was below the standard, the 97 percent wood failure indicates that the wood itself was not capable of supporting the required load.

The viscosity of COM A was between that of FPL 16A and FPL 1A. COM A met shear strength and wood failure requirements on all four species, but only when wood surfaces were primed **(Table 2).** Without priming, COM A met requirements on Sitka spruce and Douglas-fir, but failed to meet the minimum 75 percent wood failure on both hardwoods. Shear strength was also too low on the unprimed yellow birch.

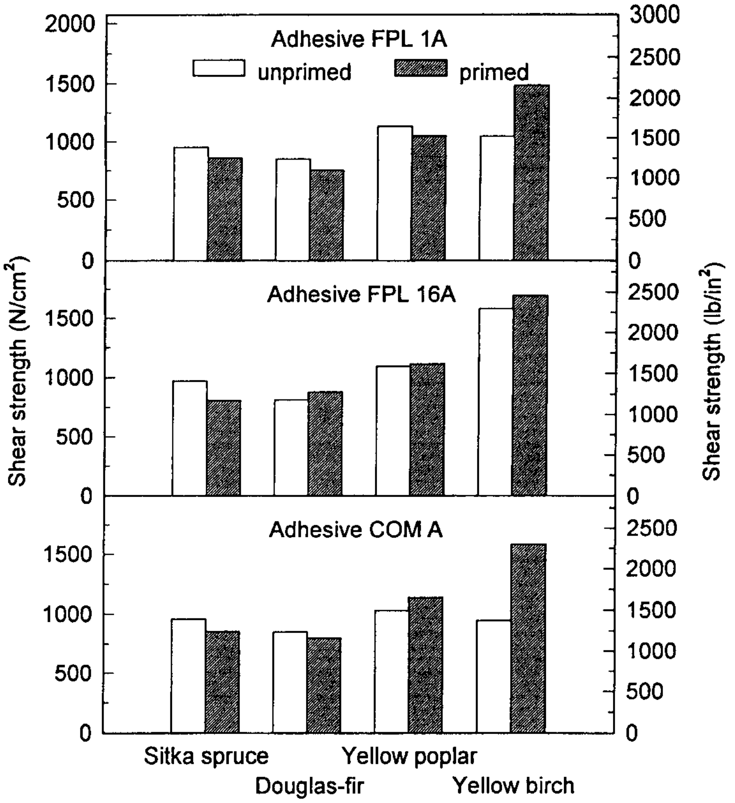


Figure 4. — Effects of epoxy adhesive, HMR priming, and wood species on shear strength of lumber joints in dry condition

RESISTANCE TO DELAMINATION

Priming wood surfaces with HMR produced statistically significant, dramatic, and consistent increases in resistance to delamination by all three adhesives on all four species of wood. Statistical data are not shown, but the effects from priming can be followed in general in the interaction plots in **Figure 3.**

The three adhesives produced significantly different resistance to delamination, depending on which wood species was tested and whether the surfaces were primed **(Fig.3).** On primed yellow birch, FPL 1A was significantly more free of delamination than was FPL 16A, but both adhesives performed significantly better than COMA. On unprimed yellow birch, FPL 1A still had better resistance to delamination than either FPL 16A or COMA. On primed yellow- poplar, FPL 1A and FPL 16A performed comparably, but both were significantly better than COM A. Similarly, on unprimed yellow-poplar, FPL 1A was significantly more delamination-resistant than either FPL 16A or COM A, but FPL 16A was significantly better than COMA.

Interestingly, but without explanation, there were no statistical differences in performance between any adhesives on primed or unprimed Douglas-fir, as shown in **Figure 3.** Even on the softwood Sitka spruce, differences in adhesive performance were not pronounced; no significant differences were seen between adhesives on the primed surfaces, although FPL 1A and FPL 16A performed significantly better than COMA on unprimed spruce.

In general, it appears that less delamination of FPL 1A occurred on the two hardwood species, whereas COMA performed better on the two softwood species. The performance of FPL 16A was mixed with respect to species, but there was clearly better delamination resistance on the three lower density woods than on the much higher density yellow birch.

SHEAR STRENGTH

Factors that strongly affected resistance to delamination had limited or no effect on adhesive shear strength. Priming of wood surfaces with HMR was a very important factor in reducing delamination of every adhesive on every wood species **(Fig. 3).** However, priming had no significant effect on adhesive shear strength on either yellow-poplar or Douglas-fir **(Fig. 4).** On Sitka spruce, priming even significantly decreased shear strength of all adhesives compared to that on unprimed surfaces. Wood failure was 100 percent for all three adhesives on primed Sitka spruce. On yellow birch, effects on shear strength were mixed as a result of interaction between adhesive and primer. When surfaces were primed, there were no differences in strength between adhesives. However, priming did significantly enhance shear strength of both FPL 1A and COM A. On unprimed yellow birch, FPL 16A had significantly greater strength than either FPL 1A or COM A. Compare the high strength of FPL 16A on unprimed yellow birch **(Fig. 4)** with the poor delamination resistance of the adhesive **(Fig. 3).** This is a good example of the dichotomy in performance of epoxy adhesives where shear strength in the dry condition is excellent on a high-density wood species, yet resistance to delamination under cyclic moisture conditions is poor. However, the HMR coupling agent dramatically enhanced the ability of the adhesives to resist cyclic aging.

WOOD FAILURE

Like shear strength, experimental factors had limited and mixed effects on wood failure percentages **(Fig. 5).** Priming was the only factor to significantly affect wood failure, increasing failure on Sitka spruce; the difference amounted to only 3 percent.

On primed Douglas-fir, FPL 16A had significantly higher wood failure than did FPL 1A and COM A, but only by 3 percent. On unprimed wood, all three adhesives produced overall wood failures that were statistically different, but the differences were of no practical consequence. COM A did have 10 percent greater wood failure on primed Douglas-fir, but results from priming were not significantly different from those obtained with the other adhesives.

Whether or not yellow-poplar surfaces were primed was inconsequential for FPL 1A and FPL 16A. However, COM A produced significantly lower wood failure on both primed and unprimed yellow-poplar. Although significant differences between COM A and FPL 1A were meaningless on primed wood, on unprimed wood the 23 percent difference was important.

The interacting effects of adhesive and surface priming were much more pronounced on yellow birch than on the other species. This was reflected in the sharply higher percentages of wood failure of FPL 1A and COM A on the primed wood, but insignificant differences between primed and unprimed wood with FPL 16A adhesive. The three adhesives performed comparably on the primed surfaces, but on the unprimed surfaces, FPL 16A performed much better than the other adhesives.

All three adhesives produced high levels of wood failure (above 75%) on all four wood species when surfaces were primed with HMR, although it was clear that the lowest percentages of wood failure occurred on the species with the highest density (yellow birch). When the surfaces were unprimed, only FPL 16A produced an acceptable level of wood failure (86%), even though all adhesives produced significantly lower percentages of wood failure on yellow birch than on the other species. The unique ability of FPL 16A to penetrate deeply and interlock into the structure of high-density yellow birch accounts for the high level of wood failure.

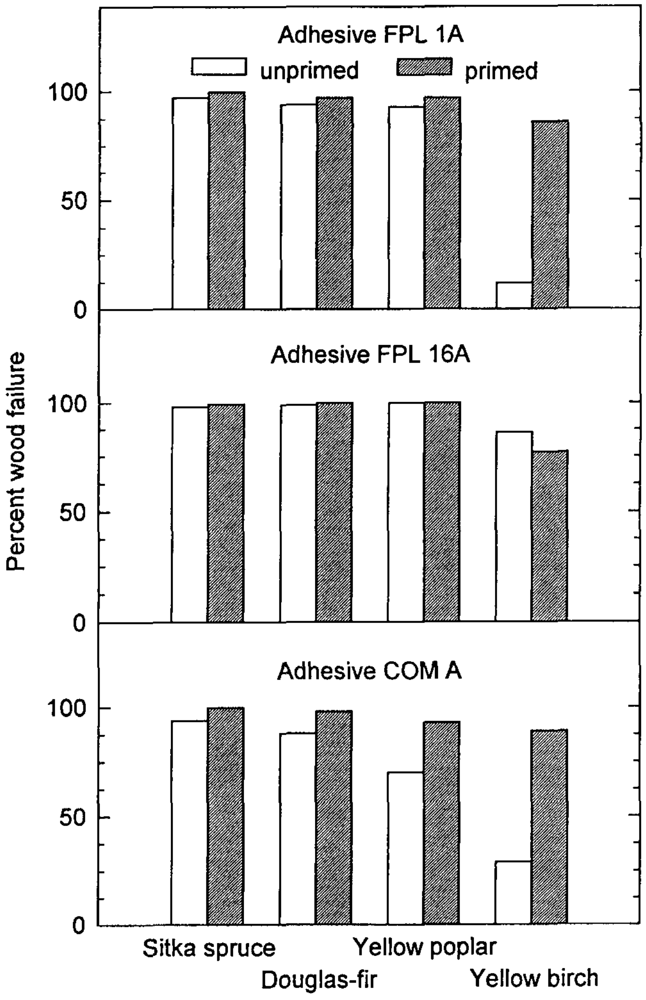


Figure 5. — Effects of epoxy adhesive, HMR priming, and wood species on wood failure after shear of lumber joints in dry condition.

RESISTANCE TO DEFORMATION

Lumber joints of yellow birch bonded with FPL 1 A epoxy adhesive have been under a static load of 166 N/cm^2(240 psi) at the required high RH and high dry heat aging conditions for 52 weeks without a deformation of bondlines. The required exposure time is 1 week, with deflection not to exceed 3.63 mm (0.139 in.).

CONCLUDING REMARKS

Hydroxymethylated resorcinol coupling agent greatly improved the structural durability of bonds made by three epoxy adhesive formulations (based on diglycidylether of bisphenol-A) on two softwood and two hardwood species commonly used to construct aircraft components. Formulation FPL 1A met requirements for resistance to delamination, shear, and deformation in HMR-primed lumber joints on all four species of wood, in accordance with ASTM Specification D 2559. Two commercial formulations met delamination and strength requirements on three woods of moderate density, but failed delamination tests on higher-density yellow birch. Without the HMR coupling agent, none of the epoxy adhesives had sufficient delamination resistance to meet ASTM requirements on any of the four species of wood.

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