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Adhesive Bond Performance of Historic Casein-Bonded Glued-Laminated Beams

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Abstract

An historic 1934 building on the campus of the U.S. Department of Agriculture, Forest Service, Forest Products Laboratory was deconstructed in 2010. The glued-laminated (glulam) beams from the building were preserved, creating a unique opportunity to test the casein adhesive bonding in the beams that had been in service for more than 75 years. The goal of the testing reported in this study was to extend the understanding of protein adhesives in wood products. In this study, we tested wet and dry strength of the bonds using compressive block shear tests. We also tested for delamination and looked at bond strength in bending over a range of temperatures in a dynamic mechanical analyzer. We found that the dry strength of the glulam adhesive tested in compressive block shear was very good. All the bonds failed in the wet strength testing. Because of the rigorous standards used for the delamination testing, all the beams failed the delamination testing, but surprisingly, about one-third of the beams showed only a small amount of delamination. From the dynamic mechanical analysis thermal scans, we saw little change in stiffness at higher temperatures, which suggests that the casein adhesive retained its elevated temperature strength even after 75 years.

Keywords: casein, adhesive, wood, laminated beam, block shear, delamination, dynamic mechanical analysis, thermal stability, glulam

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Cover photo: Workers installing plywood panels on the roof of Building 2 of the Forest Products Laboratory, Madison, WI, December 20, 1934. USDA Forest Service photo.

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INTRODUCTION

In 1934, the Forest Products Laboratory (FPL) in Madison, WI, erected the second building in the United States to use glued-laminated (glulam) beams (Rhude 1996, Teesdale 1935, Wilson 1939). The building was intended to serve as a full-scale demonstration for glulam beams, which was a new technology at the time, as well as an opportunity to evaluate the mechanical characteristics and long-term performance of glulam (Wilson 1939). Over the years, the building served as FPL service shops and research space for new forest products technologies such as veneer, plywood, strandboard, fiberboard, particleboard, wood–plastic composites, and acetylation. In the 1990s, a fire started in a fiber drying oven in the center of the structure, charring some of the beams. The fire was extinguished with water by the local fire department. The beams were inspected, and the building was put back into service. In 2010, after 76 years of service, the 1934 building was decommissioned and testing facilities were moved to a new, energy-efficient building. The beams from the 1934 building were recovered and saved for future testing.

In this study, beams from the 1934 building were mechanically tested by static and nondestructive testing as well as block shear testing similar to the testing reported in Rammer et al. (2014) and Teder and Wang (2013), which was done on a different set of beams. Our focus in this study was on the casein-based adhesive, which was in common use at the time the 1934 FPL building was built. Shortly after the end of World War II, construction of glulam beams shifted to phenol and resorcinol formaldehyde adhesives (Wilson and Cottingham 1952), which remain the standard adhesives for structural glulam beams today. In this study, we tested the historical casein adhesive bonds according to modern tests used for adhesive compressive shear strength values and beam delamination. Namely, ASTM D905 (ASTM 2021) and the vacuum-pressure soaking section of ASTM D2559 (ASTM 2017) were used as the test methods in this study. Because the building was experimental, the original construction included solid glulam arches and glulam box beam arches (now referred to as double I beams). Samples were taken from both solid and box beams as well as from one of the fire-charred solid beams.

Although casein adhesive is no longer used in glulam beams, it is still used as an adhesive for fire doors. As such, the thermal performance of the casein adhesive bond was explored. Dynamic mechanical analysis (DMA) has been used to study curing of phenol-formaldehyde or soy adhesives adsorbed to glass mats (Follensbee et al. 1993, O'Dell et al. 2013), cured or dry uncured adhesive films (Lopez-Suevos and Frazier 2005), and dry wood (Sun et al. 2007, Wolcott et al. 1990). In this study, DMA was used on the historical wood specimens, applying the same ramp rate as that used in Miyamoto et al. (2021). They performed high-temperature testing of various adhesives used to manufacture cross-laminated timber. This ramp rate represents a real-world rate of temperature rise at a point approximately 38 mm from the face of the beam or cross-laminated panel. In this way, we hope to extend the understanding of protein adhesives in wood products.

METHODS

The beams used in this study were from glulam arches manufactured by Unit Structures (Peshtigo, WI, USA). Information published at the time noted that the adhesive used was casein-based, but no further information has been found about the specific formulation. The wood for the lamina was southern yellow pine as identified by Wiedenhoeft.[1] We know from other sources that some adhesives at that time would have been based on casein protein isolated from skim milk principally combined with calcium oxide (burnt lime) or calcium hydroxide (slaked lime), both of which produce a highly alkaline adhesive (Salzberg 1977). Other potential additives from that time are mentioned in the literature, but we lack any of this information for these beams. Based on the size of the beams and the technology of the era, we assume that the adhesive would have cured at room temperature. The beams show no evidence of nail lamination; therefore, presumably, clamps were used during adhesive set. No information is available on other manufacturing aspects.

Several of the beams were constructed as box beam arches. The box beams were each constructed with a top and a bottom glulam arch, ripped to form two outer flanges and a center, then bonded with plywood between the center and the outer flanges. For further information on the beam construction and design values, see Wilson (1939). Figure 1 shows a schematic of the box beam construction and the locations where vacuum-pressure soak (VPS) specimens were cut. Figure 2 shows where VPS specimens were cut from solid glulam beams. Figure 3 shows the nominal VPS specimen size.

As discussed in the introduction, some of the solid glulam beams had been charred by fire. Others had been exposed to smoke or elevated temperature that was not high enough to char. Specimens for all testing in this study were cut from the straight portions of one glulam box beam (denoted as A), one

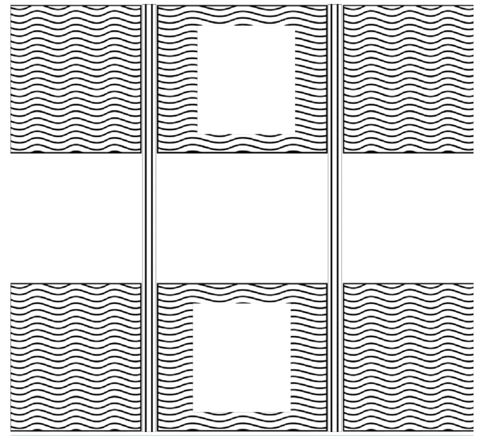

Figure 1.—Box beam showing the locations where specimens were cut for delamination testing.

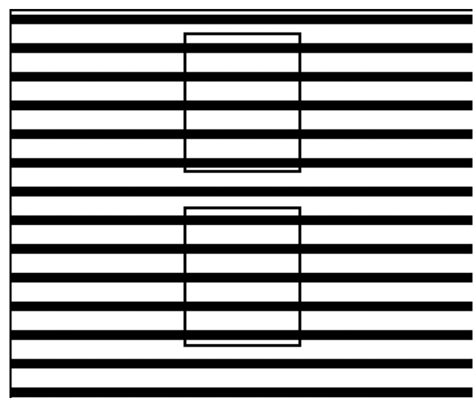

Figure 2.—Glulam beam showing locations where specimens were cut for delamination testing.

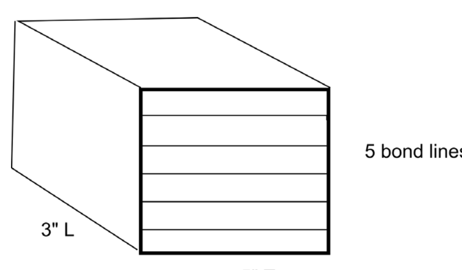

Figure 3.—Size of cut vacuum-pressure soak specimen that was tested for delamination (3 in. = 76.2 mm; 5 in. = 127 mm).

[1] Personal communication from A. Wiedenhoeft, research botanist, U.S. Department of Agriculture, Forest Service, Forest Products Laboratory, Center for Wood Anatomy Research (2023).

uncharred solid glulam beam (denoted as G), and one charred solid glulam beam (denoted as C). No specimens were cut from the knee of the arch, nor from the ends where the beams were bolted to the foundation or at the peak of the roof. There was no evidence of rot or water damage to any of the extracted sections.

Small Block Shear

From each beam section, smaller sections were excised with a bond line at the center and extending in the longitudinal direction of the lamina. These sections were planed to maintain equal thickness on either side of the bond line, then cut to form the offset according to a modified ASTM D905, forming small block shear specimens with a bond test area of 25 by 25 mm (Okkonen and River 1989). No specimens were cut where scarf joints existed within the lamina. Any specimens with voids, splits, or gaps were discarded. Specimens were conditioned at 21 °C and 50 percent relative humidity (RH) for a minimum of 2 weeks and then tested in compression shear in the fixture described in ASTM D905 at a rate of 5 mm/min. Specimen dimensions were measured just prior to the test, and the peak load (N) was recorded. Failure in this test occurs when either the bond or the wood separates and/or the load drops at least 5 percent.

Vacuum-Pressure Soak

From each beam section, smaller test specimens were cut parallel to the long axis of the beam (Fig. 1). Each test specimen contained five bond lines and had dimensions in accordance with ASTM D2559, although the box beam specimens were smaller in height than the solid beam specimens because of the difference in thickness of the lamina. For this test, five specimens each were cut from the top and the bottom of the beam, which normally are the most stressed portions of the beams. In the solid beams, specimens were cut at least one lamina away from the outer face, which kept outer surface char from affecting the charred specimens and kept the lamina the same for all specimens. The test followed the VPS cycle as in ASTM D2559.

Dynamic Mechanical Analysis

A bond line was cut with a bandsaw from each beam (A, G, and C), avoiding lamina with scarf joints and voids. This extracted portion was then supported in a channel in a thicker carrier board and planed to the target thickness, with equal thickness of wood on either side of the adhesive layer. Nonuniform thickness portions were cut away, then the specimens were cut to nominal 60 mm long by 12.5 mm wide by 2.7 mm thick where the longitudinal direction (L) was along the grain of the lamina, the width direction (W) was parallel to the bond lines, and the thickness direction (T) was perpendicular to the bond lines. Grain direction in the beams and the tested specimens varied from 90° to 45°.

The specimens were conditioned for 2 weeks at 32 °C and 15 percent RH to prevent changes in viscoelasticity during the test from drying. Each specimen was removed from the conditioning room and weighed immediately. Specimens were then measured in all three dimensions before being clamped in the dual cantilever fixture with a span between midpoints of 35 mm. Specimens were secured with a clamping torque of ~110 cN-m. The testing was conducted in dry air. Specimens were weighed after testing to verify that no change in moisture content had occurred.

One specimen was tested to determine the linear viscoelastic (LVE) region using a logarithmic amplitude sweep in the DMA (program set to 0.1 to 100 μm, 5 points per decade) at 30 °C. Testing terminated at 43 μm because the instrument reached its rated 18-N maximum force. From this, 1-μm amplitude was determined to be well within the LVE region and was used for subsequent tests.

The thermal test loading program was stress-controlled with an initial displacement of 1 μm and applied stress of 0.4 MPa. The goal of using the stress-controlled program was to perform uniform applied load because the method of specimen extraction by cutting and thickness planing meant that thickness varied among specimens and uniform displacement in strain control might have resulted in applied load varying among specimens. Each specimen was exposed to a 30-min isothermal segment at 60 °C to equilibrate specimen moisture to a low level as might occur in building materials in an attic, for example, without driving off bound water. The data in the isothermal segment were monitored to verify minimal drift in dynamic properties. Too much drift might indicate the need for further drying. After equilibration, the temperature was ramped from 60 to 260 °C at 4 °C/min. At the end of the test, the specimens were cooled ballistically and weighed after removal from the instrument.

RESULTS AND DISCUSSION

Adhesive Shear Failure

The results from the shear compression tests are provided in Table 1. The mean peak shear load for the specimens cut from casein-bonded glulam beams recovered after 75 years of service in a mixed-use building ranged from 1.84 to 2.21 MPa for the wet specimens and 10.07 to 12.54 MPa for the dry specimens. Figure 4 shows the differences among the specimens using the data from Table 1.

Table 1.—Mean and standard deviation (SD) shear stress of wet and dry casein-bonded glulam beams

Beam[a]	Wet/dry	Mean stress (MPa)	SD stress (MPa)
A	dry	10.07	1.16
A	wet	1.84	0.33
G	dry	12.54	2.20
G	wet	2.42	0.40
C	dry	10.99	3.94
C	wet	2.21	0.29

[a]A is the box beam; G is the full-depth, solid beam without charring; and C is the full-depth solid beam with bottom and side char.

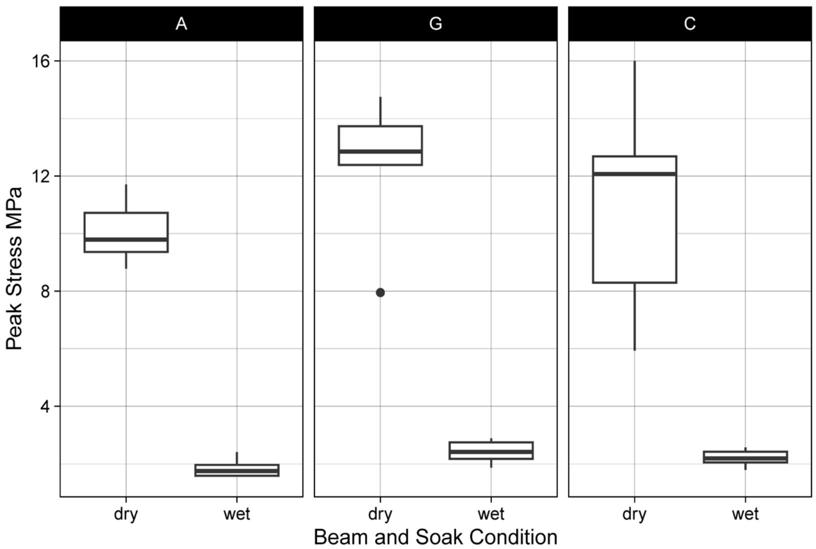

Figure 4.—ASTM D905 shear stress results for wet and dry specimens. A is the box beam; G is the full-depth, solid beam without charring; and C is the full-depth solid beam with bottom and side char. The box and whisker plots represent the distribution of the data. The lower and upper bounds of the box mark the 25th and 75th percentile of the data, the center bar is at the median of the data set, the whiskers extend to the minimum and maximum of the data set excluding outliers, and the circles represent outliers.

The difference between dry and wet groups of specimens is visually apparent in Figure 4 (numerical summary in Table 1). Differences observed among the mean shear stress values for each group (wet and dry) of A, G, and C beams were small and were within the observed data distribution of the dry and wet groups. Inspection of specimens after testing showed sliding cohesive failure of all wet specimens and 100 percent wood failure of dry specimens. In general, after testing, structural exterior adhesives exhibit considerable wood failure, and wet cohesive failure indicates an unsuitable adhesive (bond line failure is defined as failure within the adhesive layer; wood failure is failure that occurs more than two or three wood cells from the adhesive layer).

ASTM D905 is intended for advisory comparisons of adhesives and contains no required shear strength. ASTM D2559 requires a shear strength for southern yellow pine of 10.5 MPa at 8 percent equilibrium moisture content (EMC) and 8.6 MPa at 12 percent EMC. At the test conditions of 21 °C and 50 percent RH, wood is shown to have 9.2 percent EMC (Glass and Zelinka 2021). Interpolating between the given data points in ASTM D2559 gives a required shear strength of 9.9 MPa at 9.2 percent EMC. All the beam averages exceeded this threshold when dry but failed to meet this threshold when wet.

Casein adhesives in water-resistant formulations were considered suitable as structural adhesives for glulam beams in 1935. Raknes (1997) summarized the adhesive types in use in Norway in the 1960s and reported that casein adhesives met dry strength requirements of Norwegian Standard 3470 after 22 years of protected outdoor exposure and 30 years of interior use. Wet strength was not tested (Raknes 1997). Even if used as an interior adhesive, an adhesive that is sensitive to water is probably unsuitable for critical structural uses because of the accelerated creep that will occur during the inevitable cyclic humidity swings.

Vacuum-Pressure Soak

The VPS test metric in ASTM D2559 specifies that there be less than 1 percent delamination, determined as the sum of lengths of delaminations of all visible bond lines on both end faces of the specimen divided by the total length of bond lines. By that standard, all three beams tested here would fail. The results for the tested specimens are shown in Figure 5 (numerical summary in Table 2).

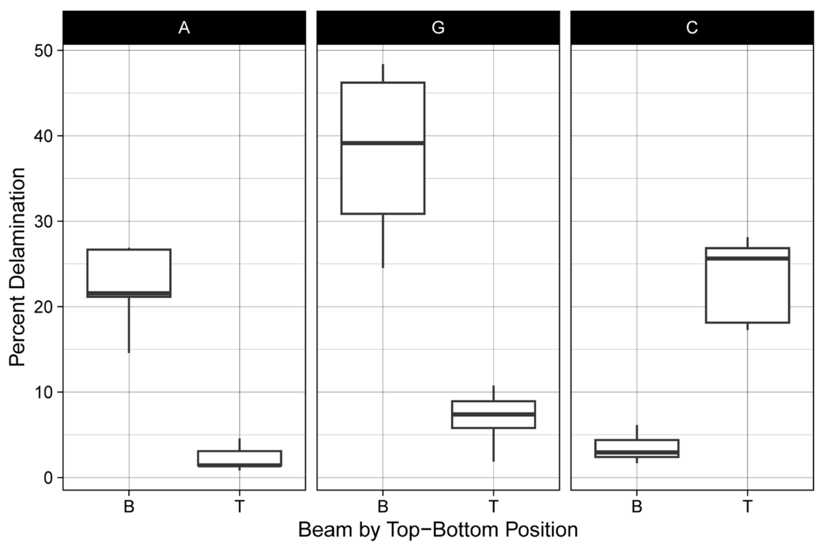

Figure 5.—Vacuum-pressure soak delamination. A is the box beam; G is the full-depth, solid beam without charring; and C is the full-depth, solid beam with bottom and side char. The box and whisker plots represent the distribution of the data. The lower and upper bounds of the box mark the 25th and 75th percentile of the data, the center bar is at the median of the data set, the whiskers extend to the minimum and maximum of the data set excluding outliers, and the circles represent outliers.

Table 2.—Beam delamination (mean and standard deviation [SD] as percentage of five bond lines

Beam[a]	Top/bottom	Mean delamination (%)	SD delamination (%)
A	B	22.17	5.04
A	T	2.27	1.54
G	B	37.82	10.12
G	T	6.95	3.37
C	B	3.51	1.77
C	T	23.20	5.11

[a]A is the box beam; G is the full-depth, solid beam without charring; and C is the full-depth solid beam with bottom and side char.

This standard for exterior structural adhesive is met by very few adhesives, most notably phenol-formaldehyde and phenol-resorcinol formaldehyde. These adhesives create a cross-linked network by diffusing several cells away from the glue line, which is believed to change the distribution of swelling stresses (Hunt et al. 2015). The fact that the casein adhesive in some of the tested specimens in this study demonstrated less than 10 percent delamination after three cycles of vacuum, soak, and oven-drying was surprising.

When the A (box) and G (solid) beams were compared, the distributions overlapped between beam types for the top specimens and the bottom specimens; however, for both beam groups A and G, delamination was greater for the bottom than for the top. For the C (charred) beam, the delamination was greater for the top than for the bottom. If the heat of the fire had weakened the adhesive, we would expect the bottom to be more susceptible to delamination. Therefore, this result might indicate improved bond properties caused by the heating. However, we might also expect overall reduction in delamination compared with the undamaged beams, which we did not see, so indicating that there may be other factors that caused the observed differences, such as variation in adhesive formulation, open or closed time, wood preparation, or bond curing. However, none of these variables in the beam construction were reported. Further studies would be needed for complete explanation.

Dynamic Mechanical Analysis

Minimal difference was observed between storage and loss modulus of all DMA specimens, and the trend of modulus loss with heating was identical (Fig. 6). These observed changes in storage modulus with increasing temperature are consistent with loss of wood strength caused by thermal decomposition during this test above 225 °C.

Because our specimens were aged (75 years old) and bonded with casein adhesive, we were interested in how the DMA of our specimens differed from that of other dry specimens. When we compared our results (Fig. 7) to the literature, wood with EMC below 1 percent (Sun and et al. 2007) had lower storage moduli in the temperature range 60 to 150 °C than did our specimens. Also, the tan delta of the specimens in Sun et al. (2007) was an order of magnitude lower in the same range than that of our specimens, indicating lower viscous dissipation of the former. Our specimens included both sapwood and heartwood, whereas Sun et al. used only sapwood. In addition, their specimens were dried over phosphorus pentoxide and treated at 150 °C for 10 min prior to DMA testing, whereas ours were conditioned at 32 °C and 15 percent RH and were equilibrated at 60 °C in dry air for 30 min prior to thermal scans. Our drying procedure is more likely to have retained some of the bound moisture that makes some wood polymers more mobile and contributes to viscous dissipation, which is measured by tan delta.

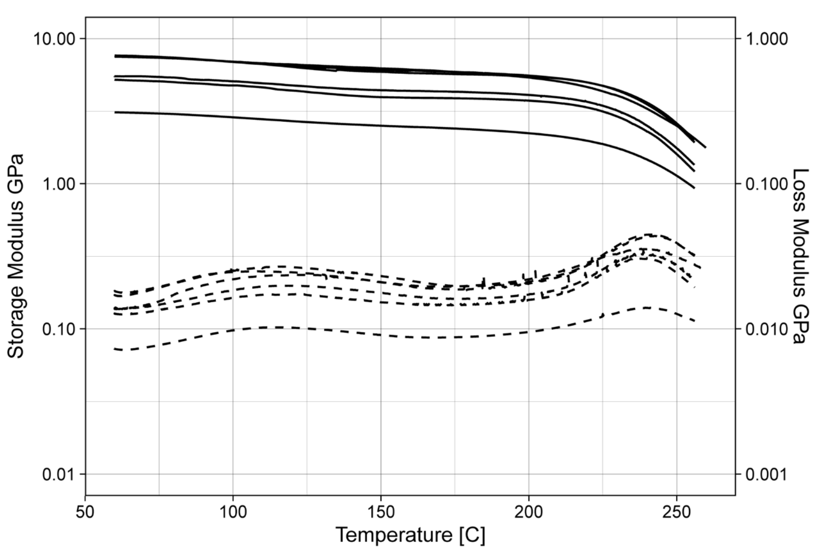

Figure 6.—Dynamic mechanical analysis storage (solid lines) and loss modulus (dashed lines) during temperature ramp from 60 to 260 °C at 4 °C/min for two specimens each from two uncharred beams and one charred beam (six specimens total). The observed curve displacements reflect variation in specimen morphology (e.g., cellular structure or specimen preparation).

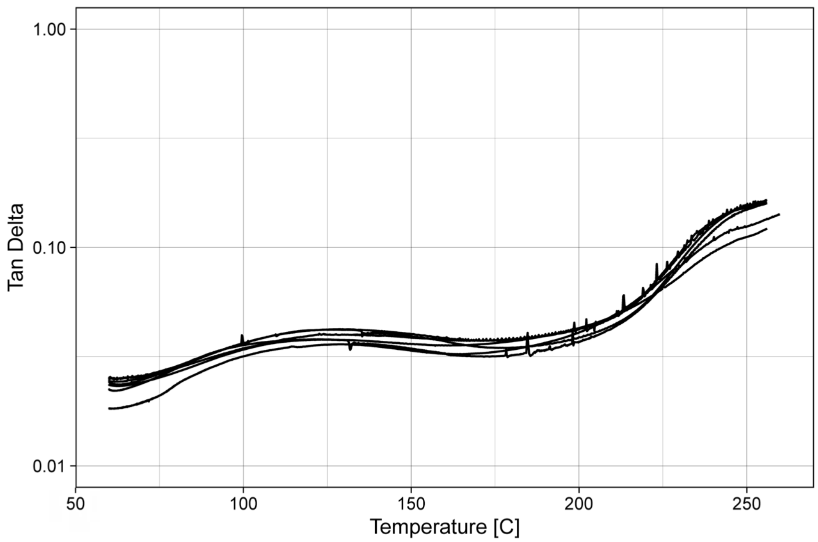

Figure 7.—Dynamic mechanical analysis temperature ramp showing tan delta (viscous dissipation) for two specimens each from two uncharred beams and one charred beam (six specimens total) heated from 60 to 260 °C at 4 °C/min.

Based on the fact that all of our specimens tested with ASTM D905 dry adhesive shear conducted at room temperature had 100 percent wood failure, we conclude that the elevated temperature performance observed in the DMA was dominated by the wood polymers. Because the wet ASTM D905 specimens had no wood failure and failed only at the adhesive, a useful future study would be to explore the viscoelastic properties and creep performance of casein bond lines across a range of practical relative humidities.

CONCLUSIONS

The casein adhesive of glulam beams that had been in service for 75 years retained good dry strength with 100 percent wood failure in compressive block shear tests (ASTM D905 small block specimens). Specimens tested in wet conditions all failed with sliding cohesive shear in the casein adhesive layer. All beams tested failed the rigorous delamination standard (less than 1 percent delamination) outlined in ASTM D2559, but one-third of the specimens showed less than 5 percent delamination, which is surprising because casein was not rated for exterior exposure. Sandwich specimens incorporating one bond line and the adjacent wood members tested in dynamic dual cantilever bending, heated in air at 4 °C/min from 60 to 260 °C, showed minor decline in storage modulus below 220 °C and increasing tan delta above that temperature. Tan delta did not rise above 0.10 until the temperature was above 230 °C. Future work on improving wet bonds would help decrease delamination of this adhesive and could lead to improved biobased structural adhesives.

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