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Small scale tests on the performance of adhesives used in cross laminated timber (CLT) at elevated temperatures

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ABSTRACT

This study is part of a research project examining fire risk in cross laminated timber (CLT) buildings. "Delamination", a major concern for CLT fire safety, can occur when the adhesive bondline fails in the panel before it chars which can lead to additional fuel for fire regrowth. Understanding the mechanical strength and stiffness of the adhesive bond under elevated temperature is one of the fundamental steps towards better understanding delamination. In this study, the material properties of wood and wood adhesive bonds are studied at various temperatures levels below the char temperature of wood (300 °C) to characterize the loss in strength and stiffness as a function of temperature. Four adhesives, three of which are commonly used in CLT production, were studied: two formulations made from polyurethane, one made from melamine formaldehyde, and one made from phenol-resorcinol formaldehyde. Tests were performed in a half-lap shear joint and compared against a specimen with identical geometry made from a solid piece of wood with no joint. The phenol-resorcinol formaldehyde joint had approximately the same shear strength as solid wood at all temperatures, whereas the other adhesives had noticeable reduction in strength at high temperatures. The method presented herein is unique among other CLT adhesive tests as it measures the performance of the adhesive under heat and load.

1. Introduction

Currently, there is interest in building tall wood buildings (> 26 m or 85 ft) utilizing mass timber materials. Mass timber refers to a class of massive engineered wood composites, including structural composite lumber (SCL), glue laminated timber (glulam), and cross laminated timber (CLT). While glulam and SCL have been around since the latter half of the twentieth century and are used in both residential and commercial construction applications, the development of CLT in the 1990's and its increased production in the 2010s have fueled a rebirth of timber construction and the development of tall wood buildings. CLT consists of layers of dimension lumber whose long axis is orientated 90° from its neighboring laminations. This results in massive panels that can be manufactured up to 500 mm thick and 18 m long and delivered to the jobsite with precut openings for doors and windows. The properties of CLT allow it to be used in both horizontal (floor assembly) and vertical (wall assembly) applications. CLT is featured prominently in recent mass timber buildings such as the eighteen story Brock Commons in Vancouver BC, the eight story Carbon 12 building in Portland OR, and the nine story Murray Grove building in London, UK.

One major perceived concern about the use of mass timber is fire performance. Despite perceived concerns, the fire resistance of wood members is well understood and has been studied since the 13th century according to Knowles and Pitt [1]. When exposed to fire, wood forms an insulating layer of char that develops between 280 °C and 300 °C. While char cannot carry a structural load, it has a low thermal conductivity and as a result, the uncharred cross-sectional area remains at a relatively low temperature and can continue to carry a load [2]. The char rate can be assumed to be roughly 0.6 mm min^{-1} (1.5 in. h^{-1}) in the absence of other data and adjustments to the char rate for species, grain orientation, wood moisture content and other variables have also been well characterized [2].

While charring of solid wood is generally understood, CLT compartment fire dynamics also depend upon the wood adhesives used in the CLT, given that some of the largest fire safety challenges of tall wood buildings are directly or indirectly related to adhesive performance in fire scenarios [3]. As the char front and thermal wave approach the bondline, delamination can happen if the thermal wave temperature weakens the adhesive. Because the thermal wave causes the temperatures at the bondline to increase before char is formed,

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delamination exposes two pre-heated, uncharred surfaces to the fire (the next layer of the CLT and the unburned back side of the delaminated layer). In a compartment fire, delamination can lead to a second flashover, where a fire that was decaying grows in heat and intensity with the addition of more fuel [4]. Delamination has been observed in compartment fire tests conducted at Carleton university [5], the Fire Protection Research Foundation (FPRF) tests carried out at NIST by the National Research Council of Canada [6], and the tests in support of the ICC code changes performed by the Forest Products Laboratory at the Alcohol Tobacco and Firearms laboratory [7]. In the Carleton and FPFR tests, the delamination led to a second flashover, however, in the ICC tests, a second flashover was not observed as the compartment temperature was below the ignition temperature of wood when the delamination occurred. Understanding adhesive performance under fire and its effect on CLT compartment fires is key to designing more fire resistant CLT structures and robust building codes.

Adhesive performance under high temperatures is less researched than char formation. Seminal work by Schaffer in the 1960's [8] examined how the char front moved through adhesive bondlines and measured the residual bond strength after exposing the bonds to flaming combustion. Shaffer's work was one of the first papers to report delamination for certain adhesives. Schaffer found that for melamine-formaldehyde (MF) and phenol-resorcinol-formaldehyde (PRF) adhesives, the char front moved through these adhesives as they did in solid wood. Furthermore, the bonds made with these adhesives remained intact even after the fire exposure and there was not a significant loss in residual capacity. Since engineered wood predominantly used these adhesives for many years, the fire performance of engineered wood composites was not a concern [9].

In the early 2000's several studies examined the performance of polyurethane adhesives at high temperatures and under fire loads. Part of this research stemmed from the fact that the wood industry discovered that wall assemblies constructed with finger jointed lumber, manufactured with polyurethane and poly vinyl acetate adhesives, may not achieve a 1-h fire resistance rating when tested per ASTM E119 [10]. This led to the development of a "heat resistant adhesive (HRA)" class of adhesives for finger jointed lumber. To achieve an HRA rating, finger jointed lumber made with the adhesive must pass a full-scale 1-h ASTM E119 wall test [10]. Adhesives used in CLT must also pass a large-scale fire test. The current PRG 320 product standard for CLT in North America requires that adhesives used must not delaminate in a large-scale test developed by Southwest Research Institute [11]. In that test, a compartment with non-combustible walls and a CLT ceiling is exposed to a fire load and ventilation to match the FPRF tests where delamination occurred. The adhesive passes if the panel does not delaminate. While these large-scale tests currently serve as benchmarks to enter the marketplace, they are expensive and pass-fail tests are not helpful in quantifying and modeling the adhesive performance.

Use of large-scale ASTM E119 wall tests or the PRG 320 ceiling test are intended to yield overall performance of the product. The overall performance of the product in these large tests is dominated by the charring of the wood and other test details. As a result, small differences in the adhesives are masked by the inherent variability in construction details of the assemblies and variability in the charring of wood.

An ideal test method to fill this knowledge gap would focus on quantifying strength loss in the adhesive below the char temperature of wood. A number of small-scale tests have been developed to evaluate the strength of adhesive bonds at high temperature; most of these tests evaluated polyurethane (PUR) and polyvinyl alcohol (PVA) adhesives. Clauβ et al. [12] tested single lap shear bonds in a universal test machine at temperature and found that polyurethane adhesives had 80–95% of the strength of PRF at 200 °C. Large differences were found between tests where the joint was heated and then tested to failure and tests where the samples were heated under a constant load. For instance, Frangi heated finger joints to a temperatures up to 140 °C and then tested them in tension [13]. He found that the tensile strength of the finger joints at 140 °C were 40–60% of the room temperature strength. However, Rammer et al. [14] placed a constant load on a finger joint and then heated them and examined the creep behavior and observed failures of the finger joints made with PURs and PVAs below 200 °C but did not observe any failures of finger joints made with PRF and MFs. These previous studies on adhesive performance under high temperatures were focused on understanding finger jointed lumber that resists vertical loads during a fire scenario. While they are a valuable starting point for CLT test methods, it is likely that CLT will require its own test method since delamination is different from the combined axial and second order flexural loading failures observed in finger jointed lumber.

In this paper we examine the strength, stiffness, and ductility of wood and wood adhesive bonds at elevated temperatures below the char temperature of wood (300 °C) to better understand delamination. Charred wood is assumed to have zero strength; the goal of this work is to characterize the degradation of the strength of wood and the reduction of the strength of wood adhesive bonds below the char temperature of the wood. The adhesive bonds are examined in a lap shear geometry and are compared against a specimen made of solid wood so the performance of the adhesive can be normalized against solid wood. This work is part of a larger program of research on understanding delamination and CLT fire performance. The results from this study (i.e. material properties) will be fed into a multiphysics finite element model to evaluate the risk of delamination. Furthermore, the results from the lap shear tests can be compared against full-scale compartment test data to evaluate its potential utility as a small-scale screening test for adhesives.

2. Materials and methods

Two different mechanical tests were carried out: tensile tests on solid wood and lap shear tests on wood bonded with adhesive. Both tests were carried out in an Instron 5589 (Norwood, MA) universal testing frame with a temperature controlled BEMCO FTU7.0-100x600 (Simi Valley, CA) testing oven that contains the specimen gripping and loading apparatus. The oven had access ports at the top and bottom such that the applied load could be transferred into the oven to the gripped specimen which was located inside the oven for the duration of the experiment. Prior to testing, all specimens were dried at 103 °C for a minimum of 24 h to reduce the effects of drying on testing time. To save testing time, samples were preheated in a secondary oven held at 100 °C and then transferred to the test apparatus where they were held at the target temperature until a thermocouple embedded mid-depth in the specimen had stabilized to the prescribed oven temperature (thermal equilibrium). By performing the final preheating in the test fixture, sample deformation prior to testing was minimized. Displacement was applied at a constant rate of 1 mm min^{-1} by the Instron test machine until failure of the specimen.

The deformation was measured using 2D digital image correlation (DIC) [15]. Deformed configurations of the specimen were monitored with a 5 MP Sting Ray F-504B camera (Allied Vision, Exton, PA) using a Schneider Optics (Bad Kreuznach, Germany) Xenoplan 1.4/23 mm lens. Images were acquired at 1 frame per second with a resolution of 2452 × 1200 pixels and a dynamic range of 12-bits. The images were post processed using 2D digital image correlation (DIC) with the Istra4D (Dantec Dynamics, Ulm, Germany) software package to obtain local displacements. A random speckle pattern was applied to the specimen surface with Rust-Oleum (Vernon Hills, IL) High Heat spray paint. The DIC system was calibrated following the Dantec Dynamics procedure with the calibration panel inside the oven at the plane of the specimen surface. The region of interest for the analysis was 1200 × 1000 pixels which corresponded to 23 × 19.5 mm on the specimen surface plane, with an image scale of 19 μm per pixel. A facet size of 40 pixels (0.76 mm) and a step size of 12 pixels (0.23 mm) were used as the correlation parameters, which resulted in a displacement resolution of 0.5 μm.

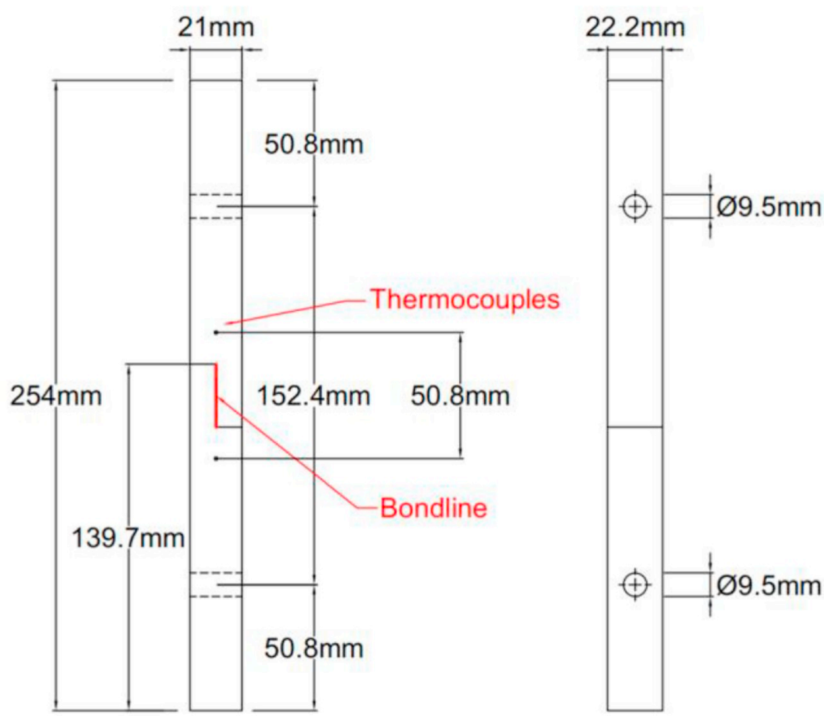
2.1. Tensile test

Tensile tests were carried out to obtain the elastic modulus and ultimate strength of wood at various temperatures. The specimen geometry conformed to the “parallel to grain” configuration found in ASTM D143 [16]. To ensure the specimen was at the target temperature during testing, thermocouples were installed at two depths near the grips. Three species groups were tested: southern yellow pine, Douglas fir, and spruce-pine-fir. The latter two groups are commonly used in CLT production in North America and southern pine is starting to enter the marketplace. Ten replicates of each species were tested at the following temperatures: 25 °C, 100 °C, 140 °C, 180 °C, 220 °C. For the tests conducted at 180 °C and below, a clip-on extensometer was used to measure displacements (the extensometer could not be used above 200 °C). Digital image correlation measurements were run concurrently with the extensometer allowing for verification of the DIC algorithms. Strains were computed using a virtual extensometer through DIC, with a gauge length of 25.4 mm, matching that of the extensometer.

2.2. Adhesive test

A half-lap joint was used to evaluate the adhesive bond performance at high temperatures. The geometry of the test specimen is given in Fig. 1 and only one wood species group, Douglas fir, was used for all adhesives tests. The specimens were glued within 3 h after the half lap joint was cut. The size of the bondline surface was 22.2 × 25.4 mm. Adhesives were hand applied to the manufacturers' recommended spread rate and a bondline pressure of 0.69 MPa was applied and held in the presses overnight at room temperature. Four adhesives were tested: a two part phenol resorcinol formaldehyde (PRF) adhesive, a two-part melamine-formaldehyde (MF) adhesive, and two different formulations of a 1-component polyurethane (PUR) adhesive, hereafter referred to as PUR-1 and PUR-2. Additionally, a solid Douglas fir sample with the same geometry but no adhesive bond was run in parallel with these groups so that the adhesive performance could be normalized to that of solid wood at each temperature.

Tests were run at the following temperatures: 100 °C, 140 °C, 180 °C, 220 °C, 260 °C; it is well known that structural engineered wood adhesives are designed to be stronger than solid wood at room temperature [17]. Ten replicates were tested for each adhesive formulation at each temperature. For these tests, the strain field around the bondline was examined using digital image correlation. A simplified approximation of the shear strain was calculated by investigating the displacements across the bondline. Axial displacements along transverse contours at each correlation point (located approximately every 0.23 mm along the length of the bondline) were smoothed using a uniform weight convolution with a kernel of 5 points. The local slope of the smoothed axial displacement contours was calculated using linear regression with a 5 point kernel. The 5 point kernel used in the convolution and regression resulted in an effective gauge length of approximately 1.14 mm. The shear strain at the bondline was determined by the maximum slope along the transverse contour, which was averaged over each effective gauge length. This process followed for each transverse contour along the bondline resulting in approximately 83



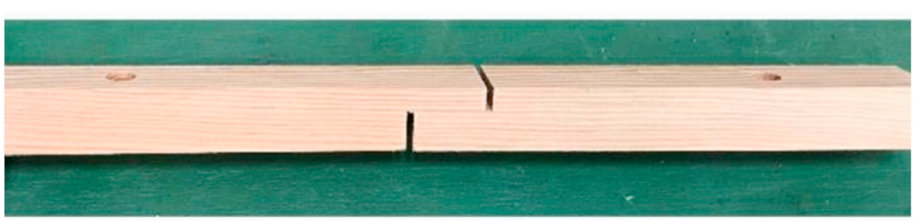


Fig. 1. Geometry of the shear test specimen (tensile test used geometry specified in ASTM D143). Bottom: photograph of a typical shear test specimen; for shear “solid wood” specimens, the two grooves were cut into a single piece of wood to give a sample with the same geometry as the lap shear specimens.

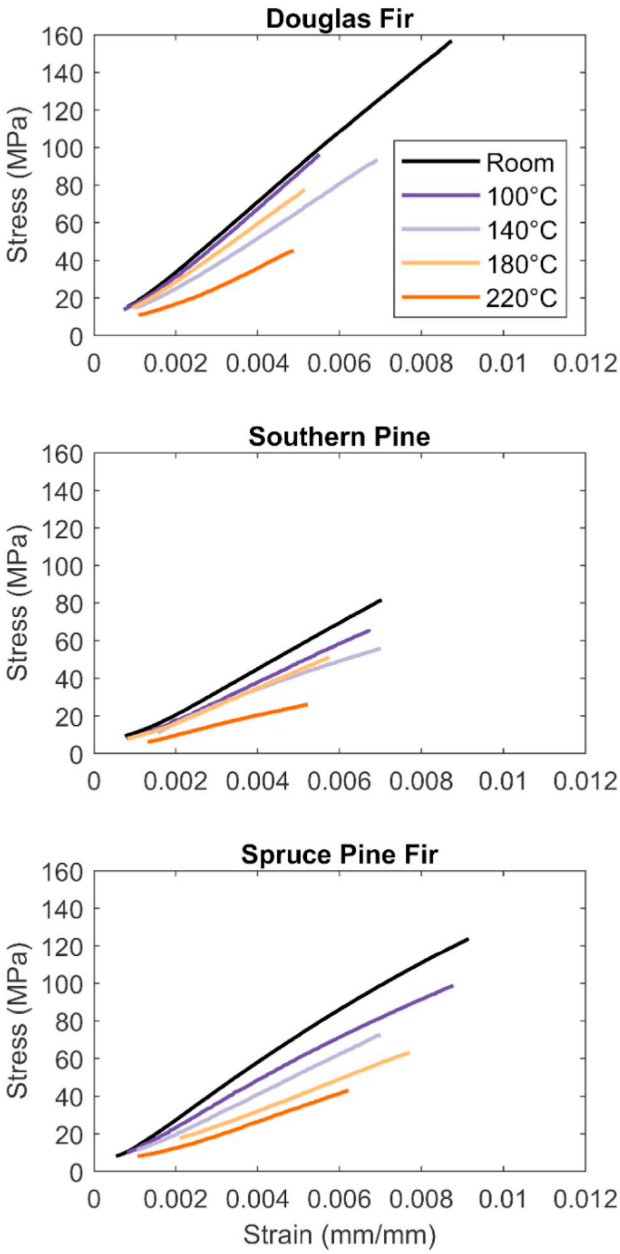


Fig. 2. Representative tensile stress strain curves at different temperatures for the three wood species tested. Representative curves were selected from the median value of the maximum load.

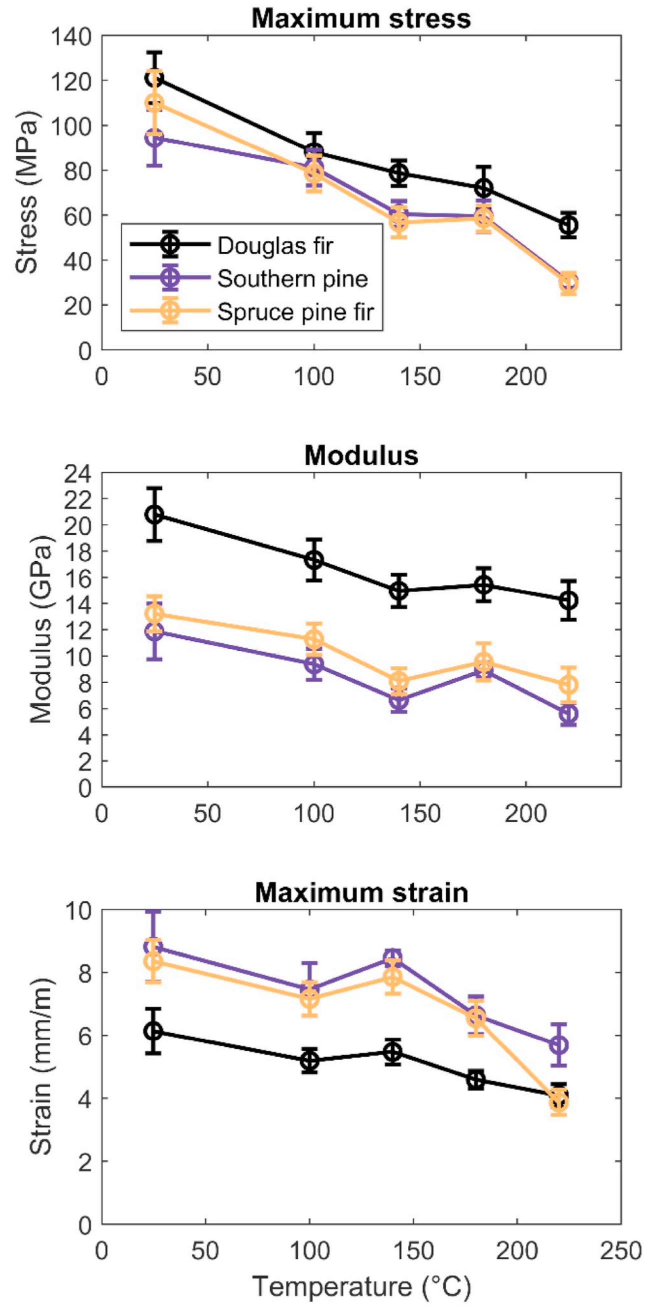


Fig. 3. Average (mean) values of the maximum tensile stress, longitudinal strain, and elastic modulus as a function of temperature for the wood specimens. The error bars represent one standard deviation.

contours per image. The final shear strain was computed as the average for all transverse contours for a given image. DIC was used to calculate stains in this initial research. However, it is envisioned that a simplified method for measuring shear strains will be used on further tests and verified with the full strain field data collected herein.

3. Results

3.1. Tension

Fig. 2 shows representative stress-strain curves for each of the wood species tested at different temperatures. The example curves were selected by choosing the median maximum load (i.e. failure strength) for each combination of wood species and temperature tested. The plotted strains were determined from a virtual extensometer using digital image correlation and any initial compliance of the system was corrected for by adjusting the linear portion of the curve so that it extrapolated through the origin. In general, the stress strain curves were linear-elastic until failure, with the SPF samples exhibiting the most ductility. For all wood species, as the temperature was increased, the maximum stress, modulus, and strain at failure all decreased, with the exception of data collected at 180 °C.

These trends can be observed more clearly in Fig. 3 which plot the modulus, maximum stress, and strain at failure. In Fig. 2, the mean is plotted along with the standard deviation (between 5 and 10 useable replicates) for each combination of wood species group and temperature. All three variables exhibit decreases from room temperature to their values at 220 °C. The maximum stress exhibits a linear decrease with increasing temperature and the ultimate tensile strength at 220 °C dropped to 46% of the room temperature strength for Douglas fir and to only 27% for spruce pine fir, indicating a large range of variation between species. Notably, the maximum stresses are lower at 140 °C than at 180 °C for the southern pine and spruce pine fir groups; the reason for this increase is unclear and further study is needed to understand these results. The modulus also decreases with increasing temperature, but the decrease with temperature is less severe; the mean elastic modulus of Douglas fir decreased by 32% from room temperature to 220 °C and 53% for southern pine. While the trend in modulus was a slight decrease with each increase in temperature, the observed moduli were higher at 180 °C than 140 °C for all wood species tested. For all species, the strain at failure was lower at 220 °C than room temperature. The strain decreased by as little as 50% for Douglas fir to over 100% for spruce pine fir. There was more observed variation in the mean strain than that of the stress or modulus, but general trend was that the strain at failure decreased with an increase in temperature.

One surprising feature of Fig. 3 is that the southern pine data exhibited the lowest strength and modulus of the three species groups examined. Based off of the small-clear test data for these species in the *Wood Handbook*, the southern pine should be the strongest of the species tested and have a stiffness comparable to that of Douglas fir [18]. We believe the observed differences in this study are a result of the grain pattern in the small-clear samples used in this study. Fig. 4 shows representative samples from each of the wood species tested. Even though the southern pine was select structural grade, it had larger growth rings than that of the Douglas fir and spruce pine fir specimens. This resulted in some of the pieces having nearly all early wood in the necked-down region of the ASTM D143 samples. The result is that the southern pine properties reported in Figs. 2 and 3 likely represent lower bounds on the strength of clear southern pine at high temperatures.

3.2. Shear

Fig. 5 shows representative shear stress-strain curves for each of the bonded assemblies at four of the five temperatures tested. As the temperature is increased, the maximum shear stress decreased while the deformation increased. For the PRF and PUR assemblies, the amount of deformation increased appreciably at higher temperatures. This trend in the maximum shear stress can be more easily seen in Fig. 6 and Fig. 7 which plot the maximum shear stress at failure and percent wood failure as a functions of temperature.

For the solid wood and all adhesive systems, the shear strength decreased as the temperature increased. A notable decrease in the strength occurs between 220 °C and 260 °C. From Fig. 6 it can be seen that the PRF adhesive had a similar strength to solid wood over all temperatures. The largest differences in shear strength between most of the adhesive formulations and the solid wood could be observed at 260 °C. Comparing mean values at 260 °C, the PUR1 formulation had only 30% of the strength of the solid wood control; the PUR2 had over 60% and the MF had 85% of the strength of solid wood at 260 °C.

It is also interesting to note differences in the failure surfaces as summarized in Fig. 7. The MF and PRF adhesives maintained a high degree of wood failure throughout all temperatures. However, at 260 °C, both of the polyurethane adhesives exhibited nearly a 100% failure within the adhesive layer with both surfaces appearing coated with adhesive when examined with the naked eye. It is also worth noting that some of the peculiarities in the trends in Fig. 6, namely MF & PUR2 at 140 °C and PUR1 at 180 °C correspond with low average percent wood failures and high standard deviations in the percent wood failures in Fig. 7. In these three groups there were several samples with low strengths and low percent wood failures.

4. Discussion

The goal of this research was to understand the change in mechanical properties of engineered wood materials and adhesives at elevated temperatures. Both tensile and shear stress-strain curves of the wood material and shear stress-strain curves for wood-adhesive bondline were collected so that they could be utilized in a multiphysics finite element model to predict the structural performance of CLT subjected to fire conditions. The ultimate goal of such a model is to better understand when delamination will occur and ultimately develop an understanding of the risk of delamination (i.e. fragility curves) for different engineered wood adhesives.

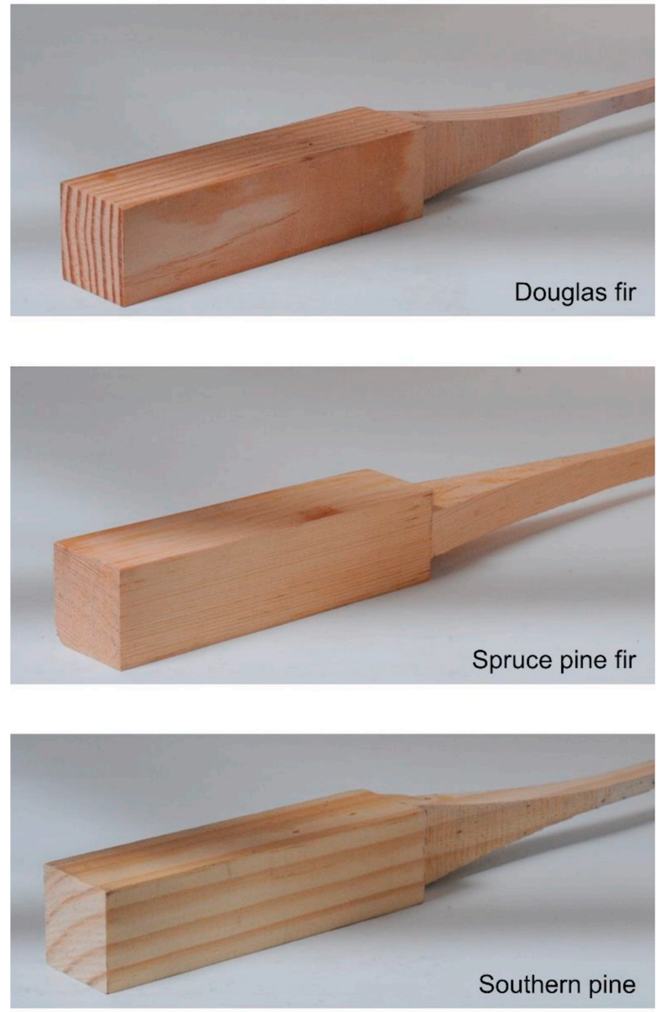


Fig. 4. Representative photographs of the tension specimens showing the grain pattern. The southern pine specimens had wide growth rings which resulted in the some of the samples being comprised predominately of earlywood in the gauge length.

In addition to being useful as inputs into a multiphysics model, the results of this study are also valuable in understanding the fire resistance of engineered wood products. The tensile data gives further insight into the reduction of wood strength under fire scenarios. For standard fire resistance calculations, charred wood at 300 °C is assigned zero-strength [2]. To account for the strength loss prior to the char level, several different approaches can be used, including adding a "zero-strength layer" to the char depth, typically a 20% increase in the charring rate [2]. The data displayed in Fig. 1 can be used to better understand these strength reductions with more detail and can be used to better predict the residual load carrying capacity of wood members.

The results of the adhesive performance at elevated temperatures are also important for the development of new adhesive formulations and small-scale qualification tests. The current PRG-320 standard [11] requires a test of a full-scale CLT compartment which is not only cost prohibitive but also has uncertainties in many other manufacturing details aside from the effect of the adhesive itself. A more cost-effective

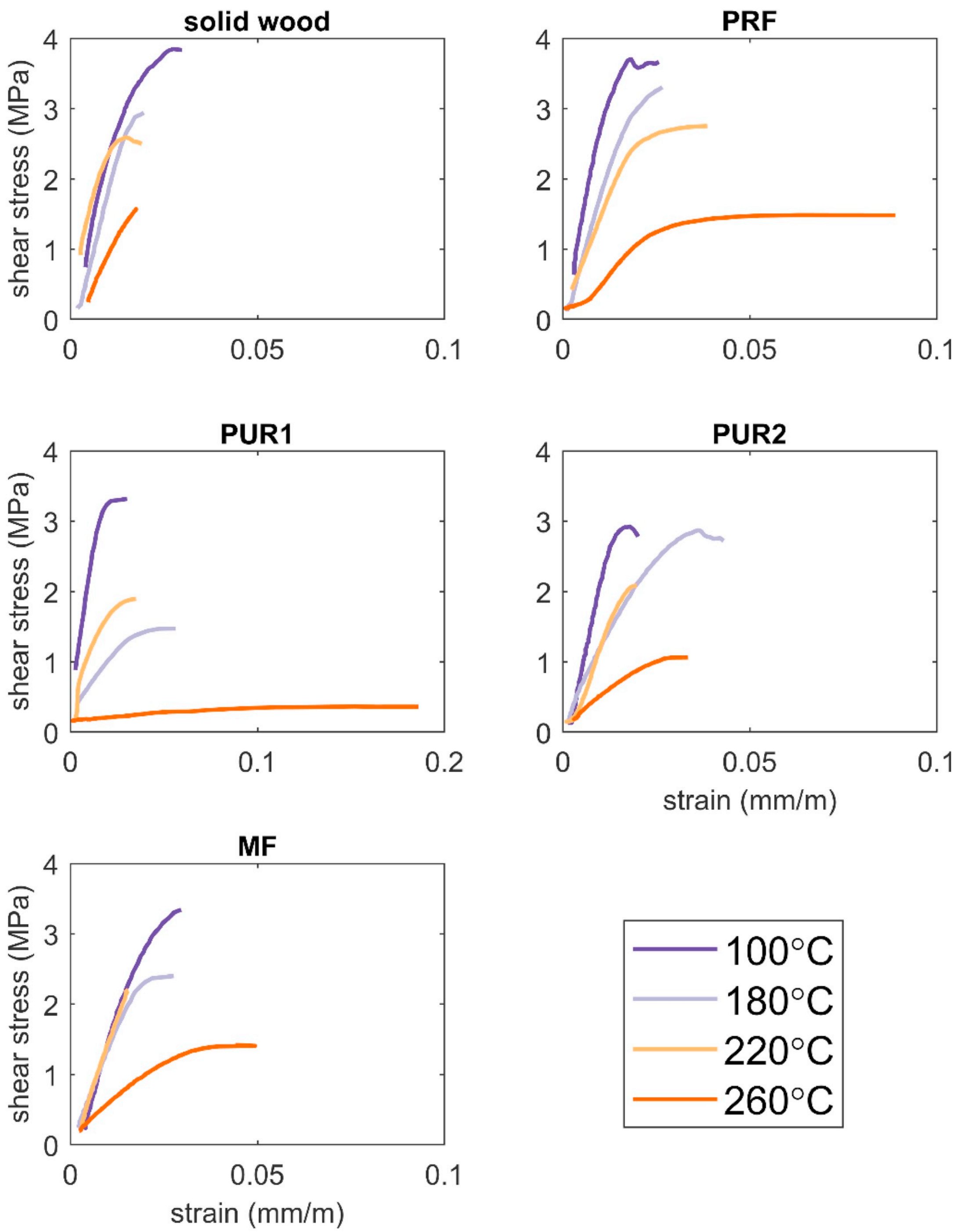


Fig. 5. Representative shear stress strain curves for the adhesive tests at four temperatures on Douglas fir. Note the different x-axis for the PUR1 adhesive.

testing method is needed to better facilitate the introduction of new adhesive formulations to the marketplace. Several researchers (discussed below) have been working on developing small-scale tests that can be used as a potential qualification test for new adhesives for CLT.

Most of the proposed small-scale methods involve looking at the char rates of a 5-ply CLT exposed in an intermediate-scale furnace (ca. 1 m^2). In the method developed at Eidgenössische Technische Hochschule Zürich (ETHZ), the panel is attached to a load cell and the mass loss is measured as a function of time during a standard fire exposure [19]. Finally, the Research Institutes of Sweden (RI.SE) performed tests using a non-standard fire exposure that exactly matched the temperature and oxygen content of large-scale fire tests on delaminating CLT [20]. Although there are differences in the exposure and evaluation criteria, the methods are similar in that they evaluate the adhesive performance based on how fast char forms on the wood, with the assumption that delamination will result in a higher average char rate as measured at the end of the test. While the char rate is undoubtedly higher in panels with delamination, the method does not directly test the adhesive performance, but rather the entire panel performance. For example in the RI.SE tests, it took between 45 and 50 min for the bondline temperature to rise above 250 °C [20]. Given that all adhesives tested in this study performed well until this temperature, the method is not ideally suited for detecting small differences between adhesives, and is best for determining whether the adhesive loses strength well below the char temperature of wood. Moreover, none of these methods apply a mechanical load to the CLT. Loading the panel will result in an

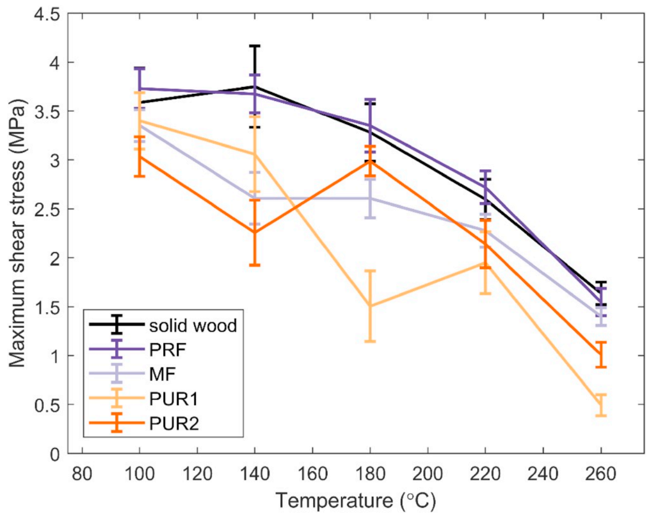


Fig. 6. Maximum shear stress as a function of temperature for the adhesive systems examined. Error bars represent the standard error.

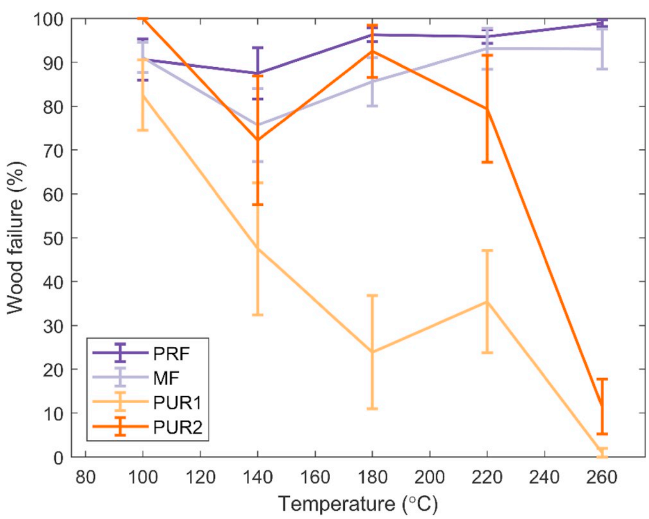


Fig. 7. Percent wood failure as a function of temperature for the adhesive systems examined. Error bars represent the standard error.

increased shear stress at the lowest bondline which will presumably cause failure earlier than in an unloaded situation. Furthermore, CLT panel tests are more costly and complicated than a test focused on a single adhesive bondline that has similar physics and scalable mechanics to the bonded interfaces in CLT below the char temperature.

The lap shear test presented herein allowed for the strength of the wood-adhesive bond to be determined as a function of temperature up to the char temperature of the wood. By comparing them against the strength of solid wood, the independent relative strength of the adhesive could be determined. It is generally understood that the wood-adhesive bond should be stronger than solid wood at room temperature. However, only one of the four adhesives tested in this study could retain that capacity throughout the range of temperatures. A small-scale adhesive qualification test could easily specify that the wood adhesive bond needs to retain a certain percentage of the strength of solid wood at 260 °C or another temperature approaching the char temperature of wood. Selecting the appropriate percentage could be developed from correlating results from formulations that have passed and failed the large-scale PRG 320 test.

An alternative, small-scale qualification method could involve a similar test geometry but instead of loading to failure at a certain temperature, the specimen could be loaded to the shear stress under the design load of a CLT floor system, and then subsequently heated to fire conditions until failure. This modification to the test presented herein is in some ways more similar to a delamination event where the temperature is increased under constant load until failure. However, the selection of a temperature ramp rate and thermocouple methodology could pose challenges.

Small-scale methods have the following advantages over panel level tests: (1) they mitigate full-scale nonlinear interactions which allow for small differences in the adhesive performance to be quantified, (2) they can be conducted in any laboratory and do not require a fire laboratory, and (3) they test the adhesive performance under load. While a panel level test may ultimately be required for qualification under PRG-320, the small-scale tests presented herein do provide valuable, complimentary information to the panel level test which may be of value not just to regulators, but also in the development of new adhesive formulations.

5. Conclusions

The material properties of solid wood and wood-adhesive bondlines were studied to better understand the risk of delamination with different adhesive formulations. The results provide material properties as a function of temperature that can be used for a multiphysics model of delamination.

For the solid wood, the ultimate strength and modulus decreased linearly with temperature across all species. The southern pine data had a low tensile strength relative to previously published values. This was attributed to the width of the growth rings and the fact that many samples contained mostly earlywood in the gauge length. While higher values may have been obtained with quarter sawn samples with a tight growth ring, the tensile values likely are representative of the clear properties of commercially available southern pine.

For the shear tests, a noteworthy decrease in strength was observed at 260 °C for both the solid wood and the adhesives. However, the PUR1 adhesive exhibited the greatest decrease in strength at 260 °C. Over all temperatures the PRF assembly had similar strength properties to solid wood.

While a number of small-scale tests may be used to test the adhesive performance of CLT in fire scenarios, it is important that the test capture both the mechanical and temperature loads. A unique aspect of this study when compared against other small scale tests proposed for CLT adhesives is that it explicitly measured the mechanical strength of the adhesive bond at high temperatures. By testing a single adhesive bond under shear, it is possible to measure the adhesive performance in the absence of wood charring.

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