EFFECT OF TIE-LAYER ON THE BOND STRENGTH BETWEEN THERMOPLASTIC AND BORATE-TREATED WOOD SUBSTRATE

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Abstract. A challenge in using wood or wood composites for exterior applications is durability. Borate-treated wood substrates are durable if leaching of the chemical over time can be reduced to acceptable levels. The goal of this project was to encapsulate borate-treated structural wood and wood-based composites with thermoplastic to extend their durability. In this study, the efficacy of two tie-layers (maleic anhydride modified high-density polyethylene and styrene–butadiene polymer) in bonding high-density polyethylene (HDPE) to treated wood substrate was examined together with determining the ideal hot-press parameters necessary to achieve a good bond. Boric oxide treated Douglas-fir and southern pine Parallam® and untreated Douglas-fir solid wood were the substrates investigated. The optimum processing parameters were 180°C platen temperature, 1035 kPa press pressure, and 300 s press time. Bond strength was determined by conducting a 90° peel test and a block-shear test. Durability of the thermoplastic barrier layer was evaluated by subjecting specimens to an accelerated aging test and reevaluating the bond strength. Maleic anhydride-modified HDPE tie-layer yielded improved bond strength that was durable, especially when bonded to a treated southern pine substrate.

Keywords: Thermoplastic, tie-layer, wood composite, peel strength, preservative treatment, Parallam, Douglas-fir, southern pine.

INTRODUCTION

With increasing environmental concerns and regulations, there is a drive toward environmentally benign methods of protecting wood against fungal decay and moisture. In response to this need, wood–plastic composites (WPCs) have evolved and are gaining a market share for exterior applications. WPCs are durable in part from the polymer matrix, which restricts moisture uptake and inhibits fungal decay and insect attack (Morrell et al 2006). Another study (Morris and Cooper 1998) showed that WPCs can also be attacked by wood-rotting and staining fungi in service if the wood component is not protected with a preservative. Therefore, an effective method of protecting wood composites should improve moisture resistance as well as protect the wood component from decay fungi. Most applications of WPC lumber are for residential decking and railing systems.

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There are a wide range of engineered wood composites, including laminated veneer lumber, parallel strand lumber, and oriented strand lumber, that are designed for more demanding exterior applications such as the substructure of a deck or balcony; however, in some cases, these materials need to be treated with preservative chemicals. A relatively benign preservative chemical treatment that can be used on structural wood composites, without the corrosion problems of copper-rich formulations, is a borate-based compound. The drawback to using borate-based compounds is that they do not chemically bond with wood and can leach over time, especially water-soluble compounds such as borax–boric acid mixture (Levi 1973). A technical solution to this leaching problem would have tremendous practical value.

One method to protect structural wood composites from moisture as well as contain the preservative chemicals and prevent leaching could potentially be encapsulation of wood substrate with a thermoplastic polymer such as high-density polyethylene (HDPE). Several researchers (Gardner et al 1994; Davalos et al 2000; Tascioglu et al 2003) have studied shear strength and durability of fiber-reinforced polymer composites and wood-bonded interfaces for applications in structural glued laminated beams. Thermoplastic–wood interfaces were investigated by Gacitua (2008) and Gacitua and Wolcott (2009) and the anatomical features of wood species had significant influences on the performance of the interface bond. An interaction between HDPE and wood substrates is very complex and often poor. HDPE is hydrophobic and nonreactive, whereas wood is hydrophilic resulting in two materials that have poor interaction at the interface (Liu et al 1998). There is clearly a need for some method to improve the interfacial bond such as a tie-layer between HDPE and wood or perhaps a chemical pretreatment of the wood before bonding.

Dai et al (2004) used two types of thin tie-layers (polyolefin-based and modified HDPE) to bond E-glass/polypropylene (PP) yarns to an oak substrate. Examining the failure mode of the composite in lap-shear specimens, it was observed that a cohesive failure of the composite was common in areas that were weakened by voids. The number of voids could be reduced by increasing the consolidation time and the press pressure. A higher heating temperature and pressure were found to improve the bonding strength.

Kumar and Ramani (2000) utilized a polyethylene/elastomer blend-based tie-layer film to bond a unidirectional continuous glass fiber-reinforced polypropylene (UCGPP) composite to an oak substrate. Effects of changing moisture on composite modulus using dynamic mechanical analysis were investigated. It was found that under desorption/sorption cyclic conditions, the wood–UCGPP composite underwent significant modulus changes because of the differential shrinkage/swelling of the oak compared with the UCGPP composite. However, the tie-layer did increase the storage modulus of the wood–UCGPP composite compared with the neat wood.

Surface modification of the wood substrate instead of a tie-layer was explored by Mahlberg et al (2001) to increase the adhesion between wood substrates and thermoplastics. Mahlberg used succinic and phthalic anhydride as modifiers to laminate birch veneers with PP. A 90° peel test yielded values ranging 0.29-0.64 kN/m for phthalic anhydride-treated specimens and 0.38-0.63 kN/m for succinic anhydride-treated specimens. Kolosik et al (1993) used maleated PP wax as a modifier to laminate birch and aspen veneers with PP. The untreated specimens had a peel strength of 0.08-0.1 kN/m; treated specimens had varying degrees of treatment, but had peel strengths of 0.04-0.1 kN/m.

In this study, the use of a tie-layer between HDPE and wood-based substrates was examined to strengthen the bond and provide a barrier to moisture infiltration. Two commercially available tie-layers were used to bond a thermoplastic resin to Parallam® as well as a solid wood substrate using hot-pressing. Hot-press parameters affecting the bonding between thermoplastic and wood substrates, bond strength, and bond durability were investigated. Specific objectives addressed are the influence of temperature, pres-

sure, and press time on the bond strength of the thermoplastic barrier layer to the wood substrates and bond performance at predetermined ideal hot-press conditions.

MATERIALS AND METHODS

For determining ideal hot-press conditions, only pressure-treated Douglas-fir Parallam® was used for the substrate because of the refractory nature of Douglas-fir. However, for the purposes of comparing the differences in bond strength and durability between thermoplastic and wood substrates, Douglas-fir and southern pine Parallam® as well as Douglas-fir solid lumber were used. All materials were chemically pressure-treated with boric oxide (B_2O_3) by Pacific Wood Preserving of Oregon at a retention level of 4.5 kg/m^3. The thermoplastic used as a barrier layer was HDPE, and the two types of tie-layers evaluated were a maleic anhydride-modified HDPE tie-layer (TL-A) and a styrene–butadiene polymer tie-layer (TL-B). The plastics were obtained in either a powder or pellet form and extruded into a film using a Leistritz 18-mm corotating extruder. The approximate thickness of the films was about 0.5 mm. Properties of all thermoplastic polymer and tie-layer copolymers are summarized in Table 1.

Thermal transitions of the thermoplastic barrier layer and the two tie-layers were characterized using differential scanning calorimetry (DSC) to determine the corresponding melt temperatures for checking the platen temperature settings during the hot-press process. The HDPE and TL-A have distinct melt temperatures of 130 and 132°C, respectively. Tie-layer B, however, does not have a true melting point but softens gradually because it is an amorphous polymer material.

Hot-Press Parameters

Because HDPE and TL-A have similar melt temperatures and because of time constraints, only TL-B was used in determining ideal hot-press parameters (platen temperature, target pressure, and press time) for bonding HDPE to wood substrates using the tie-layers. A quick screening analysis was conducted following a Box-Behnken design because it requires only three levels of each quantitative factor (Breyfogle 1992; Stat-Ease 2005). The Box-Behnken design procedure using a design of experiment statistical package, Design Expert® (Stat-Ease 2006), generated experimental design points at three levels of temperature (160, 180, and 200°C), pressure (1.035, 1.380, and 1.725 MPa), and press times (120, 300, and 600 s). A total of 69 specimens was tested across the three levels of each variable.

Before hot-pressing, specimens were conditioned in a forced-air oven at 105°C until the specimens mass stabilized, which took approximately 7 da. Once the specimens were oven-dry, a thin barrier layer was applied to one end and placed on a caul sheet. This thin barrier was used to prevent the plastic from bonding to the wood so that a tab was created for a 90° peel test (Fig 1). The specimen was then placed in the hot-press heated to the desired platen temperature. The gap between the platens was reduced to 27 mm to allow the wood to heat for approximately 300 s (enough time for the first few millimeters to reach the melting temperature of the plastic). On comple-

Table 1. Properties of polymers layers bonded to wood substrate provided by manufacturers and determined using DSC.

Material	Manufacturer	Melt index (g/10 min)	Density (kg/m^3)	Tensile strength (MPa)	MAH graft level (wt% MAH)	Melt temperature (°C)
HDPE	Equistar	0.5	953	27	—	130
TL-A	Dow Chemical (Amplify GR 205)	2.0	960	15.6	>1.0	132
TL-B	BASF	—	—	—	—	Amorphous polymer

DSC, differential scanning calorimetry; HDPE, high-density polypropylene; MAH, maleic anhydride; TL-A, maleic anhydride-modified HDPE tie-layer; TL-B, styrene–butadiene polymer tie-layer.

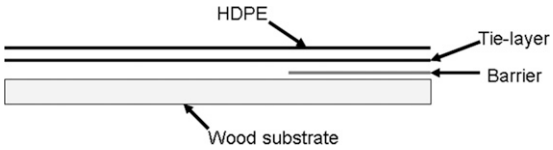

Figure 1. Lay-up configuration of polymer layers on wood substrates for fabrication of peel test specimen.

tion of heating the specimen surface, a single tie-layer and HDPE film was placed onto the heated surface of the specimen (see Fig 1 for specimen lay-up). After placing a release sheet and a caul sheet on top of the layered surface, specimens were hot-pressed following a stepwise pressure control schedule (Michael 2008) that slowly worked the plastics into the wood substrate. After hot-pressing the thermoplastic layers onto the specimens, they were trimmed to a 51-mm wide by 229-mm long by 25-mm thick block for a 90° peel test following D6862 (ASTM 2007b). Speed of testing was 254 mm/min and specimens were tested within 24 h of pressing. Peel strength was calculated using Eq 1 where average recorded load was determined by taking the mean load over the entire peel length.

$$\text{Peel strength} = \text{avg. recorded load}/\text{peel width} \quad (1)$$

Based on the ideal parameters determined, 90° peel test specimens and shear block specimens were prepared for evaluation of bond performance when bonded with TL-A and TL-B.

Bond Performance Evaluation

Test specimens of all three wood substrates bonded with TL-A were evaluated for their bond performance and durability as per D905 block-shear testing procedures (ASTM 2007a). Additionally, bond performance on both radial and tangential surfaces of Douglas-fir solid wood was evaluated. The peel test was not used for TL-A because the tab that is comingled HDPE and TL-A failed in tension before bond failure was initiated between the HDPE barrier layer (bonded with TL-A) and wood substrate. Block-shear specimens were cut into 50-mm wide by 50-mm long by 19-mm thick blocks after undergoing the same hot-press schedule as previously described to bond HDPE to wood substrate using tie-layers. Then, plastic surfaces of two blocks were bonded together by reheating the surfaces with an IR lamp. The blocks were placed approximately 100 mm below the lamp and allowed to heat until the surface temperature reached 140-150°C. Within this temperature range the entire surface of the small blocks was "wetted" (the plastic had softened). Once the plastic was remelted, the two surfaces were placed together and cold-pressed at approximately 700 kPa for 120 s. The specimens were then cut to size according to the testing standard. Testing speed for the block-shear test was 5 mm/min.

Bond performance and durability of the specimens bonded with TL-B was evaluated using the 90° peel test as previously described. All three wood substrates were evaluated as well as the bond performance on radial and tangential surfaces of Douglas-fir solid wood. Speed of testing for the peel test was 254 mm/min. Block-shear and peel test specimens were tested within 24 h of pressing as well as after subjected to an accelerated aged condition. The aging test used was D1101 (ASTM 2006), which is a test method for the integrity of adhesive joints in structural laminated wood products for exterior use. This test method involves two vacuum-pressure cycles while the specimens are completely submerged in water followed by drying for 22 h to complete one full cycle. This weathering test requires a total of two full cycles. Results of bond strength using tie-layers for different wood substrates were compared with those bonded without any tie-layers. However, because two different testing methods were used for evaluating the tie-layers, they were not compared with each other directly.

RESULTS AND DISCUSSION

Hot-Press Parameters

Two primary modes of failure were observed during the peel test, which included the plastic peeling off the wood or the plastic tearing and

eventually failing. A few of the specimens failed by the preferred mode, peeling off the wood, but the majority of the specimens failed by the layered HDPE/TL-B tab tearing during the peeling process (the specimens that failed solely because of plastic layer tearing were not included in the results). It is speculated that the macrovoids that were present on the surface of the Parallam® were being filled in with the HDPE that made the surrounding plastic thinner and more susceptible to tearing. Another place that the plastic would tear was at the tab interface. Figure 2 shows a typical graph of the peel load compared with the peel length for a peel width of 51 mm for a specimen that failed at the wood interface and without any tearing of the plastic. As per Eq 1, the average peel strength was found by averaging the peel load over the entire peel length for a given width.

Response surfaces, based on the Box-Behnken design, at three press times with varying pressure and temperature over the design space are shown in Fig 3. Response surface analysis indicates that peel strength tended to peak between 1.035 and 1.380 MPa in all cases. Based on the response surfaces at 1.035 and 1.380 MPa over changing temperature and time (Fig 4), there is a decrease in peel strength with increase in press time at lower press temperatures. Highest peel strengths were achieved at the maximum press temperature and time studied; however, this had a large variation in the data. Similar studies in the past that looked at thermoplastic bonding to wood substrates yielded lower peel strengths than the process implemented in this study (ie use of a tie-layer at the interface of thermoplastic and wood substrate). Mahlberg et al (2001) looked at the effect of chemical modification of wood using a 90° peel test to determine bond strength and found values of 0.16-0.64 kN/m. The processing parameters were 165°C, 120 s press time, and 300 and 900 kPa. Kolosik et al (1993) used a maleated PP wax modifier to laminate wood substrates with PP. A 90° peel test yielded strength values of 0.04-0.1 kN/m. The processing parameters were 175°C, 275 kPa, and 180 and 360 s press times. In comparison, the peel strengths were 0.82-6.61 kN/m with similar press temperature and times.

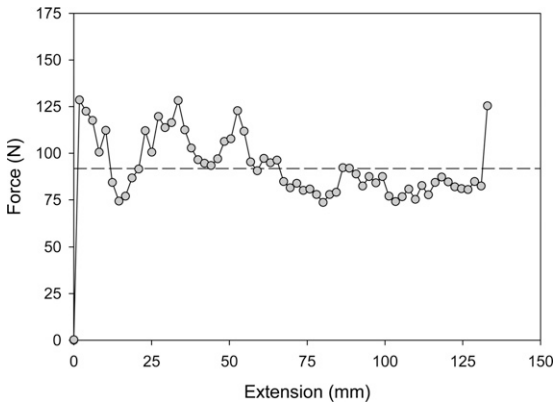

Figure 2. Typical peel load vs extension behavior for specimens with high-density polypropylene bonded using styrene–butadiene polymer tie-layer (TL-B). Dotted line is the average peel force used in Eq 1 for peel strength calculations.

Using Design-Expert® software, numerical optimization was conducted by setting goals that included minimization of time, temperature, and pressure while maximizing peel strength to determine optimal conditions for achieving consistent and maximum possible peel strength values. In setting goals, several combinations of importance levels for the three processing variables could be assigned with one being the lowest and five the highest. It was our intention in this study to minimize energy required during the hot-press operation yet maintain processing parameters that will yield best possible bonding at the interface as measured by peel strength. Therefore, we opted to avoid high platen temperatures, long press times, and large pressures during the bonding process. Thus, we chose to assign an importance level of three to temperature, five to pressure, two to press time, and five to peel strength over a scale of one to five. Results indicate optimum conditions to be 176°C, 1.140 MPa, and 296 s. Visual inspection also showed that barrier layer thickness was consistently uniform in the case of specimens

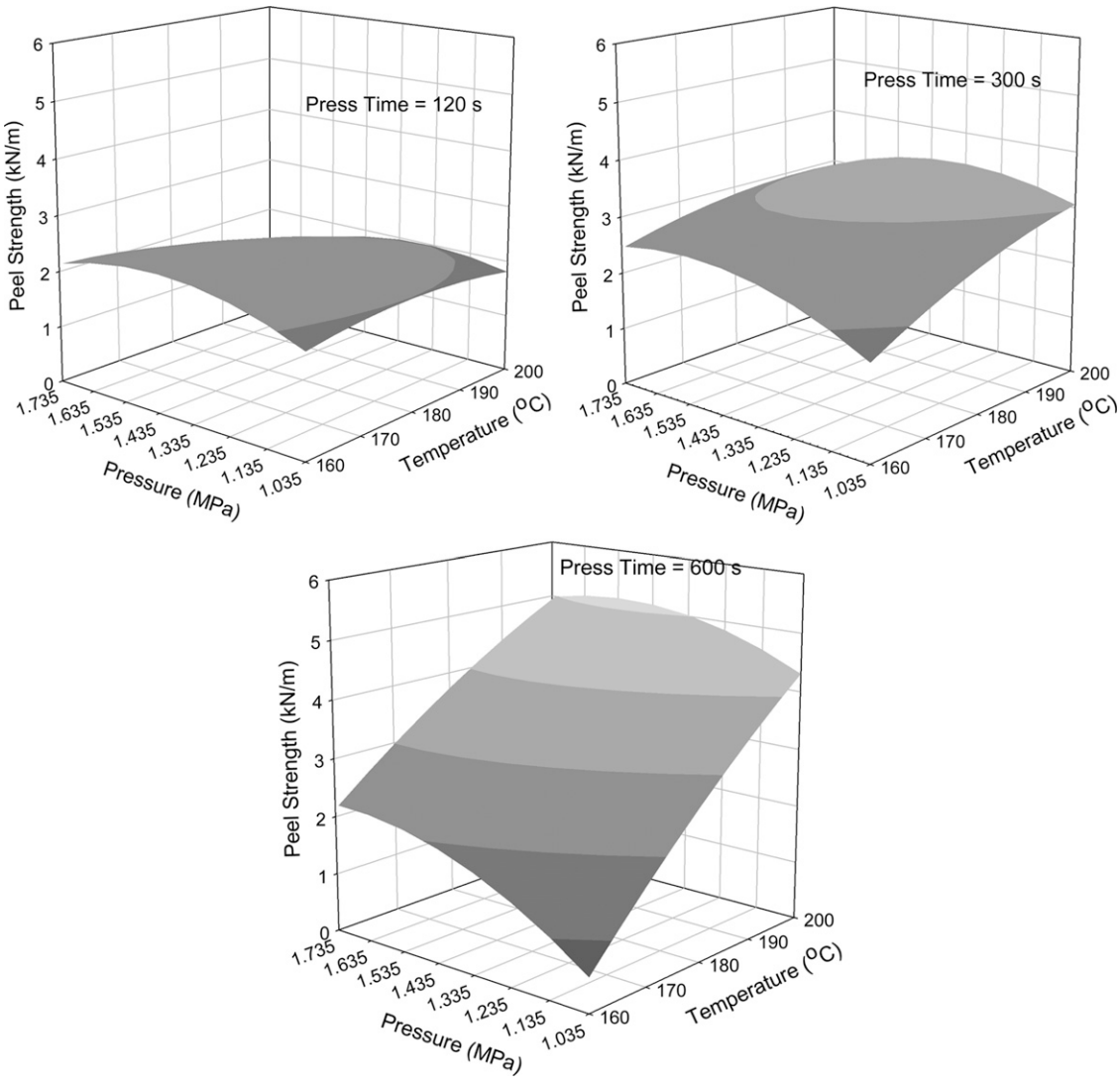

Figure 3. Response surface analysis at three press times to determine hot-press parameters.

that were pressed for 300 s. Additionally, quality of the barrier layer in terms of uniformity in thickness and smoothness was similar for specimens pressed at 1.035 or 1.380 MPa. Also, there was not a significant practical difference between peel strength values when pressed at 1.035 and 1.380 MPa for 300 s at 180°C. Therefore, it seems reasonable to choose the lower pressure value for bonding the thermoplastic barrier layer when the optimum value is 1.140 MPa, especially if it is desired to minimize energy requirements and mechanical damage to the composite during the pressing process.

Bond Performance

The results from the block-shear tests are tabulated and illustrated in Table 2 and Fig 5. For solid wood specimens, shear value of Douglas-fir (7.8 MPa) from the Wood Handbook (FPL 1999) was taken as a benchmark for comparison. In every case, the shear strength of

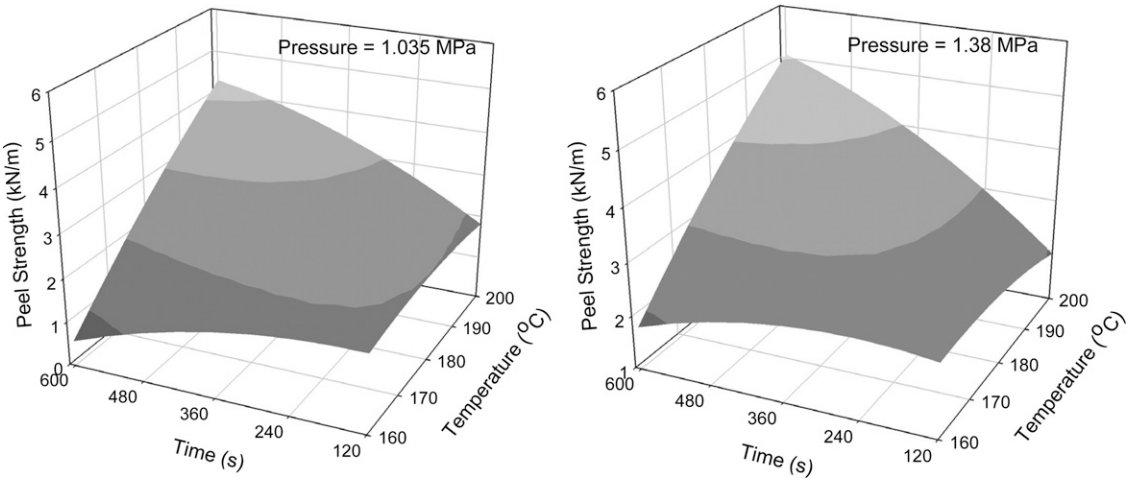

Figure 4. Variation in peel strength over time and temperature in the region of interest at constant pressures of 1.035 and 1.380 MPa.

Table 2. Shear strength results for Parallam® and solid wood specimens in both the initial and aged conditions.

Substrate	Specimen ID[a]	No. of specimens	Mean (MPa)	COV (%)	Minimum (MPa)	Maximum (MPa)
Parallam®	DF-A	12	6.5	39.1	2.9	11.1
	DF-A-WT	12	5.6	23.5	2.3	6.9
	DF-CON	24	5.9	20.3	2.8	8.1
	SP-A	12	8.2	20.8	6.1	11.7
	SP-A-WT	12	7.4	21.0	5.0	10.5
	SP-CON	24	7.5	13.0	5.5	9.5
Solid wood	RAD-A	12	8.8	16.6	6.5	10.6
	RAD-A-WT	12	2.8	52.9	0.8	5.8
	TAN-A	12	8.0	46.4	2.4	15.7
	TAN-A-WT	12	3.6	70.7	0.5	8.0

[a] The first abbreviation is for the species in the case of Parallam® or surface orientation in the case of solid wood (DF = Douglas-fir, SP = southern pine, RAD = radial surface, and TAN = tangential surface). The second abbreviation is for the treatment type (A = tie-layer A and CON = control block-shear specimens cut from the substrate with no bond layer at the shear interface). The last abbreviation is for the conditioning of the specimen (no entry means that the specimen was tested within 24 h of pressing and WT for after accelerated aging test).

specimens bonded with TL-A yielded higher values than control specimens, indicating that the bond formed with TL-A is as strong as the shear strength of the wood substrate (Parallam®). This establishes at least the lower limit of the bond strength that can be achieved with TL-A.

Southern pine Parallam® specimens had higher shear values (8.24 MPa) than the Douglas-fir Parallam® specimens (6.48 MPa). Accelerated aging of the block-shear specimens did not significantly reduce the shear strength between the HDPE barrier layer and Parallam® substrate. On the average, the shear strength of Parallam® specimens of both species decreased by less than 15% after undergoing accelerated aging. Even with this decrease in shear strength, it was not significantly different than the shear strength of the substrate itself. Solid wood, conversely, seemed to be greatly influenced by the differential shrinking/swelling of the wood, perhaps because of less mechanical interlocking of the polymer within the surface compared with Parallam®. After the accelerated aging of the solid wood, the shear strength was greatly reduced for both surfaces (by 68% for radial and 56% for tangential). It was observed during testing that nearly one-half of the specimens had some degree of interfacial separation because of shrinking or swelling of the wood. Analysis of variance results on block-shear values indicated

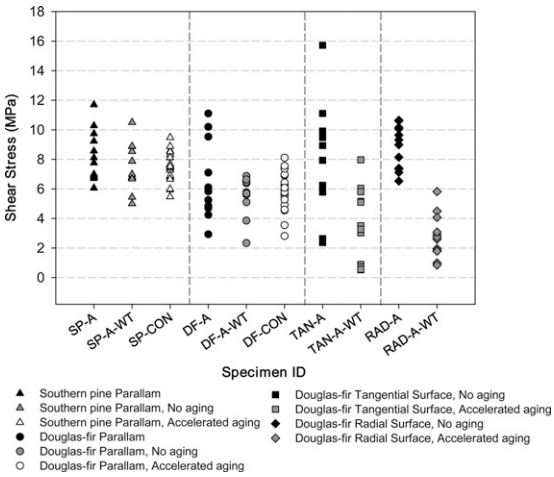

Figure 5. Block-shear test results of specimens with high-density polypropylene (HDPE) at the bond interface with and without maleic anhydride-modified HDPE tie-layer (TL-A) for southern pine and Douglas-fir Parallam® and with TL-A for Douglas-fir solid wood (tangential and radial surfaces).

Table 3. Duncan's multiple range test for shear strength of specimens with and without tie-layer A (means with the same letter are not significantly different at significance level of 0.05).

Specimen type[a]	Mean value (MPa)	Duncan grouping
RAD-A	8.8	A
SP-A	8.2	A
TAN-A	8.0	AB
SP-CON	7.5	AB
SP-A-WT	7.4	AB
DF-A	6.5	BC
DF-CON	5.8	C
DF-A-WT	5.6	C
TAN-A-WT	3.6	D
RAD-A-WT	2.8	D

[a] The first abbreviation is for the species in the case of Parallam® or surface orientation in the case of solid wood (DF = Douglas-fir, SP = southern pine, RAD = radial surface, and TAN = tangential surface). The second abbreviation is for the treatment type (A = tie-layer A and CON = control block-shear specimens cut from the substrate with no bond layer at the shear interface). The last abbreviation is for the conditioning of the specimen (no entry means that the specimen was tested within 24 h of pressing and WT for after accelerated aging test).

a significant difference between the treatments investigated. Further comparison of means based on Duncan's multiple range test was performed at a significance level of 0.05. Comparison of means result is shown in Table 3, where means with the same letter are not significantly different at a significance level of 0.05.

Peel test results with TL-B are summarized in Fig 6. The second abbreviation in the specimen identification is for the treatment type (N indicating HDPE only with no tie-layer and B indicating tie-layer B). Other abbreviations are similar to those for TL-A (Table 2). Only about one-third of the specimens had a "good" peel from which a peel strength value was determined. If the barrier layer started to tear, it was not valid because it did not reflect the peel strength but more of the yield strength of the thermoplastic/tie-layer as it would tear.

In general, peel strength improved significantly with the use of TL-B for all substrates. This improvement was not as significant when bonded to the radial surface of Douglas-fir solid wood specimens. When comparing the solid wood peel strengths, we can see that the TL-B specimens

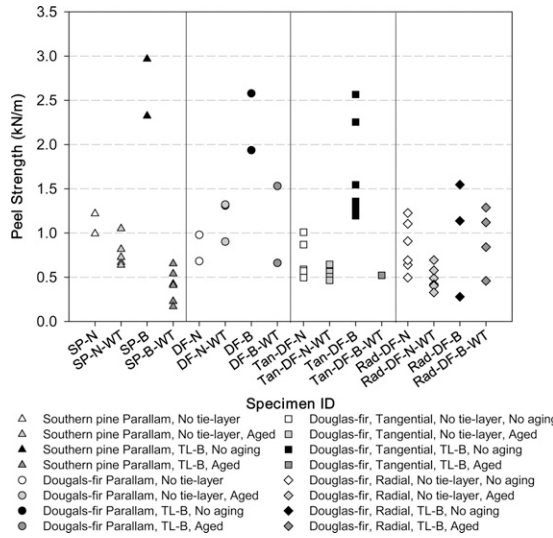

Figure 6. Peel test results of high-density polypropylene bonded to wood substrate specimens with and without styrene–butadiene polymer tie-layer (TL-B) for southern pine and Douglas-fir Parallam and Douglas-fir solid wood (tangential and radial surfaces).

have higher peel strengths than the specimens without a tie-layer. This was more significant on the tangential than the radial surface. Specimens with HDPE bonded without a tie-layer performed better on the radial specimens. This could be

because of the ability of the HDPE to penetrate through the radial surface because of the presence of pits between ray parenchyma and longitudinal cells in Douglas-fir (Summitt and Sliker 1980). Specimens with HDPE bonded to the tangential surface using TL-B yielded greater peel strength than when bonded to the radial surface. The southern pine specimens yielded slightly higher peel strengths than the Douglas-fir specimens, which could be because of the open pit structure of the southern pine cell walls. Scanning electron microscope micrographs of southern pine (Gacitua and Wolcott 2009) revealed that thermoplastic penetrates the pits; however, micrographs of a Douglas-fir composite revealed no such penetration because it is generally considered to be refractory due to a common occurrence of pit aspiration during drying (Meyer 1971; Islam et al 2008).

For the Parallam® substrate, the HDPE barrier layer without a tie-layer had an average peel strength of 0.97 kN/m, and the specimens with TL-B had an average peel strength of 2.45 kN/m, an increase of over 150%. However, there is no significant difference in the bond strengths between the two Parallam® species, Douglas-fir and southern pine. Peel strengths of specimens subjected to accelerated aging were significantly reduced. Visual observations indicate a separation of TL-B and HDPE from the surface of the wood. In both cases, aging had a detrimental effect on peel strength. The Douglas-fir Parallam® specimens with TL-B retained approximately 49% of the initial peel strength, however southern pine Parallam® specimens with no tie-layer retained approximately 71% of the initial peel strength. HDPE bonded with TL-B delaminated from the wood during the accelerated aging test. The southern pine Parallam® specimens with tie-layer B only retained approximately 15% of their initial peel strength.

CONCLUSIONS AND RECOMMENDATIONS

Thermoplastic encapsulation is a promising way to improve the durability of wood composites by creating a barrier to moisture uptake and leaching of preservative chemical treatments. Because most polyolefins such as HDPE do not bond well to a polar material such as wood, a possible solution is to use a tie-layer to improve the bond. In this study, the efficacy of two types of tie-layers in bonding HDPE to treated wood substrates was examined along with determining the ideal hot-press parameters necessary to achieve a good bond.

Based on the general trends and to minimize the required energy during the hot-press process, the best combination of the processing parameters appeared to be 180°C, 1.035 MPa, and 300 s. Block-shear tests of specimens bonded with TL-A yielded very good adhesion properties. A large majority of the shear blocks failed not at the wood–plastic interface, but in the wood structure itself. The results of the peel tests were mixed; however, it was shown that TL-B did increase the peel strength when compared with specimens with no tie-layer in the initial condition. Aged specimens bonded with TL-B had a high percentage of the bond interface delaminating. In conclusion, the best bond that was produced was the maleic anhydride-modified HDPE tie-layer–HDPE plastic combination bonded to a southern pine Parallam® substrate. The shear strength of the interface between the layered thermoplastic/tie-layer and wood composite was higher or equal to the wood composite shear strength with no significant loss of shear strength after the aging process.

The potential to achieve a durable bond between thermoplastic and wood substrate using tie-layers has been demonstrated; however, methods to effectively and economically encapsulate structural members such as Parallam® beams has to be further explored. A suggested method would be to wrap the beams with a tie-layer and thermoplastic barrier layer films, place them into a sealed vacuum bag, and apply optimum pressure and temperature in an autoclave. A more economical and efficient method for industrial production is the use of pultrusion technology, which can potentially be integrated with a continuous hot-press process for wood composite manufacturing. Another issue to

address is *in situ* coating of areas exposed after onsite trimming to length or drilling holes for fasteners. A field treatment such as a commercial sealant could be effective under these circumstances.

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