Improving the Adhesion of High-Density Softwoods with Isocyanate Based Adhesives through Surface Incision

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Glue laminated timber is currently manufactured using classical adhesives such as resorcinol formaldehyde and phenol resorcinol formaldehyde. These are proven structural adhesives; however their long cure times and rising costs are creating opportunities for newer technology adhesives. One such class are the structural polyurethanes with decreased spread rates and faster curing times. Their limitation lies in their inability to adhere timbers of densities exceeding 800 kg/m^3. When used on species including the southern pines (*Pinus* spp.) with a high frequency of latewood, they delaminate after accelerated weathering tests due to stresses imposed on the glue line during the drying process. Surface incision has been trialed in this study to increase the penetration of polyurethane adhesives and reduce glue line stresses. The study shows that incisions to a depth of 2 mm decreases delamination when compared to matched samples with no incisions. The significant increase in glue line surface area may result in stress reduction as the more compliant adhesive may distribute the stresses better across the glue line. Furthermore, microscopic analysis suggests that the incisions are reducing glue line stress through crack propagation into the timber pointing to the possibility of increased timber compliance at the glue line.

Keywords: 1C-PUR; Incision; Slash pine; Caribbean pine; Glulam; Delamination; Stresses

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INTRODUCTION

Timber is an economically and environmentally favorable alternative to steel and concrete and is rapidly gaining popularity in both the domestic and the commercial building industries. The move towards timber is driving an increased demand for engineered wood products (EWPs), which in turn is giving rise to new techniques enabling more efficient use of the timber resource.

Most of the timber used in the construction industry in Australia is currently sourced from faster grown plantations, which produce smaller diameter logs with a higher range of defects and are normally deemed of lesser commercial value when compared to the larger timber produced from native forest sourced logs. The majority of plantation sourced logs have a limited product scope within the light construction industry such as house framing and trusses (Walker 2015).

Engineered wood allows these lower quality logs to be converted into more versatile products such as veneer for plywood and laminated veneer lumber, or short length solid timber for cross-laminated timber (CLT) and glued laminated timber. This converts the plantation grown timber into products that can now be used in structural applications.

Glued laminated timber, or glulam for short, is an EWP that can be manufactured from finger-jointed short lengths of timber to produce longer length elements suitable for structural or non-structural applications. The strength-reducing defects are normally removed from the timber prior to finger jointing to produce long defect free lengths of timber that are glued together horizontally to manufacture a glulam beam. The mechanical performance of the laminated product would be as good, if not better, than that of the solid timber equivalent (How *et al* 2016).

Historically the most common adhesives used in the manufacture of glulam are polycondensation curing resins such as resorcinol formaldehyde (RF) and phenol resorcinol formaldehyde (PRF). In recent years the polyaddition curing adhesive, polyurethane, has started to replace the classical adhesives based on its fast curing properties, lack of formaldehyde, and the single component system that is supplied ready to use (Lehringer and Gabriel 2014). Single component polyurethanes (1C-PUR) are manufactured with differing rates of reactivity depending on the application they are used for. Open times range from 70 min to as little as 5 min. The cure time of the 1C-PUR adhesives is governed by the reactivity of the isocyanate groups with both the atmospheric moisture and moisture within the substrate. The increase of reactivity and/or moisture levels results in an increased curing rate (Avar *et al* 2012).

Studies have shown that these moisture curing pre-polymer adhesives are capable of high bond strengths and show a ductile behaviour under load (Leudtke *et al* 2015), with some instances showing 15 times lower elastic modulus than Melamine Urea Formaldehyde (MUF) adhesives (Lehringer and Gabriel 2014). This is also reflected in the stress strain curves for the 1C-PUR adhesives. When compared to the more rigid polycondensation adhesives such as resorcinol formaldehyde, the 1C-PUR adhesives show a larger area under curve indicating that the adhesive can absorb higher levels of stress. This higher compliance of the adhesive can be beneficial as it helps to relieve local stress concentration.

Although 1C-PUR adhesives have been used extensively on low density softwood species, they have experienced problems meeting the delamination requirements of EN302-2:2013 when the adhesive is used on higher density species or timber with high extractives' content. In an effort to improve the capacity to bond the higher density timbers, a number of studies have been published with the majority of these investigating the use of 1C-PUR on timber pre-treated with a primer solution. Luedtke *et al.* (2015) combined a primer pretreatment with various other parameters including surface machining and press time on European hardwoods such as oak (*Quercus* spp.) and beech (*Fagus* spp.). The primer treatment on these species showed a significant improvement in bond quality; however not all species had equal success. Custodio *et al.* (2008) reviews the use of primers and coupling agents that he states are prevalent in the aeronautical and automotive industry yet still underutilized in the timber products' industry. He argues that more research needs to be conducted in this space to solve important adhesion problems. Amen-Chen and Gabriel (2015) successfully used a water-based primer on southern yellow pine (*Pinus* spp.) to increase the delamination resistance of polyurethane adhesives.

Although there have been numerous studies done on mechanical surface preparation and its effect on adhesion, there are limited studies that have investigated this in relation to PUR adhesives. Knorz *et al.* (2015) compared peripheral planing, sanding, and facemilling to find that surface texture had significant impact on shear and delamination results. While PRF and MUF showed high resistance to delamination with sanded surfaces, PUR, and Emulsion Polymer Isocyanate (EPI) adhesives performed best with facemilled surfaces. Kuljich *et al.* (2013) also compared planing with sanding when using isocyanate adhesives. This study showed that sanding with a rough 80-grit sandpaper produced the most durable adhesive bonds.

There has been a recent interest in Queensland, Australia, to make use of the managed southern pine plantations to manufacture glulam beams using 1C-PUR technology. The stands comprise of slash pine (*Pinus elliotti*), Caribbean pine (*Pinus caribaea* var. *hondurensis*) and hybrids between these species (HQ plantations Southern Pine fact sheet). As the beams are intended for structural purposes, the higher density material of machine-graded pine (MGP12 to MGP15), which derive their mechanical properties from the high frequency of high-density latewood bands, will be used for production.

Early studies (Hse 1968) conducted with southern pine veneers using a phenol formaldehyde resin showed that adhesion between combinations of the low-density earlywood and the high-density latewood result in significantly different bond qualities. Panels manufactured with an earlywood to earlywood combination showed minimal delamination (1%) after three months in an exposure deck, while panels constructed with an earlywood to latewood combination showed higher rates of delamination (40%). The highest rate of delamination however was given by latewood to latewood panels with delamination as high as 80%. The poor bond was attributed to less adhesive penetration into the dense, hard latewood timber as opposed to the lower density material in the earlywood.

Recent project work carried out by the author at the Salisbury Research Facility comparing different reactivity adhesives has shown that the higher reactivity, faster curing 1C-PUR adhesives are not suitable for use with MGP12 to MGP15 graded slash pine and Caribbean pine in structural applications. The study trialed two single component polyurethane adhesives from the same manufacturer with open times of 70 min and 30 min. MGP15 southern pine timber was used to manufacture glulam test samples of six lamella. Both adhesives were applied as per manufacturer’s recommendations and tested according to AS/NZS 1328.1:1998, the Australian standard that governs the adhesive requirements for glulam element production, product verification processes, qualification, and routine test procedures.

The results showed that while the slower curing 1C-PUR showed minimal delamination and satisfied the requirements of the standard, the faster curing adhesive had much higher levels of adhesive delamination and exceeded the thresholds defined in the standard. As both adhesives have the same base chemistry and initial viscosity, the failure can be attributed to the reactivity of the adhesive. The extra reactivity of the adhesive gave a faster rate of polymerisation, which increased the viscosity of the adhesive as it cured. This may have negatively affected penetration, reducing both mechanical and chemical bonding resulting in delamination.

This study investigated the effect of surface incision on high density southern pine timbers that exhibit a high frequency of latewood. The hypothesis is twofold: 1.) By incising the timber, the adhesive will penetrate further into the cellular structure of the wood through the incisions promoting a deeper level of mechanical bonding. To ensure the best possible chance of adhesion it is imperative that adequate surface penetration is undertaken. The incisions will also open up the surface cellular structure, increasing the available active sites, which in turn increases the wetting capacity and the opportunity for chemical bonding. 2.) A second effect of the incisions will be to reduce the drying stresses on the glue line in a similar manner that stress kerfing reduces drying stresses in solid timber. The reduced stresses around the glue line should result in reduced delamination.

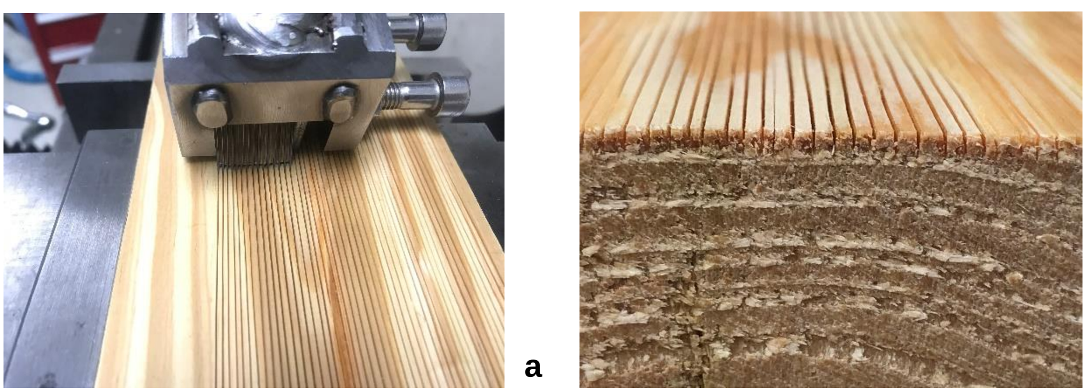
EXPERIMENTAL

Materials

A mix of two exotic pine plantation species, slash pine (*Pinus elliottii)* and Caribbean pine *(Pinus caribaea),* was supplied by Hyne Timber as untreated boards and KopCoat-treated boards. KopCoat is a preservative treatment solution applied by vacuum pressure impregnation (VPI). Test material comprised of strength graded MGP15 backsawn boards sized at 90 mm x 35 mm x 5,400 mm with an average timber moisture content of 12%, air dried density of 815 kg/m^3 (8%) and a minimum stiffness of 10000MPa. Boards that presented a large frequency of high density latewood on the face and had minimal defects were selected for the trial. Sufficient boards were selected from both the untreated and treated stock to give thirty 400 mm length boards which were then conditioned at 20 °C 65% relative humidity (RH) (12% equilibrium moisture content (EMC)) in a constant environment chamber until constant mass was achieved. Immediately prior to incision, samples were passed through a thicknesser planer equipped with freshly sharpened blades to remove 1.5 mm from both faces. The 400 mm lengths were cut in the middle to give two 200 mm matched samples and identified with a matching label on each half. One set of each of the matched samples was submitted for incision and the other was used for adhesion without incision treatment.

Incision equipment and procedures

Irwin Bi-metal safety knife utility blades were mounted 1.2 mm apart in a custom built jig fitted to a metal milling machine. Blade angle was set at 20° to the timber surface and with a feed speed of 900 mm/min, 1 mm incisions were cut into the face. A second pass was made resulting in a final incision depth of 2 mm. Average width of the incisions was calculated to be 0.2 mm at the surface. Incision was carried out on all adhesive contact faces (Fig. 1). Upon incision all samples were submitted for adhesive bonding.



a b

Fig. 1. Timber surface incised with special tool to a final depth of 2 mm

Sample manufacture

Twenty glulam elements of six lamella each were manufactured for the study as shown in Table 1. Samples were manufactured with matched pairs ensuring that all lamella were replicated between the incised and unincised samples. Lamella were oriented with growth rings in a pith to pith, bark to bark orientation. The adhesive used was the fiber filled, structural rated 1C-PUR adhesive Jowat Jowapur 686.30 (viscosity of approximately 10500 mPas at +20 °C and density 1.15g/cm^2). The adhesive is manufactured by Jowat

Switzerland with a 1 month shipping duration to Australia. This may result in a slight viscosity increase due to a slow curing reaction however this is catered for in the adhesive use-by date.

This recommended spread rate for structural applications is 150 to 250 gsm and an open time of 10 to 15 minutes at +20 °C. Pressing time is 45 min with a recommended pressure of 0.6 to 1.0 MPa. While 200 gsm proved sufficient for the manufacture of samples with no incisions, incised samples required an increased spread rate of 600 gsm to ensure sufficient adhesive was available to penetrate into the incisions. The adhesive was manually spread with a serrated applicator in a perpendicular direction to the incisions to manually force adhesive into the cavities. Pressure was applied to all samples at 1.0 MPa. Samples were allowed seven days to ensure the adhesive had achieved full cure at an average temperature of 27 °C.

Table 1. Glulam Manufacture

Sample ID	Replicates	Surface planing	Surface incision	KopCoat treatment
UC	5	Yes	No	No
UI	5	Yes	Yes	No
TC	5	Yes	No	Yes
TI	5	Yes	Yes	Yes

UC – untreated control, UI – Untreated incised, TC – Treated control, TI – Treated incised

Testing protocols

Each test sample was tested according to the requirements of AS/NZS1328.1:1998.1 (12). Glue line integrity is based on testing the glue line in a full cross-scetional specimen cut from the glulam member. The requirements of the standard are that any time a new adhesive or species combination is introduced then qualification tests have to be conducted. The elements in this study are manufactured as Service class 3 elements according to the standard, *i.e.* for applications of high moisture and direct exposure to the sun and/or rain. The qualification test required is as outlined in Appendix C, Test cycle A. This is a delamination test where the acceptance criteria allow a maximum delamination of 40% for each individual glue line and no more than 5% total delamination of the sum total length of all glue lines. Samples that showed delamination between 5% and 10% are exposed to another test cycle and the total percent delamination should not exceed 10% after this final cycle for the sample to pass.

The testing protocol was as follows. A 75 ± 5 mm test sub-sample was obtained from the centre of each 200 mm test sample. Each individual glue line was labeled from one to ten, and the length of each was determined across the grain using a magnifying lens and digital calipers. Water impregnation was done in a pressurized treatment cylinder. Samples were totally immersed in water at 20 °C, a vacuum was applied at -78 kPa for 5 min followed by a pressure cycle of 550 kPa for one hour. While still immersed, the vacuum/pressure cycle was repeated giving a total of two impregnation cycles. Samples were dried in a computer-controlled drying chamber, placed approximately 60 mm apart, and oriented so that the end grain was perpendicular to the flow of air. The chamber was baffled to ensure that all airflow was directed over the samples. An initial airflow of 2.4 m/s was set in the centre of the sealed chamber and 50 mm in front of the samples using a National Association of Testing Authorities (NATA) calibrated vane anemometer. All samples were dried under constantly monitored and controlled conditions for 21 h at 65 °C, a relative humidity of 10% and an air velocity of 2.4 m/s. The water impregnation cycles and drying cycle was carried out twice in total before samples were inspected for delamination with lengths recorded as outlined in AS/NZS1328.1:1998.

Microscopy

Fluorescent microscopy was conducted using a Nikon Eclipse LV100ND fitted with a Nikon Digital Sight DS-Fi2 colour camera. This was used to inspect the level of adhesive penetration within the glue line and the incisions. The microscope was set to an excitation range of 465 to 495 nm with an emission spectrum of 515 to 555 nm.

Micro-computed tomography, or micro-CT was carried out using a Bruker SkyScan 1272 (Massachusetts, USA). Operating conditions were as follows: Voxel size was set at 7 mm with a voltage of 30 kV and 212 mA of current. The exposure time was 850 ms using a 0.25 mm filter. Camera binning was 3 × 3, the rotation step was 0.2° (averaging 2) over a range of 180°. Scan length of 1 h 48 min and 55 seconds was used. The dataset was reconstructed using NRecon and InstaRecon software, with beam hardening correction of 30% and ring artefact correction of 3.

RESULTS AND DISCUSSION

Incision of the timber surface showed a marked improvement in the ability of the adhesive bond to resist delamination (Table 2 and Fig. 2). Both the unincised and incised for untreated and KopCoat treated samples passed the % maximum delamination; however incising the timber resulted in a significant reduction ($p = <0.001$) of the bond line failure. The % total delamination was also significantly better for all samples that had incisions to the surface ($p<0.001$), with both untreated and KopCoat treated material well within the standard requirements, whereas all the control samples with no incisions failed

Four out of the five untreated/incised (UI) samples showed levels of delamination well below the 5% limit set out in AS1328.1. UI-5 had a percentage maximum delamination well within the standard limit; however, the percentage total delamination failed after the initial cycle with a value of 6.18%. The third treatment cycle of UI-5 showed a slight increase in percentage maximum delamination from 9.0% to 9.6%, and the percentage total delamination rose from 6.2% to 7.1%, this time well within the 10% maximum. Therefore, all the untreated and incised elements passed the standard requirements for both parameters. All the KopCoat treated/incised (TI) timber passed both the percentage maximum delamination and percentage total delamination.

The untreated samples that were surface planed but had no incision cut into the contact surface (UC) resulted in levels of delamination much higher than the matched incised samples. All the untreated samples showed much higher delamination in the individual glue lines with UC-2 exceeding 40%. Of the five untreated samples, only UR-3 came in below the 10% total delamination. All KopCoat treated samples that were planed but had no incisions (TC) showed individual glue line delamination levels below the percentage maximum delamination. However, the total percentage delamination of all glue lines exceeded the requirements of the standard.

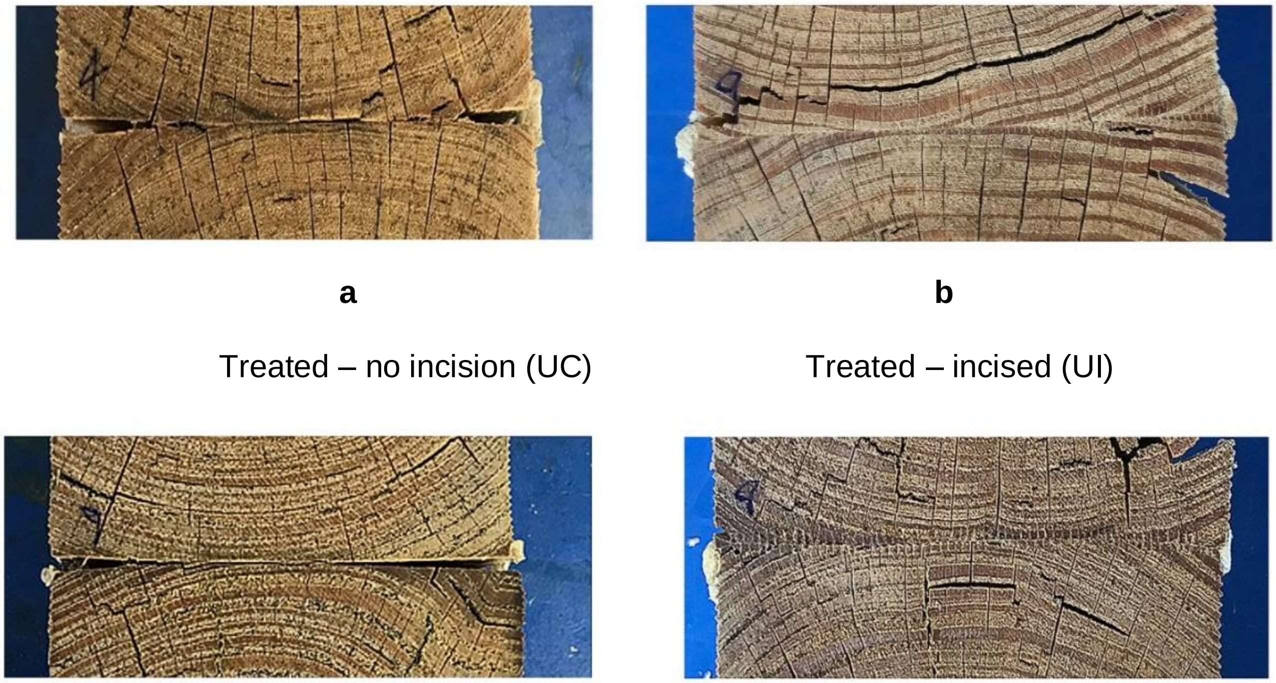
Table 2. Glue Line Delamination Outcomes Comparing Incised and Unincised Glulam Elements

Incised				Unincised			
Sample number	% Maximum delamination	% Total delamination	Outcome AS1328.1	Sample number	% Maximum delamination	% Total delamination	Outcome AS1328.1
UI-1	2.6	1.4	Pass	UC-1	34.0	18.5	Fail
UI-2	5.8	2.1	Pass	UC-2	44.5	13.2	Fail
UI-3	1.8	0.7	Pass	UC-3	23.2	9.3	Fail
UI-4	4.5	2.7	Pass	UC-4	18.8	11.7	Fail
UI-5	9.0	6.2	Fail	UC-5	30.0	18.4	Fail
UI-5 (2)*	9.6	7.1	Pass				
Average	4.9	2.8			30.1	14.2	
TI-1	5.9	2.7	Pass	TC-1	16.6	10.7	Fail
TI-2	1.5	0.3	Pass	TC-2	22.6	18.1	Fail
TI-3	5.6	1.3	Pass	TC-3	23.3	26.9	Fail
TI-4	0.0	0.0	Pass	TC-4	39.8	21.1	Fail
TI-5	6.7	2.7	Pass	TC-5	38.8	19.7	Fail
Average	3.9	1.4			28.2	19.3	

**denotes third treatment cycle conducted*

Untreated – no incision (UC) Untreated – incised (UI)

Treated – no incision (UC) Treated – incised (UI)



a b c d

Fig. 2. Delamination comparison of untreated samples (top – a,b) and KopCoat treated samples (bottom - c,d)

The Jowat Jowapur 686.30 1C-PUR adhesive contained a UV fluorescing agent that was included at manufacture to allow a visual study of the adhesive penetration into the cells under fluorescence microscopy. A cross section of the glue line was examined to determine adhesive penetration. Figure 3 (top) shows that good penetration of the adhesive was achieved in the cells around the glue line that is tight and showing no evidence of gas bubbles ensuring good cohesion of the adhesive within the glue line. The incisions also show good penetration of the adhesive into the surrounding cells (Fig. 3b). The increased level of penetration afforded by the incisions gives better adhesive interaction with the timber resulting in greater mechanical bonding. Figure 3b also shows the large number of gas bubbles present within the adhesive in the incisions. These are creating a large number of voids that reduce the cohesive strength of the adhesive within the glue line.

Micro-CT analysis also shows the large concentration of gas bubbles present within the incisions (Fig. 4). During manufacture, the adhesive spread rate was purposely increased to ensure that sufficient adhesive was available to penetrate into the incisions. It was also applied in such a manner that forced the adhesive to enter into the incisions (Fig. 5). While there is the possibility that there may be some trapped air caught in the adhesive during the manufacturing process prior to pressing, every effort was made to encourage full penetration of the adhesive into the timber from within the incisions. It is very likely that the gas bubbles were generated within the adhesive as a by-product from the reaction of the isocyanates with the water during the curing process.

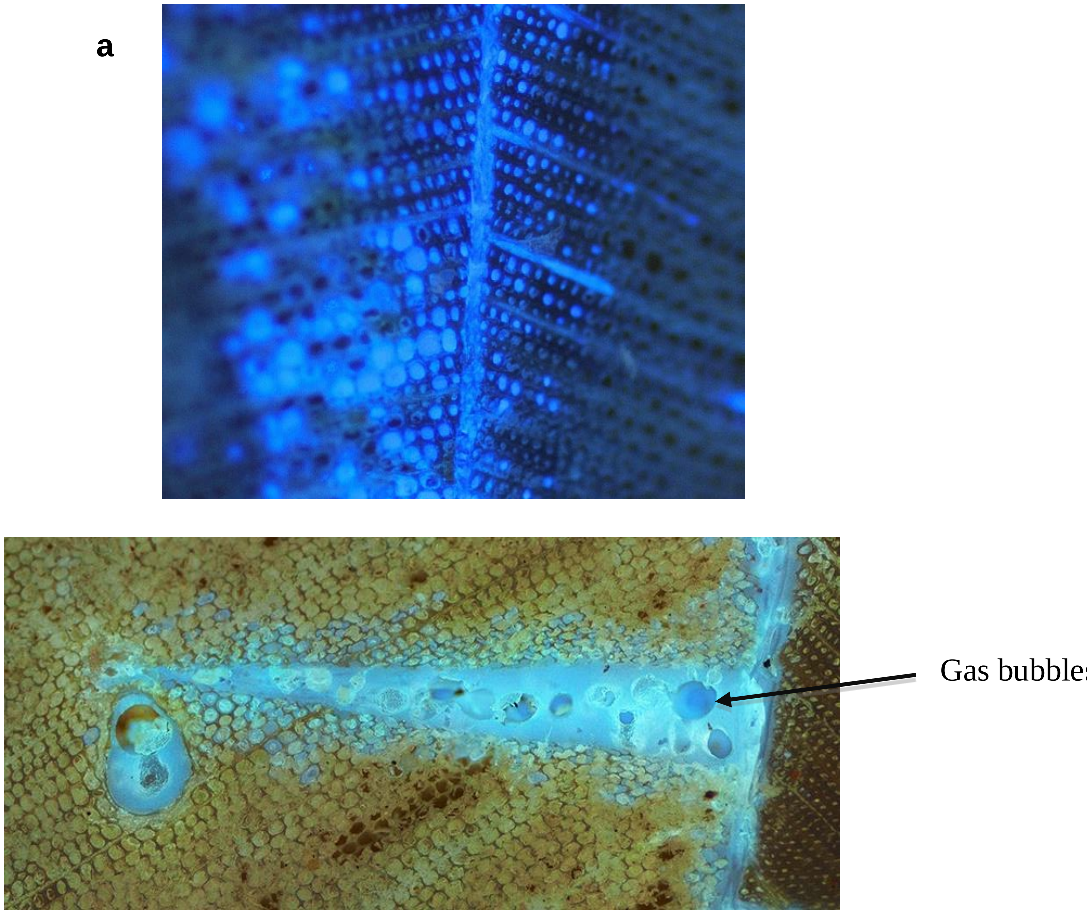


Fig. 3. Top (a) - Adhesive penetration into latewood cells (right) and larger earlywood cells (left) – 100x magnification. Bottom (b) – Adhesive penetration into earlywood cells around incision – 50x magnification.

Due to the lower rate of tangential gas permeability of southern pines (Comstock 1970), the bubbles in the incisions would be a result of the gas not totally diffusing into the timber material as external pressure is applied during curing and thus building up in the larger volume of the incision. Due to complex geometry of glue interface, it is likely that pressure in certain areas, particularly in the incisions, is lower than in zones with no incisions. These may have caused the bubbles of CO_2 formed within the glue line to travel along the glue line under pressure until reaching the larger volumes of the incisions thus creating more bubbles within the incisions.

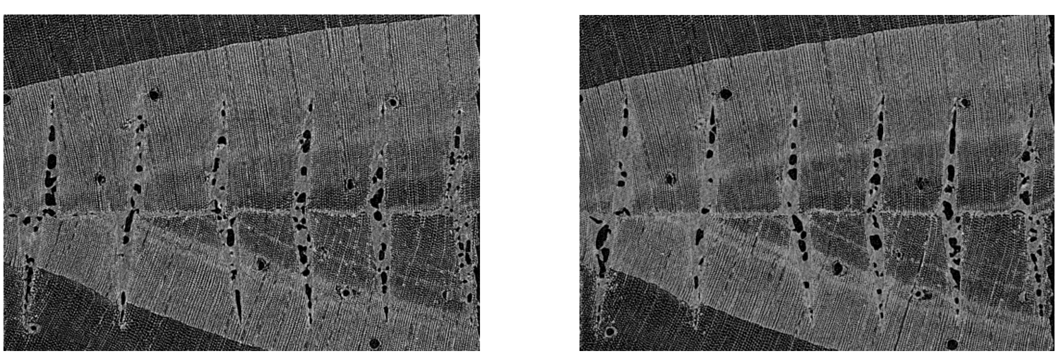


Fig. 4. Micro-CT micrographs of incised glue lines

Fig. 5. Higher spread rates and application perpendicular to the incisions were two techniques used on incised samples to ensure adhesive penetration into incisions.

The incisions may also act in a manner similar to case hardening relief in kiln dried timber. This technique is commonly used to relieve moisture differential induced stresses in flat boards that are generated by the drying process (Technical note 1952). In the same way, the incisions could be reducing the moisture differential stress in the glue line during the drying process. The voids created by the gas bubbles within the incisions could be increasing the compliance of the adhesive in the bondline. This, along with the larger glue line surface area and increased mechanical bonding of the incisions, are increasing the samples tolerance to failure. Increased timber compliance may also be a result of the properties of the incised surface. The flexibility inherent within the 1C-PUR adhesives allows the timber a certain degree of movement through the glue line. By introducing room for the timber to flex and contract as a function of varying moisture levels, the stresses are transferred through the incisions to timber around the incision zones such as those shown in Figs. 6 and 7. These cracks direct the forces away from the glue line and into the fibers deeper in the timber, resulting in less delamination of the glue line itself. Micro-fractures may also be created at the incision bed during the incision process. This causes a weak point where drying induced stresses related to moisture gradients result in further propagation of these cracks into the timber.

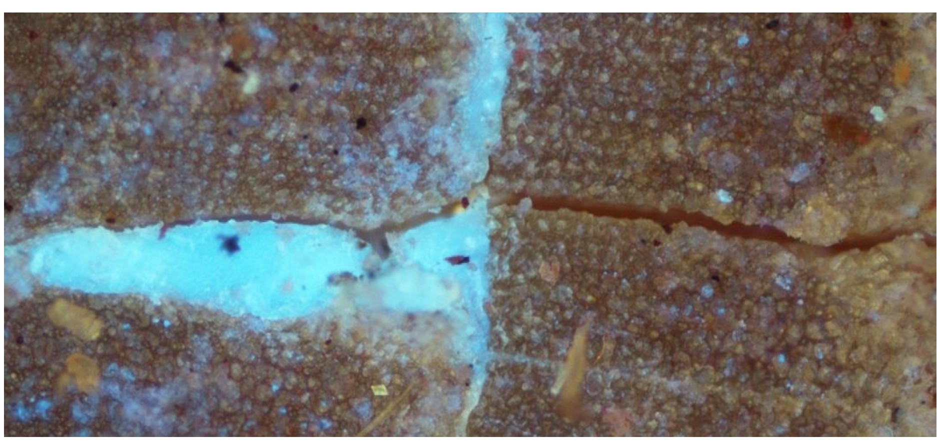


Fig. 6. Incision expanded as a result of drying shrinkage causing stress reduction in the glue line.

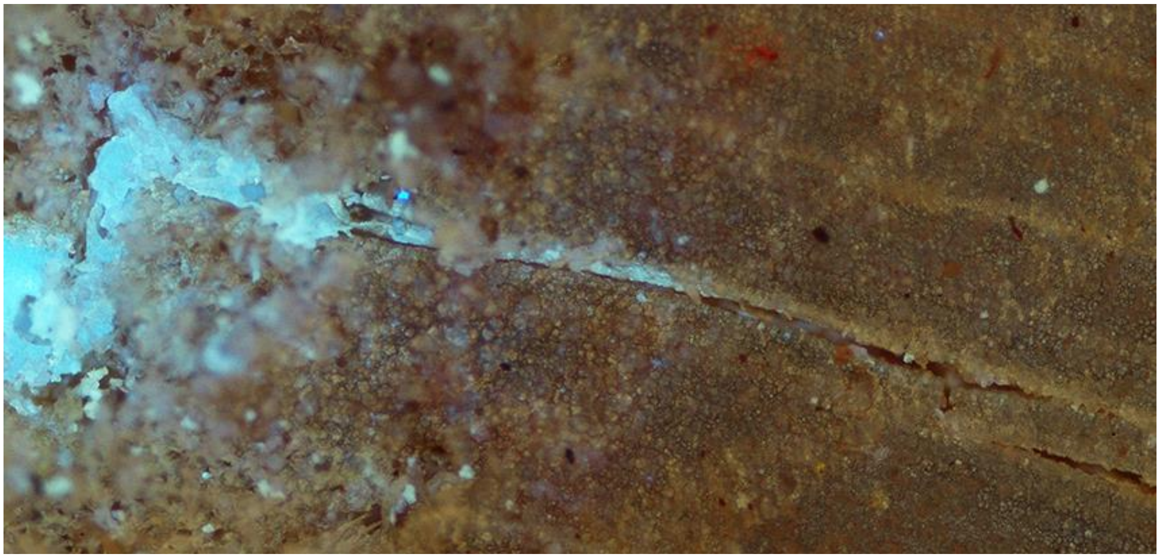


Fig. 7. Crack propagation at the base of the incision due to drying stress.

CONCLUSIONS

1. Incision into the surface of the timber to a depth of 2 mm showed a 79% and 95% decrease in delamination for untreated and KopCoat treated samples, respectively, after accelerated weather testing when compared to matched samples with no incisions.
2. The incisions increase adhesive penetration by introducing adhesive deeper into the cellular structure of the timber lamella.
3. The decrease in delamination is directly attributable to the incisions, possibly as a cumulative effect of the increased glue line surface area giving increased mechanical bonding and the increased compliance of both the glue line and the timber.
4. Stress is also reduced by crack propagation in the base of the incision dissipating stress to the timber lamella.

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