ACOUSTIC MONITORING OF COLD-SETTING ADHESIVE CURING IN WOOD LAMINATES: EFFECT OF CLAMPING PRESSURE AND DETECTION OF DEFECTIVE BONDS

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ABSTRACT

Many variables affecting adhesive bonding of wood, including moisture content, temperature, surface roughness and contamination, and wood density, are difficult to control and/or measure in industrial conditions. However, the combined effect of these factors may be compensated by controlling process variables, such as clamping pressure and time, and adhesive viscosity, concentration, and spread. This research project investigated an ultrasonic method as a nondestructive means of monitoring bonding processes and assessing the quality of the cured bonds in wood laminates. Monitoring was performed simultaneously at normal and angular (5° nominal) incidence to the bond plane, using pairs of clear Douglas-fir laminates with a single bond line. It was previously reported that ultrasonic transmission is sensitive to curing phases, such as spreading, penetration, curing, and bond thickness. This paper reports the effect of bond defects (uncured, underspread, and uneven spread) and clamping pressure on ultrasonic transmission. The results showed that defective bonds can be detected using patterns of relative attenuation changes during curing and an "unloading effect," measured as the relative transmission reduction after the clamping load is released. Also, transmission through uncured bond lines was strongly affected by pressure, an observation that can be utilized to select optimum clamping pressure.

Keywords: Ultrasonics, acousto-ultrasonics, adhesion, adhesives, laminates, glulam.

INTRODUCTION

Glulam is an adhesively-bonded wood product widely used for bridges, buildings, and other structural applications. One of the limits of its use and performance is the absence of an acceptable nondestructive method of assuring bond quality. Existing methods of glulam quality assurance are based on control of the bonding processes, visual inspection, and destructive tests of limited samples of adhesive bonds. These techniques may distort the perception of glulam bond strength, possibly causing a rejection of an acceptable product or acceptance of a defective one, neither of which alternative is desired.

The through-transmission ultrasonic method of glulam bond evaluation is sensitive to bond defects (Reis 1989; Reis et al. 1990; Biernacki and Beall 1993). However, the natural variability of wood, and grain direction and growth ring orientation have made it difficult

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to establish a reference value for normal bonds, masking the detection of defective bonds. More promising results were obtained when a modified ultrasonic method was used for the monitoring of curing of adhesive bonds (Biernacki and Beall 1995). The effects of the natural variability of wood were circumvented by using relative measurements of signal attenuation, where the transmission for the cured bond line was used as a reference signal. Monitoring was performed simultaneously at normal and nearly parallel to the bond line (5°) incidence using a single adhesive bond line between two clear Douglas-fir laminates. Angular incidence transmission provided greater sensitivity to bond quality and curing status than did normal incidence. The method was sensitive to curing phases (spread, penetration, and hardening) and was reasonably correlated with development of bond strength. Different ultrasonic curves were observed for thick (0.5 and 1.0 mm), normal (about 0.1 mm), and "kissing" bonds. (Bond thickness in this paper refers to the cohesive layer of the glueline, which does not include the distance of penetration of adhesive into adjacent wood cells; kissing bonds can occur when the adhesive is uncured.) Thick bonds caused a greater increase in transmission than did normal bonds, whereas kissing bonds showed the smallest signal increase. In angular transmission, thick-bond curing curves showed a characteristic inflection, a feature not occurring in any other bond type. In a related study (Beall and Biernacki 1993), transmission was successfully achieved through multiple laminates, demonstrating the potential for use with commercial glulam beams. The objective of this paper is to demonstrate the effect of pressure and certain types of bond defects on ultrasonic transmission through a laminate.

BACKGROUND

The transmission of elastic waves through an interface between two materials is achieved through molecular contact between those materials. Any interfacial porosities or disbonds increase reflections from such interfaces. A common technique to provide molecular continuity between two materials, such as a specimen and an ultrasonic transducer, is to apply a viscous liquid (couplant) to the interface. Molecular contact is therefore a common denominator of the quality of both adhesive bonds and acoustic transmission. However, intimate contact is a necessary but not a sufficient condition for achieving load-resisting capacities. For example, a viscous couplant provides molecular continuity between two materials but offers little load resistance. Certain types of defective bonds, kissing and partial, have particular influences on ultrasonic transmission.

Kissing bonds occur when two contacting materials are under compressive load, but otherwise without any load resistance. Kissing bonds can occur under conditions of both adhesive and lack of adhesive, simply from the compressive load that causes intimate contact of the adherends. Because the actual contact between adherends consists only of isolated islands of wood cells, high stresses at contact points cause elastic and plastic deformation and creep, resulting in a larger contact area. As a result of the friction from a compressive load, kissing bonds can support transverse motion and have substantial transverse stiffness, thereby transmitting both transverse and longitudinal waves. Very tight kissing bonds are therefore similar to perfect bonds, which makes them difficult to detect as long as the compressive load is applied. An important property of kissing bonds is a strong dependence of interfacial stiffness on compressional pressure, which may cause wave transmission to increase by several orders of magnitude with pressure (Nagy 1992). Therefore, an increase in pressure causes an increase in wave transmission, whereas removing that load would lead to a reduction of transmission. Both effects may be utilized to control and assess bonding of wood, which was explored in this project. A special type of kissing bond occurs during the initial stages of curing, where an uncured adhesive acts as an acoustic couplant. This type of kissing bond may be formed at nominal pressures on the order of 1 MPa, which

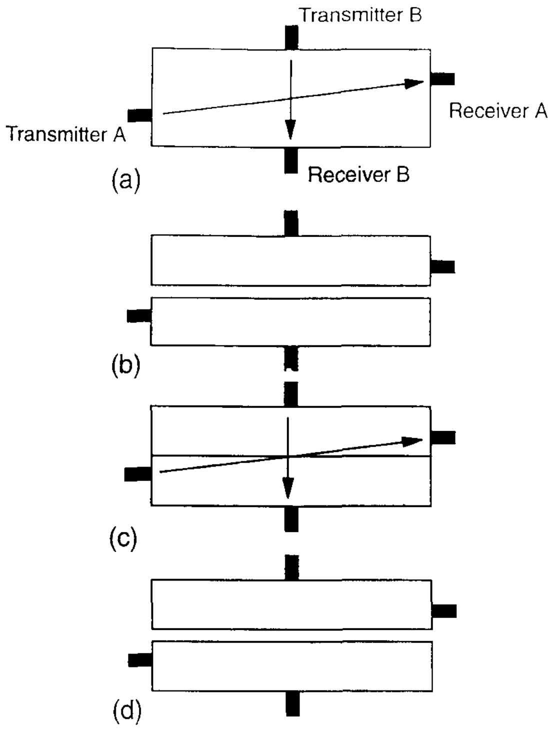

FIG. 1. Specimens with attached transducers in different testing phases: (a) transmission through a solid specimen without an adhesive bond line, (b) the specimen after sawing and planing, (c) transmission through the laminated specimen with an adhesive bond line, and (d) the resawn specimen after the first curing monitoring test, prepared for the next testing cycle.

is typical for wood-bonding processes. Because of the viscous nature of this type of interface, both loading and unloading cause time-dependent effects. Another type of kissing bond may also occur in a partial bond under a compressive normal load.

Partial bonds are adhesively-bonded interfaces that contain many small unbonded areas, but have measurable bond strength. The combined effect of unbonded areas and interfacial porosities causes an ultrasonic wave reflection from the entire interface, and therefore results in lower transmission than for normal bonds (Nagy 1992). Application of a normal pressure to an uncured bond may cause a filling of many of those areas, increasing wave transmission. Because of the elastic nature of partial bond interfaces, they are not expected to have a time-dependent response upon application or removal of load. Partial bonds can be caused by either uneven spread or underspread.

EXPERIMENTAL APPROACH

The effect of clamping pressure and bond quality on elastic wave propagation was studied by using ultrasonic transmission. Douglas-fir specimens, 100 × 200 × 600 (longitudinal) mm, were equilibrated at 12% equilibrium moisture content (EMC) for 3 months. Testing was performed in a configuration and a sequence shown in Fig. 1. To obtain reference signals, ultrasonic transmission was measured for each specimen before the interface was created (Fig. 1a). The specimens were sawn longitudinally, planed on the newly-exposed surfaces, and adhesively bonded with resorcinol (DAP Inc., 60290 resin/60291 catalyst), as shown in Fig. 1b and c. This procedure also reduced reflections caused by acoustic impedance mismatch, and therefore increased sensitivity to bond quality. Monitoring was performed starting at specimen clamping time until a fully cured bond was developed (nominally 22 h) at 25°C and 66% relative humidity (12% EMC). Next, the laminated specimen was resawn through the adhesive bond and planed in preparation for the next series of experiments. This procedure was repeated three times with the transducers remaining attached. Fig-

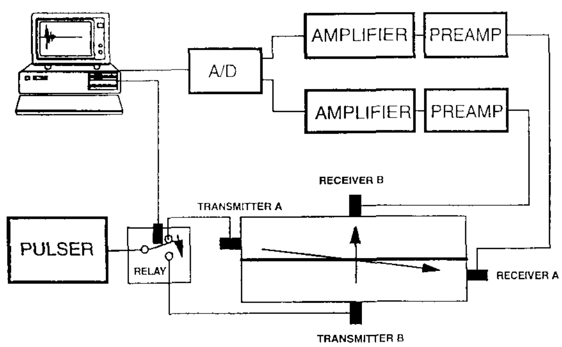

FIG. 2. Curing monitor signal flowchart with a nominal specimen.

ure 2 shows a signal flowchart, instrumentation, and a nominal specimen for the system used to monitor the curing process. A high-voltage spike pulse (200 V) was created by the pulser (Tektronix TM503) with a repetition rate of 100 Hz, and was sent to one of the transmitting piezoelectric transducers (AET AC175L, 175 kHz, 19-mm diameter), which had been bonded to the specimen with a hot-melt adhesive. The transmitted waves were captured by a receiving wideband transducer (AET FAC 550; 30-mm diameter). Waves were transmitted at angular (nominal 5°) and normal transmission using two transmitter/receiver pairs, as shown in Fig. 2. A PC-controlled relay was used to switch the input signal between the two transmitting transducers. The signals were then amplified in preamplifiers (AET 140B, wide bandpass, 40 dB gain) and amplifiers (AET 204B, 0 to 40 dB), and digitized with a 2-channel, 8-bit A/D board, Sonix STR*832, with a sampling rate of 2 MHz. Signals of 512 μs length were averaged 256 times and stored for further analysis. Transducers were attached to the specimen with a hot-melt adhesive and remained in fixed positions throughout the testing sequence.

The types of adhesive bonds studied included (a) normal (catalyzed resorcinol)—with a spread of 400 g/m^2 and an initial viscosity of 5.2 Pa·s; (b) kissing—resorcinol without catalyst, adding 30% calcium carbonate powder by mass to match the initial viscosity of resorcinol having catalyst; (c) uneven spread—adhesive at normal spread in two strips on the adherend as shown in Fig. 3; and (d) underspread—50 g/m^2. Previous work (Biernacki and Beall 1993) had shown that for very flat machined laminates, the spread had to be reduced to this level before bond weakness was significant. In commercial practice, underspread of this magnitude can easily occur at local areas of the bond line because of unevenness of the laminates caused by planing variations. All spreading was done by hand, and the pressure applied for all tests was 1 MPa. Each bond type was replicated three times to ensure consistency of data. After completion of the monitoring sequence, cured adhesive bonds were tested in shear (ASTM D 905-89).

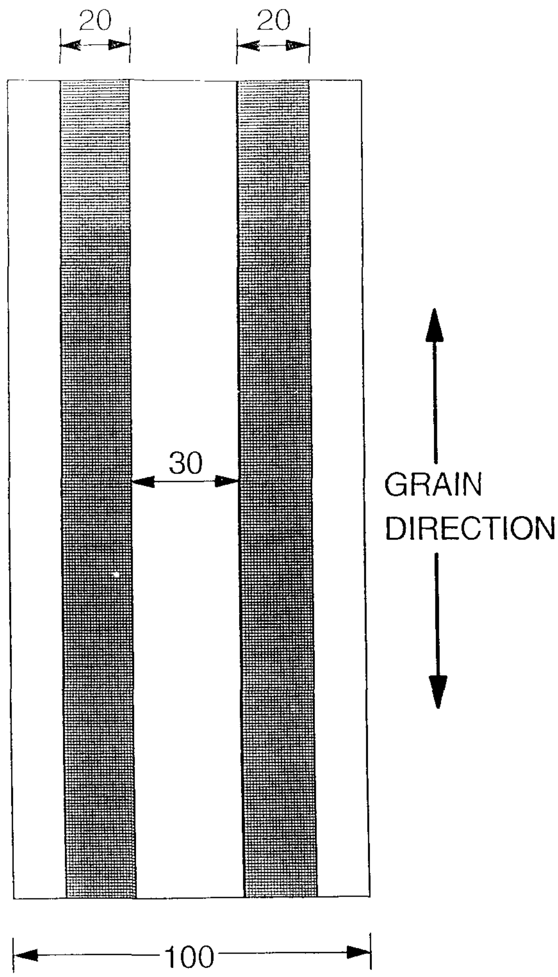

FIG. 3. Uneven bond line spread, showing the adhesive in shadowed areas (top view of the bond line plane) (mm).

RESULTS AND DISCUSSION

The results of curing are presented in terms of coefficient of transmission (CT) as a function of pressure and curing time. Two types of CT were used: (a) relative CT (CT_{rel}) calculated as a ratio of RMS amplitude of the measured signal to that of the cured state (after 22 h), and (b) absolute CT (CT_{abs}) calculated as a ratio of RMS amplitude of the measured signal to the signal through the original unsawn specimen.

TABLE 1. *Curing curve parameters and shear strength of normal and defective bonds.*

Bond type	Total increase of transmission (CT_{tot}^A)		Absolute coefficient of transmission (CT_{abs}^A)		Unloading coefficient (UC)		Shear strength (MPa)	
	Mean	Standard dev.	Mean	Standard dev.	Mean	Standard dev.	Mean	Standard dev.
Normal	0.362	0.151	1.12	0.010	0.00	0.008	10.50	1.42
Underspread	0.096	0.070	0.876	0.055	0.109	0.058	3.73	1.52
Uneven	0.220	0.066	0.901	0.034	0.201	0.151	5.07	0.923
Kissing	0.087	0.055	1.02	0.014	0.454	0.264	0	0

Influence of defective bonds on transmission

Ultrasonic transmission curves were analyzed for the four types of bonds, including the effect of removing the clamping force on transmission, referred to as an "unloading effect." Defective bonds had significant shear strength reductions from controls, as shown in Table 1, averaging 50 and 30% for uneven and underspread bonds, respectively. Kissing bonds were assumed to have no strength because of the absence of catalyst.

Typical curing curves are shown in Fig. 4. The angular transmission had a larger increase and a later equilibration than that of normal transmission. The upper curve in Fig. 4 is for a normal bond line, which was also typical for uneven bond lines. The lower curve was typical for kissing and underspread bond lines, with smaller increases in angular CT_{rel} (CT_{rel}^A). This curve may be quantified as the total transmission increase (CT_{tot}^A) defined by:

$$CT_{tot}^A = 1 - CT_{rel}^A\ (t = 0)$$

as given in Table 1. For underspread bond lines, this effect may be explained by a sequence of events, beginning with a rapid diffusion of solvent from the thin layer of adhesive, causing an increase in adhesive viscosity and concentration of the adhesive reactants. The increased reactant concentration caused a faster condensation rate, further advancing the viscosity. This premature hardening caused low penetration and inadequate flow, causing a porous bond and therefore a low CT_{tot}^A.

Although kissing bonds gave similar curing curves and CT_{tot}^A as underspread bond lines, different phenomena in the bond line may explain this behavior. Because the adhesive lacked a catalyst, the viscosity increase was slower since it was caused by only the diffusion of solvent, leading to a premature penetration and possibly starved bond lines. Because solidification did not occur, the CT_{tot}^A was small because wave transmission was affected only by flow and penetration. Kissing bonds also had a large CT_{abs} (1.02), as shown in Table 1, confirming the findings of other researchers that very tight kissing bonds cause small acoustic contrast (high transmission). Nevertheless, CT_{abs}^A for kissing bonds was lower than CT_{abs}^A for normal bonds, which had a value of 1.12. (A value greater than unity is an artifact of the lower angle of transmission for angular incidence caused by removal of material from resawing as shown in Fig. 1. Since

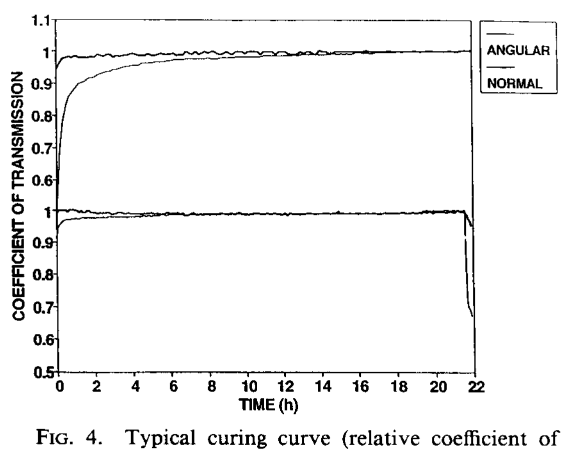

FIG. 4. Typical curing curve (relative coefficient of transmission vs. curing time) for normal bonds (upper curves) and kissing bonds (lower curves).

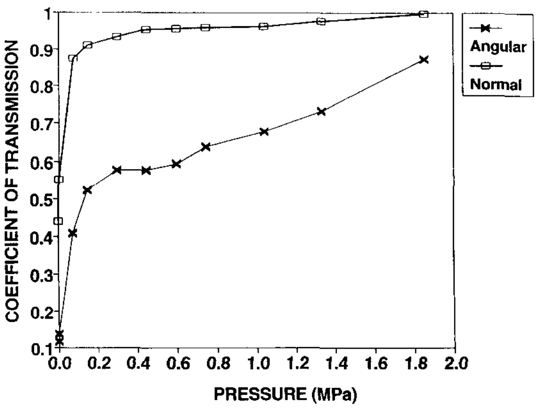

FIG. 5. Effect of pressure on relative coefficient of transmission (CT_{rel}) for angular and normal transmission for a kissing bond.

the wave energy propagated closer to the grain orientation, where the attenuation is lowest, signal transmission was increased.). As a result of relatively large CT_{abs}^{A}, kissing bonds were somewhat difficult to distinguish from normal bonds based on acoustic transmission alone. In contrast, CT_{abs}^{A} had substantially lower values for underspread (0.876) and uneven spread (0.901) than the values for normal bonds.

Pressure effect on transmission

As mentioned previously, increased pressure was expected to increase wave transmission. A strong effect of pressure on CT_{rel} is shown in Fig. 5 for a specimen with a kissing (uncured) bond. Note that for normal transmission, CT_{rel} saturated at about 0.5 MPa, but angular transmission reached only a slight plateau and then continued to increase linearly with pressure. These trends were confirmed in a replication. One possible explanation of the angular transmission effect is that the small plateau is due to saturation of the latewood-to-latewood contact areas, which achieved intimate contact at a lower stress level. In addition, wood planing produces a surface with latewood at peaks and earlywood at valleys of the surface profile, causing latewood-to-latewood and latewood-to-earlywood areas to meet first and restrain the contact of earlywood-to-earlywood areas. As the pressure increased above the 0.5 MPa plateau, the latewood contact areas were further compressed, allowing contact of earlywood-to-earlywood, causing a further increase in wave transmission.

Unloading effect.—A common feature of the defective bond line curing curves was a drop of CT when the clamping load was removed at the end of curing, referred to here as an "unloading effect," as shown in Fig. 4b for a kissing bond. The unloading effect was measured with unloading coefficient (UC) defined as:

$$UC = 1 - CT_{rel}^{A}(t_{unl})$$

where $CT_{rel}^{A}(t_{unl})$ is CT_{rel}^{A} after pressure release. The unloading coefficient for normal and defective bonds is given in Table 1. Note that there was no unloading effect ($UC \approx 0$) for normal bonds, but there was a measurable unloading effect for underspread, uneven, and kissing bonds, in order of the magnitude of the effect. The unloading effect may be explained by actual separation of small "kissing contact" areas caused by removing the clamping pressure. After the normal compressive load was removed, compressed peaks of the surface expanded elastically, shifting tensile stresses to other areas within the bond line, causing a sep-

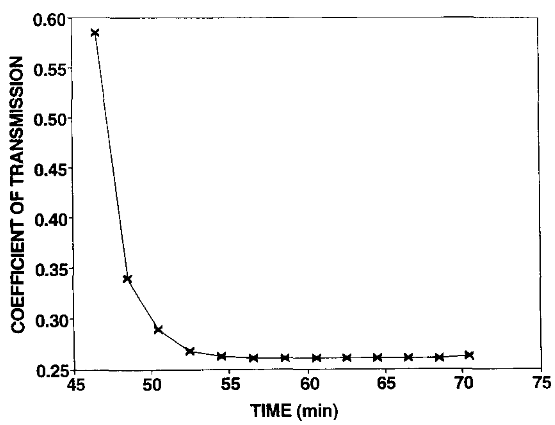

FIG. 6. Unloading effect for kissing bonds [relative coefficient of transmission for angular incidence (CT_{rel}^{A}) vs. curing time], showing time-dependent behavior after removal of the clamping load. Time t = 0 corresponds to the start of loading.

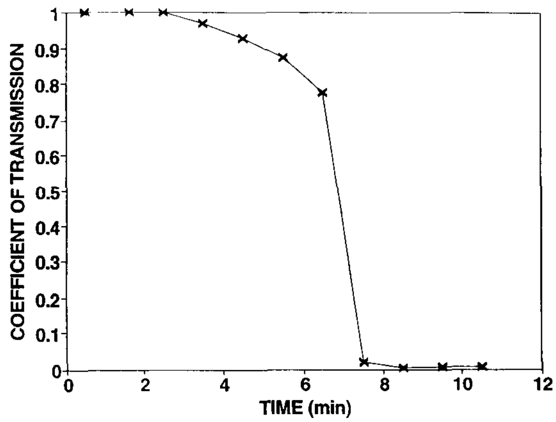

FIG. 7. Relative coefficient of transmission for angular incidence (CT_{rel}^A) for a kissing bond under tension, showing time-dependent behavior and abrupt drop of CT_{rel}^A caused by the bond separation.

aration of some small regions that were not adhesively bonded and a subsequent reduction in wave transmission.

Another interesting effect was observed after unloading the kissing bond lines and monitoring transmission at small time intervals (Fig. 6). Unlike other defects, such as underspread and uneven bonds, where CT_{rel}^A decreased immediately at unloading, the kissing bond gave a clearly time-dependent curve. Obviously, this effect is related to the viscosity of the uncured resorcinol. Because the detection of uncured bond lines is of great commercial importance for glulam manufacturing, analysis of unloading phenomena and its time dependency may be used for detection and evaluation of uncured bond lines, as well as other bond defects.

Further analysis of the curve in Fig. 6 showed that long after unloading the kissing bond laminates, there was some significant transmission ($CT_{rel}^A = 0.26$). In fact, the bond had some strength since it could not be opened by manual shearing. Two factors could contribute to this effect: (a) development of weak bonding because of adhesive drying, and (b) work of thermodynamic adhesion (work of separation), which must be added to the system to create new surfaces (Houwink and Salomon 1965). To further demonstrate the unloading effect, tension was applied to separate the bond. Figure 7 shows the result of such an experiment for a kissing bond, where a small tensile stress was introduced by suspending the specimen, allowing gravitational force (approximately 35 N) of the lower part of the specimen to act on the bond. The acceleration of disbonding with time, shown as a decreasing CT_{rel}^A rate, could be explained by the reduction of the effective contact area, which led to a concentration of stresses on the remaining areas of contact, causing an even more rapid separation. This process progressed to a catastrophic bond failure, which is shown in this figure as an abrupt change in CT_{rel}^A followed by a constant and nearly zero value.

CONCLUSIONS

The effect of clamping pressure and defective bond lines on ultrasonic transmission was studied experimentally. Transmission was strongly affected by the clamping pressure. The coefficient of transmission (CT) vs. pressure relationship gave an initially high rate of CT increase, then local saturation, followed by further increase. This behavior can be explained by wood surface topography, and the effect of compression of earlywood-earlywood and latewood-latewood contact areas.

Adhesive defects, including kissing, underspread, and uneven bond lines, were detectable by analyzing ultrasonic transmission curves and an "unloading effect," a reduction in coefficient of transmission after the clamping load was removed. Kissing and underspread bond types gave a smaller total CT increase than normal bonds, which also may be used to distinguish these defects. Furthermore, strength testing of the defective bonds gave 50 and 40% mean strength reductions for uneven bonds and underspread bonds, respectively, relative to normal bonds.

The removal of the clamping load for an uncured bond line caused time-dependent behavior in transmission. A similar time-dependency was also observed when a small tensile load was applied to the kissing bond. These effects could be utilized to identify undercured and kissing bonds.

Results of this work and the previous study (Biernacki and Beall 1995) could be utilized to develop a glulam monitoring system that could evaluate several curing parameters important in the manufacturing process:

(a) *Completeness of curing.* Identifying the optimum unclamping time would reduce total processing time and reduce the risk of bond damage due to premature release of pressure.
(b) *Bond line quality.* The information from curing curves and the unloading effect could be used to evaluate bond quality.
(c) *Clamping pressure.* The optimal clamping pressure could be found by monitoring CT changes during clamping.

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