

Wood Processing Variables and Adhesive Joint Performance

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Synopsis

In order to obtain a bonded wood product from the tree, wood must be subjected to a series of processes. These include, among others, vital processes like drying of the green wood and machining of the surfaces of the dried wood in preparation for bonding. However, when wood is machined with dull blades, thermal degradation of the wood frequently occurs. The effects of kiln drying and thermal degradation of wood on the performance of the adhesive joints subsequently formed from such wood were investigated. Tapered double cantilever beam (TDCB) test specimens obtained from hard maple and phenol-resorcinol-formaldehyde (PRF) adhesives were used in the investigations. The enhancement of fracture energy due to the reduction in the moisture content of wood, the improved performance of adhesive joints due to rejoining of wood surfaces before bonding, and the reduction of adhesive joint strength resulting from thermal degradation of wood are presented and discussed.

INTRODUCTION

Adhesives are, by definition, substances capable of fastening materials together by surface attachment. Therefore, the success or failure of an adhesive in the performance of this function depends very strongly on its ability to adhere to the materials being bonded. The success of the adhesion process itself is dictated partly but significantly by the topography of the adherends. Wood surface topography is a strong function of the processing variables. Going from the tree to the bonded wood product involves a number of processes. These include, among others, such vital factors as drying of the green wood, machining of the surfaces in preparation for bonding, and storage before bonding. We have previously reported the effects of surface roughness, the nature of surface roughness, and aging of machined surface (by storage) on adhesive joint fracture energy.^{1,2} In this paper we present results of studies on the effects of kiln drying and thermal aging of wood on adhesive joint performance.

ADHESIVE JOINT STRENGTH MEASUREMENT—THE FRACTURE MECHANICS APPROACH

Fracture mechanics is a relatively new discipline which relates the fracturing behavior of flawed bodies to applied loads. Fracture mechanics is a direct outgrowth of the Griffith theory which postulates that the lower-

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than-ideal fracture strength of all real bodies is due to the presence of initial flaws or cracks. These may be dust particles, bubbles, or nonbonded areas, in the case of adhesive joints. Failure usually occurs by a propagation of the largest of these cracks. Using the concepts of fracture mechanics, one defines fracture toughness which determines the load-bearing capacity of a structure in the presence of flaws.

The stress field in the vicinity of a crack tip can be adequately defined by the parameter K , the stress intensity factor. This parameter, a function of applied load and crack size, increases to a critical value $K = K_c$, whereupon a previously stationary or slow-moving crack propagates abruptly. This critical value K_c defines the fracture toughness. For adhesive joints, the analysis required to describe the stress field at the crack tip is extremely difficult. Consequently, fracture toughness is defined in terms of energy by exploiting the relationship between K and the strain-energy release rate G . G is related to K by the equations

$$\begin{aligned} G &= (K/E)(1 - \nu^2) && \text{for plane strain} \\ G &= K^2/E && \text{for plane stress} \\ E &= \text{Young's modulus} && (1) \\ \nu &= \text{Poisson's ratio} \\ G &= \text{a physical measure of the rate of release of} \\ &\quad \text{strain energy at the crack tip} \end{aligned}$$

For mode I or opening mode, which is the mode of failure of the vast majority of engineering materials, a cleavage stress field surrounds the crack tip. In this case $K_c = K_{Ic}$ and hence $G_c = G_{Ic}$. Mostovoy et al.^{3,4} and Ripling et al.⁵⁻⁷ have developed and applied the tapered double cantilever beam (TDCB) geometry to a number of composite systems using metal adherends. We have adapted this for use in wood-based adhesive systems. Details of this have been given elsewhere.^{1,2} Using the TDCB, the fracture energy is given by the equation

$$G_{Ic} = \frac{4P_c^2}{Eb^2} m \quad (2)$$

where P_c = critical load necessary to cause a previously stationary or slow-moving crack to propagate abruptly, b = width of beam, m = a constant chosen so that compliance changes linearly with crack length [$m = 0.52 \text{ cm}^{-1}$ (1.33 in.^{-1}) for all specimens in this study], and E = bending modulus of adherends. At P_c , the crack propagates until enough energy is released to bring the crack to rest.

Adherends

A hard maple log (*Acer saccharum*, Marsh) (green but not freshly cut) was sawed into boards. Specimens slightly greater in dimensions than the actual test specimen (specimen geometry $m = 0.52 \text{ cm}^{-1}$) were cut from the boards. Because of the dimensions of the boards, only specimens with

fiber orientation of 10° to the surface to be bonded could be cut from the boards. The surfaces to be bonded were jointed (machined) and then the specimens were placed in a dry kiln and dried under standard hard maple drying schedule shown in Table I. Starting from the green wood, specimens were removed periodically from the kiln, stored overnight in thick polyethylene bags, cut to the final dimensions and bonded. In some cases, the surfaces to be bonded were rejointed before bonding. In most cases, however, the specimens were bonded without further machining of the surfaces to be bonded.

A preliminary differential scanning calorimetric (DSC) study of wood using sealed pans was conducted to determine the regimes of thermal activity of wood (Fig. 1). The endothermic peak from 90 to 230°C obviously corresponds to evolution of volatiles while the exothermic peak, which starts at about 250°C, corresponds to thermal degradation of wood. Thermal degradation of wood occurs when wood is machined with dull blades. To simulate this situation, the last specimens removed from the kiln with moisture content of about 8% were stored overnight in thick polyethylene bags and cut to the final dimensions of test specimens, including rejointing the surfaces to be bonded. The specimens were then subjected to a 250°C thermal treatment for various lengths of time. On removal from the oven, the specimens were stored in polyethylene bags for 24 h and then bonded without further moisture reconditioning.

Adhesives

The adhesive was Koppers Penacolite G4411 phenol-resorcinol resin mixed 10 parts per hundred resin (phr) Celanese Co. paraformaldehyde powder.

Bonding and Fracture Testing

The adhesive was mixed vigorously by hand for 10 min and then hand-brushed as uniformly as possible on both surfaces to be mated (both beams). Open assembly time was less than 60 s. Beams of about the same density were mated, and a small Mylar or Teflon film of about 2.54×10^{-2} mm thickness was inserted at the jaw of the specimen to provide the initial flaw. The closed assembly time was 30 s. Glueline pressure 1.03–1.17 MPa

TABLE I
Kiln Drying Schedule for Hard Maple

Moisture ^a content (%)	Dry bulb temperature (°F)	Wet-bulb depression (°F)	Wet-bulb temperature (°F)
> 40	130	5	125
40–35	130	7	123
30–25	140	19	121
25–20	150	30	120
20–15	160	50	110
15–0	180	50	130

^a Kiln operating conditions are changed according to the moisture content of samples.

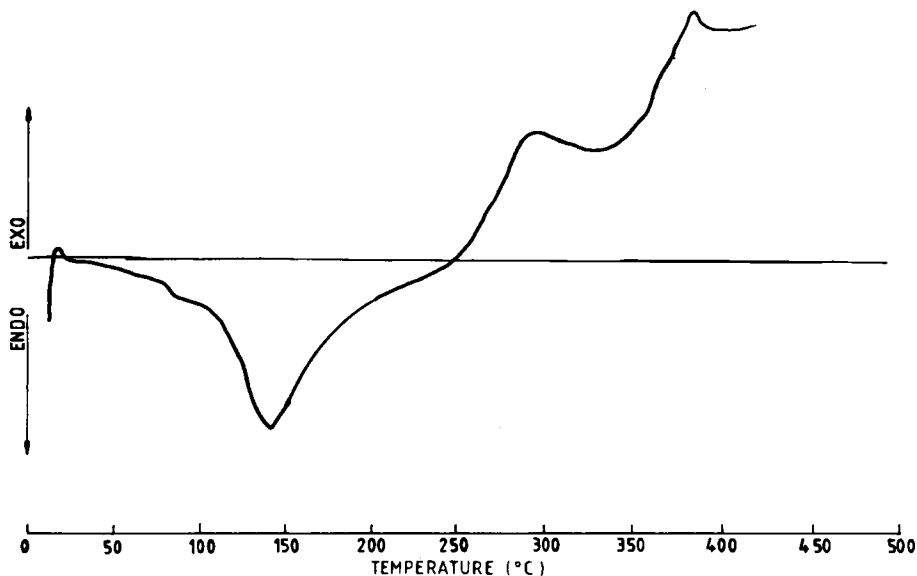


Fig. 1. DSC thermogram showing regimes of thermal activity of wood. Note the reaction exotherm starting at about 250°C, indicating onset of thermal degradation of wood.

was measured by a precalibrated compressometer (Fig. 2). Before curing, excess adhesive was cleaned from the joint with a knife. The test specimens, held in the press (Fig. 2), were placed in a forced-air oven maintained at 85°C (\pm 1°C) and cured for 4 h. Generally the applied glueline pressure dropped considerably by the time the specimens were removed from the oven due to the combined effects of metal expansion and wood drying. Specimens were fractured, without reconditioning to the original 8% moisture content, in a universal test machine (Fig. 3).

RESULTS

The moisture contents of the test specimens at the time of fracture tests (i.e., after oven cure of adhesive joints) was determined by standard ASTM method (ASTM D 2016). In addition, after fracture, appropriate ($151 \times 33.4 \times 10.8$ mm) beams were cut from the specimens and the bending moduli determined. The enhancement of bending modulus with reduction in moisture content is shown in Figure 4. Fracture energies were calculated using these modulus values obtained from the fractured test specimens. The effect of kiln drying on fracture energy is shown in Figures 5 and 6. It is important to point out here that all the specimens in these tests failed catastrophically. This means that cracks, once initiated, propagated through the entire specimen without a single arrest. This failure characteristic was unique to this particular set of experiments. In previous experiments^{1,2} cracks were initiated and arrested several times along the length of the test specimens, resulting in a saw-toothed load-deflection diagram. It must be noted, however, that fracture energies were much higher in this study than in previous studies.

Figure 5 shows the variation of fracture energy with the period of kiln drying. Specimens which were dried for the same length of time but re-

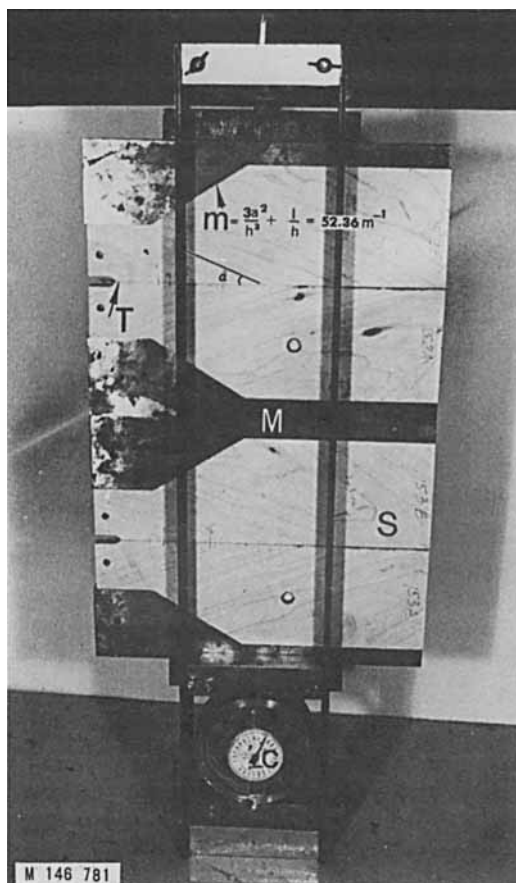


Fig. 2. Press for holding specimens (two simultaneously) during cure. Note specimen (S), metal spacers (M), Teflon tape (T), compressometer (C), and wood fiber orientation to the bonded surface (α).

jointed before gluing had superior fracture performance relative to those which were bonded without rejoining. Figure 6 shows how fracture energy increased with a drop in the moisture content of wood. Observe in particular that joints derived from green wood had essentially zero fracture energy. Specimens which were made from green wood broke apart while stored in polyethylene bags to cool down to room temperature after cure. Figure 7 illustrates the decrease in fracture energy with thermal degradation of the wood surface. Most of the specimens subjected to the 250°C thermal treatment after kiln drying failed in the wood. Failure was generally located in the wood a fraction of a millimeter (mm) below the glueline. The depth of this location from the glueline generally increased with increasing time of specimen exposure at 250°C.

DISCUSSION

In contrast to all our previous studies,^{1,2} specimens from the drying kiln experiments failed catastrophically at the crack initiation loads. One or more of the following factors were responsible for the extreme brittleness

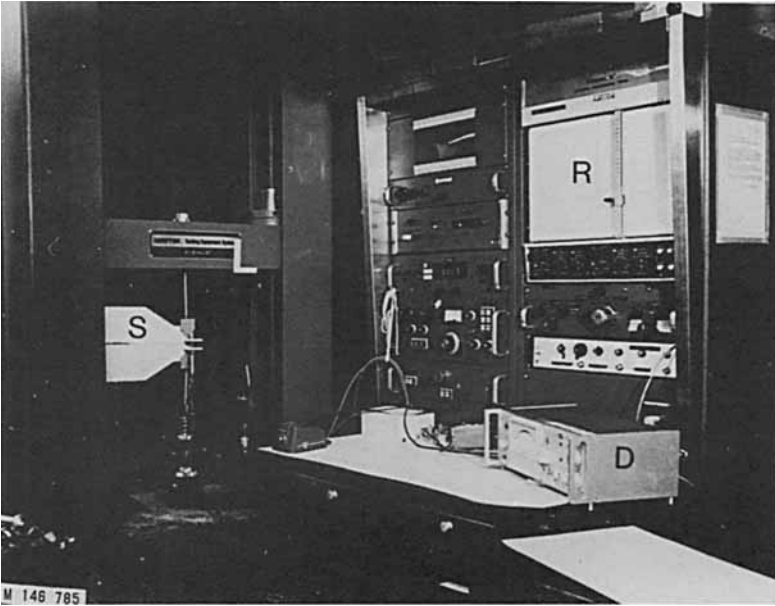


Fig. 3. Arrangement for fracture testing in a Riehle universal test machine, Daytonie signal conditioner (D), and Hewlett Packard X-Y recorder (R).

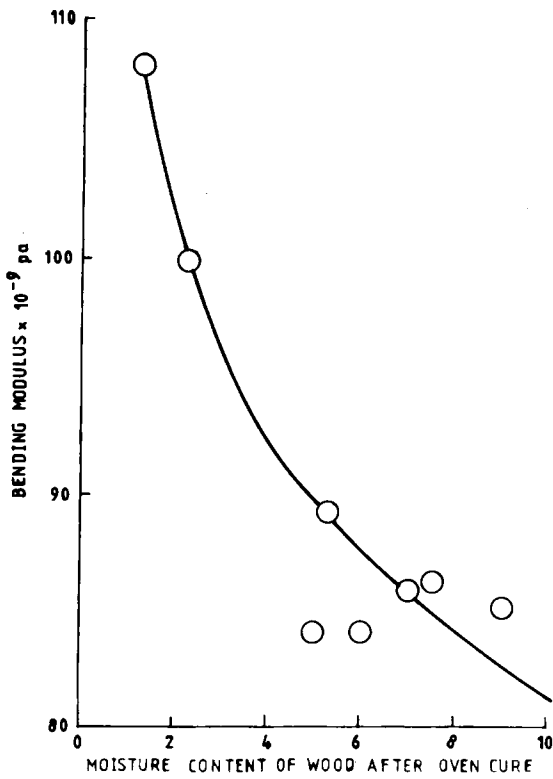


Fig. 4. Enhancement of bending modulus with reduction in the moisture content of wood.

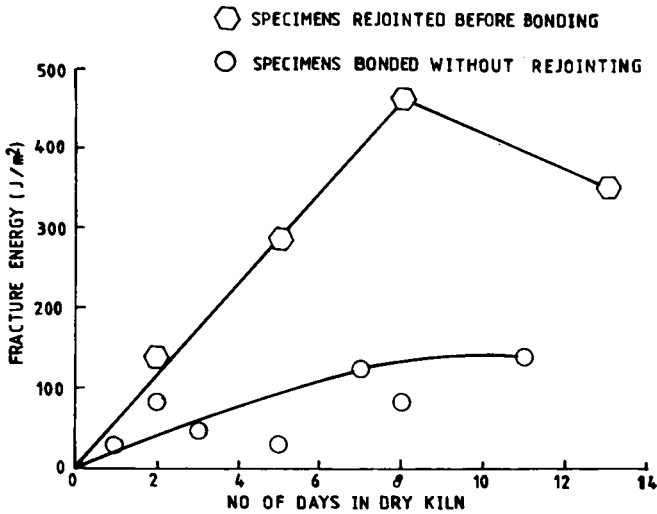


Fig. 5. Change of fracture energy with kiln drying time for hard maple. Observe that specimens which were rejointed before bonding (○) had better strength performance than those which were bonded without rejointing (○).

of these joints:

1. Unevenness of bonded surface.
2. Steam blisters or bubbles.
3. Accelerated cure.
4. Plasticity of adhesive layer.
5. Wood degradation and other wood-related properties.

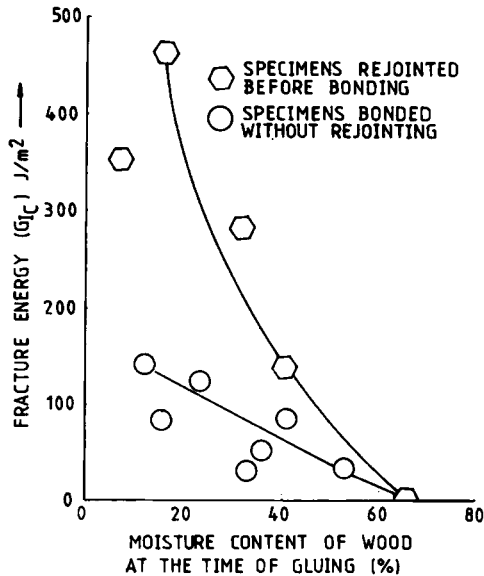


Fig. 6. Variation of fracture energy with moisture content of wood at the time of gluing. Observe the increase in fracture energy with decreasing moisture content. Observe also that joints from fresh surfaces had higher fracture energies than those from kiln dried surfaces, which were bonded without resurfacing. (○) Specimens rejointed before bonding; (○) specimens bonded without rejointed.

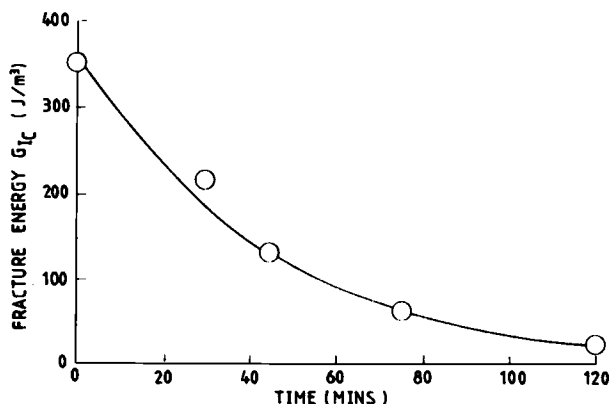


Fig. 7. Reduction of strength properties following thermal aging (dry heat). After kiln drying, specimens were exposed to 250°C for various lengths of time before bonding.

For the best adhesion between two surfaces, a reasonable degree of intimate contact is imperative. Experience has shown that jointing yields a surface sufficiently smooth to provide adequate surface contact for good adhesive bond formation. Uneven surfaces produce unbonded areas and a glue-line of extremely variable thickness—factors which accentuate susceptibility to catastrophic failure (1). But, since catastrophic failure was exhibited by specimens derived from both rejoined and unrejoined surfaces, unevenness of bonded surfaces does not provide the entire explanation for the failure characteristics shown by the specimens.

Evolution of steam from the wood during cure could conceivably lead to steam blisters and microscopic voids in the adhesive layer that would effectively reduce it to a brittle but rigid sponge. This situation could easily lead to catastrophic failure of joints. However, at a cure temperature of 85°C, the probability of such a phenomenon would be unlikely. Besides, strength reduction due to such process would have been more evident in specimens with higher wood moisture content. But this was not the case.

During cure, the evolution of volatile products, particularly wood extractives, could accelerate cure rate and also affect fracture energy. Here, again, differences would have been observed between specimens derived from wood with high moisture content and those obtained from wood with low moisture content. These differences were not observed.

In previous experiments,^{1,2} we generally reconditioned the specimens to EMC 8%. However, in this set of experiments, after cure, specimens were cooled down to room temperature (while still stored in thick polyethylene bags) and fractured without reconditioning. We have observed from gel permeation chromatograms that adhesives cured at 85°C for 1 h, then exposed to a humid atmosphere do absorb some moisture⁸: An additional negative peak appears in the chromatogram; this peak has been attributed to water by Quinn et al.⁹ It is thus possible that reconditioning of specimens may lead to the inhibition of water by the adhesive layer. If this happens, a certain degree of adhesive plasticization will occur when specimens are reconditioned to 8% wood moisture content, and this could enhance the crack arrest capability. Note that these specimens were cured at 85°C for

4 h. Obviously this would reduce the tendency for adhesive plasticization. But, to assess this possibility, we made joints from the same stock of wood which had been used in the experiments where we observed several crack arrests before failure. The specimens were fractured without reconditioning. Here, as in all the other cases, propagating cracks were arrested several times before ultimate failure. The number of crack arrests were, however, generally fewer.

It thus appears that some other wood-related property may have been largely responsible for the observed catastrophic failure of specimens. In our previous report,¹ we showed that specimens where the orientation of the fibers with respect to the bonded surface was 10° failed at higher fracture loads than those whose orientation was 20°. The bulk of our previous work was conducted with specimens whose grain angle was 20°. In the present set of experiments, however, the grain angle was 10°. It is possible that this inherent effect of wood anisotropy on fracture energy coupled with the long cure time resulted in joint with such high initiation loads that a propagating crack could not be arrested.

Our results indicate that moisture content of wood affects not only the mechanical response of wood (Fig. 4), but also the fracture strength of the adhesive joint (Fig. 5). Wood moisture content induces or contributes to other wood-related factors affecting adhesive joint strength. One factor is the effect of wood moisture on adhesive viscosity. High wood moisture content often leads to starved joints. In making specimens, it was observed that, even after 30 min of closed assembly time, the adhesive squeezed out of the joints on the application of pressure was still quite fluid. Further evidence of joint starvation is the fact that the fractured surfaces of these joints had a "washed-out" appearance. As should be expected, the average bondline thickness for these specimens was about 70–80 μm , which incidentally falls in the region where fracture energy drops drastically with bondline thickness.¹ Secondly, low adhesive viscosity induced by the high moisture content of wood may have also reduced the reactivity of the adhesive—i.e., retarded adhesive cure. Evidence for such reduction in strength properties due to low initial adhesive viscosity has been presented by Ebeuele.⁸ Lastly, excessive shrinkage of the wood at high moisture content imposes serious stresses on the bondline at an early stage during cure (before the adhesive develops full strength). These factors in the extreme are presumably responsible for the almost zero fracture energy of the adhesive joints made from green wood.

The consistently higher fracture energies of the rejoined surfaces relative to the surfaces bonded without rejoining indicate that the surface conditions of wood, both physical and chemical, have an even greater effect on joint performance than wood moisture content. Kiln drying of wood could have the following effects:

1. Uneven surfaces: Warpage during drying results in improper surface contact between adherends, leaving pockets of unbonded areas along the bondline and extremes of adhesive layer thickness.

2. Concentration or deposition of a thin film of wood extractives on the surface. This film may present both physical and chemical barriers to subsequent bonding operations and cure of the adhesive.

3. Excessive drying may cause cracks and checks to develop on the surface. In extreme cases, the bonding functionalities on the wood surface will be destroyed—a situation normally called “case-hardening.”¹⁰ These factors undoubtedly detract from the attainment of optimum joint performance.

The darkening of the wood surface and the exotherm peak observed in the DSC thermogram confirm that wood exposed to 250°C for various lengths of time is thermally degraded. Considerable attention had been devoted to the thermal degradation of wood.¹¹⁻¹³ There is a general consensus that decomposition of the carbohydrate component of wood (specifically hemicellulose, the least stable component) results in severe loss of mechanical properties and a reduction in bondability of wood. In our tests, the specimens exhibited wood failure just below the limit of adhesive penetration, and the severity of wood failure increased with exposure time. This agrees with the well-known fact that thermal aging weakens wood but because the adhesive bond was stronger than the wood, we failed to reveal an effect of thermal aging on the bondability of wood surfaces.

CONCLUSIONS

1. There is an optimum wood moisture content during bonding for the highest joint fracture toughness.
2. Surfaces rejoined prior to bonding producing joints with far better fracture toughness than those bonded without rejoining.
3. Thermal aging (dry heat) significantly decreases adhesive joint performance by reducing wood fracture toughness.

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