FUNDAMENTAL ASPECTS OF WOOD DEFORMATION PERTAINING TO MANUFACTURE OF WOOD-BASED COMPOSITES

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ABSTRACT

During processing, wood-based composites are pressed using extreme heat and pressure for varying lengths of time. Evidence exists that the environmental conditions under which the wood densifies can alter the properties of both the solid wood and the composite product. Given the larger number and extreme nature of variables that exist during composite manufacture, it is imperative that the deformation process be understood from a fundamental standpoint. The objective of this research was to determine the applicability of basic materials engineering theory to the viscoelastic deformation of wood in transverse compression under a variety of temperatures and moisture contents.

Theories of cellular solids were used to model the nonlinear compression behavior of small wood elements. For low-density woods, it was determined that cellular collapse can result from elastic buckling of the cell wall. The dependence of inelastic behavior of the gross wood on the elastic properties of the cell wall allows the time, temperature, and moisture dependence to be modeled with classical linear viscoelastic theory of amorphous polymers. Time-temperature-moisture superposition was shown to be applicable to stress relaxation data collected for temperatures between 39 and 99 C and moisture contents between 3 and 16%. The shift factors derived were described using free volume and entropy-based equations. This research demonstrates that wood behaves similarly under those conditions to the general class of cellular amorphous polymers. This conclusion opens many possibilities for experimentally and mathematically modeling the pressing of wood-based composites.

Keywords: Viscoelasticity, cellular metering, free volume, composites pressing.

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INTRODUCTION

One advantage of composites over traditional materials is the ability to tailor the material properties to meet structural design requirements. The properties of the composite will be a function of the raw material properties in situ and the geometry in which the components are arranged. Scientists and engineers have been more successful in designing synthetic fiber-reinforced composites than wood-based composites, mainly because of the microstructural complexity and variability of the latter. However, proficiency in designing wood-based composites is necessary if these products are to compete with other materials in future markets. The research presented here is aimed at improving our fundamental understanding of material behavior during the processing of wood-based composites. This improvement can be accomplished through mathematical and experimental modeling of the pressing cycle. These models can be useful as a tool to understand complex behavior that is not immediately intuitive.

For many non-veneered wood-based composites, the focal point of manufacturing is a highly automated process in which wood and adhesive components are flat-pressed, using extreme heat and pressure. During pressing, a simultaneous heat and mass transfer results in transient temperature and humidity gradients in the mat (Suchsland 1962; Humphrey 1979; Kamke and Casey 1988a). When force is applied to consolidate the mat, the wood components deform in bending and compression. The densification resulting from compression alters the physical and mechanical properties of the wood component. Changes in the strength, stiffness, sorption, and swelling of wood were found to be strongly influenced by the environment in which the densification occurred (Casey 1987; Geimer et al. 1985; Price 1976; Koch 1964; Kunesh 1961; Seborg and Stamm 1941). Environmental conditions also significantly influence the degree of damage to cell walls (Geimer et al. 1985). Significant evidence exists that these changes in wood properties can be manifested in the composite product (Hsu 1987; Geimer et al. 1985; Hujanen 1973; Heebink and Hefty 1969; Suchsland and Enlow 1968).

Clearly, the interaction of time, temperature, and moisture with nonlinear mechanical properties presents tremendous complexities to this problem. Empirical research for wood-based composites will be determined by our ability to simultaneously control the vast array of interacting variables, many of which are governed by inflexible laws of nature. Eventually, a thorough understanding of this complex system will depend on theories founded on fundamental principles. The necessity to develop theories will arise from two distinct needs: (1) a means to eliminate and narrow the focus of parameters in experiments, and (2) a framework to interpret experimental results. The objective of this research is to demonstrate the applicability of materials engineering theory to wood deformation, as it pertains to the pressing of wood-based composites. Demonstrating that wood behavior is governed by the same physical laws as other less complex materials will allow a significant step forward in the engineering design of wood-based composites.

In general, wood can be classified as a polymeric cellular material. The properties of cellular materials are governed by the cellular geometry and properties of the solid cell wall. Temperature and moisture are not likely to significantly alter the cellular structure; therefore, the influence of these factors must be primarily restricted to the cell-wall material. For polymers, the dependence of mechanical properties on time, temperature, and moisture is unified through the viscoelastic properties. The primary thesis of this paper is that the interaction of time, temperature, and moisture on the nonlinear mechanical properties of wood in transverse compression can be described using fundamental theories of cellular materials and viscoelasticity of polymers. An underlying assumption of this approach is two-part: (1) nonlinear behavior arises solely from collapse of the wood cellular structure, and (2) the material behavior of the cell-wall material can be described using linear viscoelastic theory. These assumptions are also used by Gibson and Ashby (1988) in deriving the linear viscoelastic behavior of honeycomb structures.

Theories of cellular materials

A characteristic mechanical response for cellular materials in compression is shown in Fig. 1. The material begins deforming in a linear elastic manner. A yield point is exhibited at the onset of cellular collapse. The material continues to deform at nearly a constant stress level. When a majority of the cells have collapsed, densification begins. During densification, stress rapidly increases as the collapsed cell walls consolidate.

Many micromechanics models have been developed to predict linear elastic and yielding properties of cellular materials from cell geometry and mechanical properties of the cell-wall material (Warren and Kraynik 1987; Gibson et al. 1982; Gibson and Ashby 1982; Meinecke and Clark 1973; Leaderman 1971; Whittaker 1971; Chan and Nakamura 1969; Ko 1965; Gent and Thomas 1959). Recently, Gibson and Ashby have produced a comprehensive set of theories that address the linear, nonlinear, and failure properties for elastic, plastic, and brittle cellular materials (Gibson and Ashby 1988; Gibson et al. 1982, 1988; Gibson and Ashby 1982; Ashby 1983; Maiti et al. 1984). The primary difference between Gibson and Ashby's original work and that of their predecessors is that they correctly assumed that cell-wall bending rather than extension governed deformation. These theories have been applied to a number of natural materials including wood (Ashby et al. 1985; Maiti et al. 1984; Easterling et al. 1982), cork (Gibson et al. 1981), bone (Gibson and Ashby 1988), and the iris leaf (Gibson et al. 1988).

The major shortcoming of utilizing the current applications of Gibson and Ashby's theories to wood is that nonlinear behavior is assumed to be a function of plastic yielding or fracture of the cell wall. These failure-oriented cell-wall properties are difficult to measure and will most certainly vary tremendously with environmental conditions. The variable nature of cell-wall collapse is evidenced in Fig. 2.

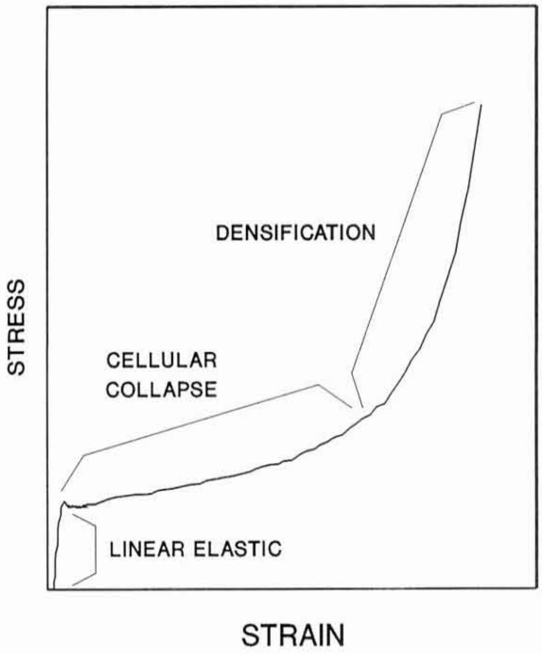

FIG. 1. A characteristic stress-strain diagram for a cellular material in compression.

Viscoelastic behavior of amorphous polymers

Amorphous polymers are termed viscoelastic because they can exhibit a range of properties from viscous fluids to linear elastic solids depending on the time scale, temperature, diluent concentration (i.e., moisture content) of the test (Fig. 3) (Ward 1983). In relative terms, at short times, low temperatures, and low diluent concentrations, the polymer exhibits glassy behavior, which can be characterized as stiff and brittle. At long times, high temperatures, and high diluent concentrations, the polymer exhibits rubbery behavior that is characterized as compliant and elastic. Between these two distinct regions is termed the transition phase; the temperature associated with the phase change is typically called the glass transition temperature (T_g). At extremely high times, temperatures, and diluent concen-

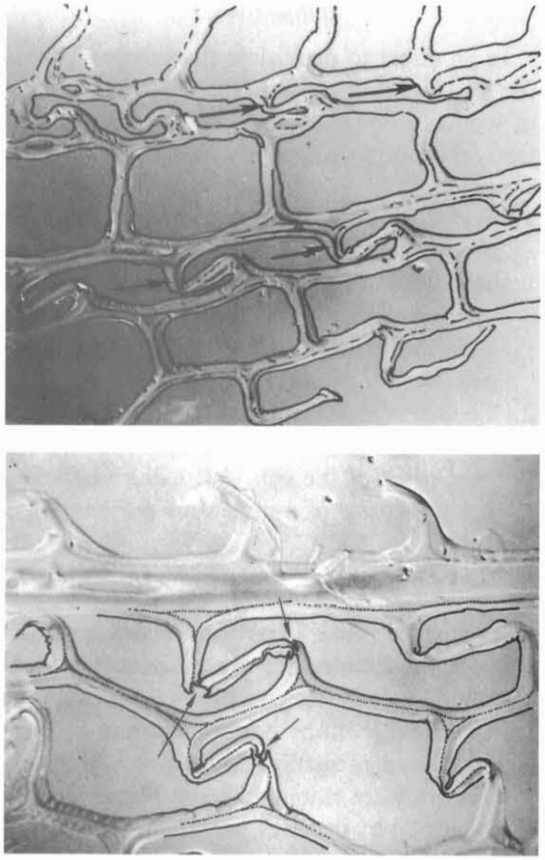

FIG. 2. Cross-sectional views of Douglas-fir flakes recovered from a pressed panel showing (a) elastic collapse and (b) fractures in the cell walls.

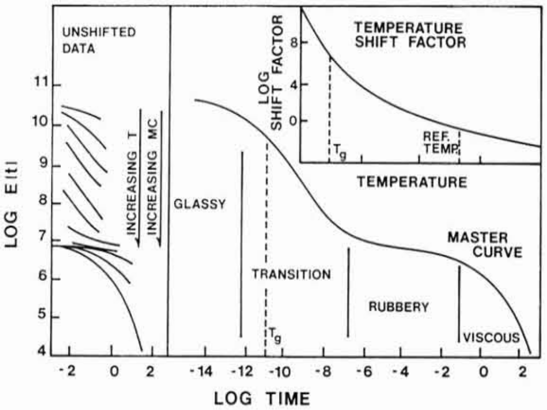

FIG. 3. Typical variation of relaxation modulus [E(t)] for an amorphous polymer with time, temperature, and diluent concentration. The master curve denotes the specific viscoelastic regions of polymer behavior.

trations, some polymers (i.e., thermoplastics) exhibit viscous flow. However, unmodified wood undergoes thermal degradation before this phase is reached.

The analogous behavior of polymers with different loading time, temperatures, and diluent concentrations is exhibited best in thermorheologically simple systems (Christensen 1982). In these polymer systems, the time-dependent mechanical responses at different temperatures and diluent concentrations are related by a change in the time scale only. From this equivalence, the long-term behavior of mechanical properties can be derived using time-temperature superposition. With this technique, the time-dependent stiffness (E(t)) of a material is determined with short-term tests at different temperatures as shown on the left in Fig. 3. These curves are then shifted along the log time axis to form a smooth, contiguous curve similar to that shown on the right in Fig. 3. The horizontal shift needed at each temperature is recorded as the shift factor shown in the upper right of Fig. 3. Once the time-dependent response of a polymer is determined over a large range of times (termed a master curve), the effect of a temperature change is equivalent to a horizontal shift of the master curve via a time multiplier (the shift factor) (Fig. 3). This relation is expressed mathematically as reduced time (t′) by the relation:

$$t' = \int_0^t \frac{d\xi}{a[T]} \qquad (1)$$

where:

t' = reduced time
t = time
$a[T]$ = temperature shift factor

At a constant temperature and diluent concentration, Eq. [1] will reduce to: $t' = t/a(T,M)$, where $a(T,M)$ is the shift factor for temperature and moisture content.

The power of this analytical technique does not lie solely in the ability to determine long-term properties using short-term experiments, but also in the ramifications of the behavior as defined by the Williams-Landel-Ferry (WLF) equation (Ferry 1980). The WLF equation indicates that for temperatures above T_g, the shift factor for time-temperature superposition is similar for all amorphous polymers. Although this observation was originally empirical, a fundamental basis for the equation has been provided using free volume theories. Free volume can be defined as the difference between the occupied volume and specific volume of a polymer (Billmeyer 1984). Occupied volume is defined as the volume occupied by the actual mass of molecule plus any volume it occupies from thermal vibrations that exclude other molecules from its domain (Adamson 1980). The influence of temperature on the viscoelastic properties of polymers results from the linear increase of free volume with temperature above T_g. Simply stated, viscoelasticity can be viewed as a self-diffusion of polymer molecules in response to a mechanical perturbation. Therefore, the mobility of polymer molecules is related to the amount of free volume at any given condition (Ferry 1980). This leads to the conclusion that increased states of free volume result in an increase in the rate of deformation. A thorough mathematical derivation of these concepts is beyond the scope of this paper but is presented elsewhere (LeFebvre et al. 1989; Knauss and Emri 1981; Ferry 1980).

The WLF equation has been shown to hold for temperatures in the range of T_g to T_g + 100 C for most amorphous polymers (Ward 1983). Salmen (1984) showed that shift factors determined for water-saturated wood are consistent with the WLF equation for temperatures in the range of T_g to T_g + 55 C. Kelley et al. (1987) determined that the WLF equation is valid between T_g and T_g + 85 C for wood plasticized with formamide. These agreements with the WLF equation lend credibility to the concept that the viscoelastic behavior of wood is a direct function of the viscoelastic response of the amorphous constituent polymers (i.e., lignin, hemicelluloses, and amorphous cellulose).

Objectives

In an effort to provide a fundamental basis for understanding the deformation processes of wood during hot-pressing, the specific objectives of this work are:

1. To formulate a methodology for describing the nonlinear transverse compression behavior of wood using theories of cellular materials in a manner that can be integrated with the viscoelastic response of cell-wall polymers.
2. To describe the interaction of time, temperature, and moisture with the mechanical properties of the cell wall using viscoelasticity theories of amorphous polymers.

EXPERIMENTAL METHODS

The experimental portion of this research involved determining the nonlinear compression and viscoelastic properties of wood in the transverse directions under a variety of temperature and moisture conditions. These experiments were intended to provide data for the analysis of theories presented above, not to provide a comprehensive description of the behavior of wood at these conditions.

Specimen preparation

Green yellow-poplar (*Liriodendron tulipifera*) blocks were split into large sections to aid in grain orientation. In preparing compression samples, the sections were conditioned to a nominal 8% moisture content, planed to 20 mm thick, and then cut to 20- × 20- mm square cross section. In addition to cubes, wood flakes were produced with a tangential face on a laboratory scale disk flaker from green blocks. The flakes were approximately 0.9 mm thick with a 20- × 20- mm square cross section. For stress relaxation tests, prismatic tension specimens were machined from the large sections. The parallel-sided tension specimens measured 20 × 8 mm in cross section and were 155 mm long. The radial direction was ori-

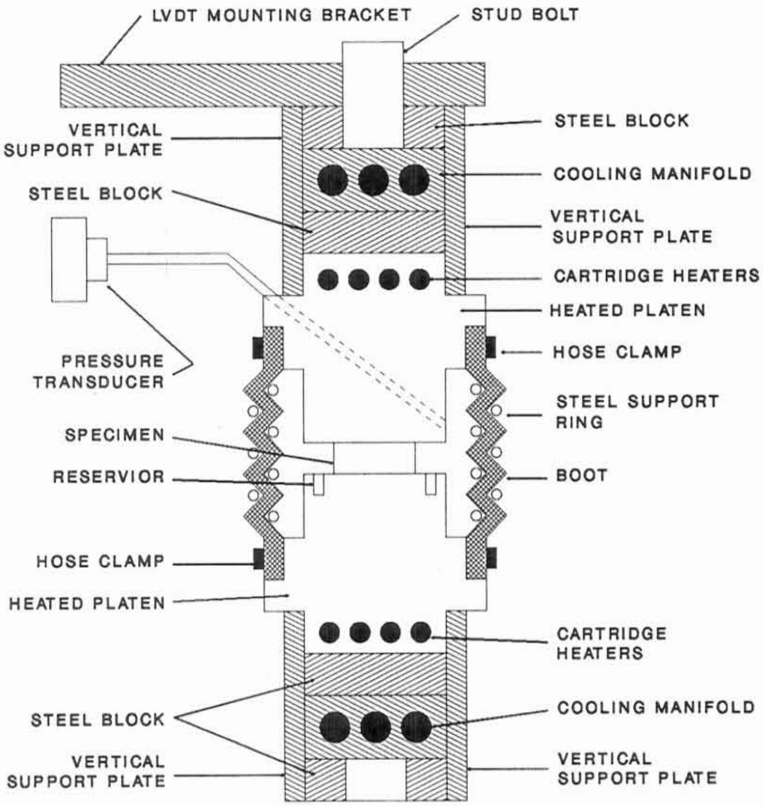

FIG. 4. Assembled test apparatus for the nonlinear compression tests.

ented with the long axis of the specimen. All specimens were loaded in the radial direction.

Nonlinear compression tests

Specimens were compressed at 6% strain per minute on a universal hydraulic testing machine equipped with heated platens rigidly attached to the crosshead and base (Fig. 4). Both aluminum loading blocks were machined flat and parallel to 0.25 mm. Each block was heated with four cartridge heaters that were inserted near the block surface remote to the specimen. A thermocouple was placed near each loading surface to provide feedback for a temperature controller. A steel block separated the loading block from the cooling manifold. Water was passed through the cooling manifold to maintain an acceptance temperature at the load cell and base. Deflection was measured with the linear variable displacement transformer (LVDT) attached to the crosshead with a stiff aluminum arm. The load-deflection relationship of the test apparatus was measured and compensated for in the test data. Both load and deflection were acquired in real time by computer and immediately converted into stress and strain.

Moisture content and temperature conditions below 100 C were maintained during the test by sealing the specimens prior to heating and compressing. The 20-mm cubes were heat-sealed in poly(vinylidene chloride) (SARAN Wrap 560, 0.15-mm-thick) envelopes. Flakes were sealed in three layers of aluminum foil (0.05 mm thick) because the deformation of the polymer film was significant compared to the deformation of the wood specimen. Pressures exceeding one atmosphere were needed to maintain saturated conditions at 120 C and 140 C. For these conditions, the compression apparatus was fit with a bellows-style reinforced rubber boot that was clamped around the cylindrical loading blocks (Fig. 4). A channel machined near the perimeter of the lower platen was filled with water. The temperature of the blocks was increased after sealing the apparatus with the test specimen inside. Gas pressure inside the sealed apparatus was monitored using an electronic pressure transducer to assure that a saturated environment was achieved.

Stress relaxation tests

Relaxation modulus was determined using a single specimen tested at a series of different temperatures and moisture contents with a universal servohydraulic testing machine equipped with an environmental chamber. The test specimen was held at each end between two serrated steel plates that were bolted together. The gripped length of the specimen was 40 mm at each end. The total gage length between the grips was 75 mm. The plates were fastened to the test machine by steel pins. This gripping arrangement allowed the specimen to be unfastened from the test machine at the pinned ends without removing the specimen from the grips.

The stress relaxation tests were conducted by imposing a step increase in strain of 0.005 and maintaining this strain for approximately 600 seconds. The specimen was then returned to zero strain for an equal recovery period. Strain was measured using an extensometer (25.4-mm gage length) attached to the specimen with metallic coil springs. The load and strain data were acquired in real time by a computer.

Each group of stress relaxation tests was conducted at a constant nominal moisture content between 3% and 16% in a single day. In general, tests were conducted at temperatures between 39 C and 99 C at 3 C intervals. The actual temperatures deviated from this range at low and high moisture contents because of equipment limitations. The actual moisture content and temperatures are listed in Table 1. The relative humidity needed to achieve a target moisture content at each temperature was calculated using an inverted form of the one-hydrate Hailwood-Horobin equation as presented by Simpson (1973). The moisture content of the test specimen was estimated using control samples that remained in the test chamber at all times except during weighing. The moisture content of the control samples was measured at the beginning and end of each day's tests. Moisture content was found to vary less than 1.5% between the beginning and end of the day; however, the variation between these times is unknown.

TABLE 1. *Environment conditions used in stress relaxation tests to construct relaxation modulus master curves. Tests were conducted at 3 C intervals.*

Moisture content (%)		Temperature range (deg C)	
Nominal	Actual	Low	High
3	3.1	57	99
6	5.6	39	99
10	9.3	39	99
12	12.0	39	90
16	16.6	39	75

RESULTS AND DISCUSSION

Nonlinear compression behavior

Elastic versus plastic collapse mechanisms.—It has been shown that micromechanical models developed for honeycomb and closed cell foams can be applied to wood (Maiti et al. 1984; Easterling et al. 1982). These researchers assume that the nonlinear mechanical response of wood results from the plastic yielding and fracture of the cell walls. Without doubt, the cell walls of wood are damaged during transverse compression to large strains. However, the question arises whether the elastic buckling of cell walls contributes to the collapse mechanisms and can be used to adequately model the nonlinear mechanical behavior. To answer this question, experimental evidence is evaluated using the theories developed by Gibson and Ashby (1988).

The Young's modulus (E) of a closed cellular solid can be written as:

$$E = C_2 E_w \delta^3 \quad (2)$$

where:

C_2 = constant
E_w = cell wall modulus
δ = relative density (ratio of the cellular density to the cell wall density)

This equation is derived from classical plate theory and applies to elastomeric and rigid plastic cellular materials.

The yield stress (σ_y) of cellular materials is influenced by the collapse mechanism of the cell wall. Yielding in elastomeric foams results from elastic buckling of the cell walls. The yield point can be calculated by:

$$\sigma_y = C_3 E_w \delta^3 \quad (3)$$

where:

C_3 = constant

For rigid plastic cell-wall materials that exhibit a yield point, yielding in the cellular solid results from the formation of plastic hinges. Yield stress in the cellular material can be determined from the fully plastic moment in the cell wall:

$$\sigma_r = C_4 \sigma_{yw} \delta^2 \quad (4)$$

where:

C_4 = constant
σ_{yw} = cell wall yield stress

Given Hooke's Law ($E = \sigma/\epsilon$), C_3 in Eq. [3] is equivalent to the yield strain (ϵ_y). Therefore, ϵ_y should be independent of density for cellular materials that deform nonlinearly from elastic buckling of the cell wall. However, it can be shown from Eqs. [2] and [4] that this relation does not hold for rigid plastic foams. Inspection of data presented by Easterling et al. (1982) showed that ϵ_y did not vary significantly for balsa (*Ochroma lagopus*) with density ranging from 0.078 to 0.218 g/cm^3. Mechanical tests conducted by Kasal (1989), using balsa, yellow-poplar, and maple (*Acer rubrum*) at a range of moisture contents and temperatures, showed that ϵ_y consistently equalled approximately 0.015. This agrees closely with the value of 0.014 determined by Maiti et al. (1984) and 0.012 from Easterling et al. (1982). These results can be reconciled if elastic buckling occurred prior to developing a fully plastic moment in the cell wall. The cell wall could then be damaged after collapse. This concept is proposed by Gibson and Ashby (1988) to explain the shape of failure envelopes for multiaxial loading.

To illustrate the argument presented above, ϵ_y and σ_y were calculated for wood, assuming both elastic and plastic collapse. The results are presented in Figs. 5 and 6. Because E_w and σ_{yw} are unknown, they were calculated from the measured modulus and σ_y using Eqs. [2] and [4] respectively. For 20-mm cubes conditioned to 8% moisture content and 30 C, E_w = 12 GPa and σ_{yw} = 49 MPa.

For low relative densities, the difference between the predicted values of σ_y assuming elastic and plastic collapse mechanisms is small. However, large differences exist between the predicted values of ϵ_y. For higher relative densities when plastic collapse may actually occur, the error introduced to the predicted σ_y by assuming elastic collapse is not serious for relative densities less than 0.35. Comparing these results with those observed experimentally, it seems likely that the cell walls collapse from elastic buckling at low relative densities. It is important to realize that this conclusion does not exclude the possibility that the cell walls yield or fracture after collapse has occurred.

Modeling the structural nonlinearity of the cell wall.—Clearly, the characteristic shape of the stress-strain diagram is a direct result of the geometric nonlinearity resulting from the response of the cellular structure. Rusch (1969) first proposed the separation of the structural and cell-wall material response through a modification of Hooke's Law:

$$\sigma = E\epsilon\phi(\epsilon) \quad (5)$$

The nonlinear strain function, $\phi(\epsilon)$, is a function of the applied strain that governs the geo-

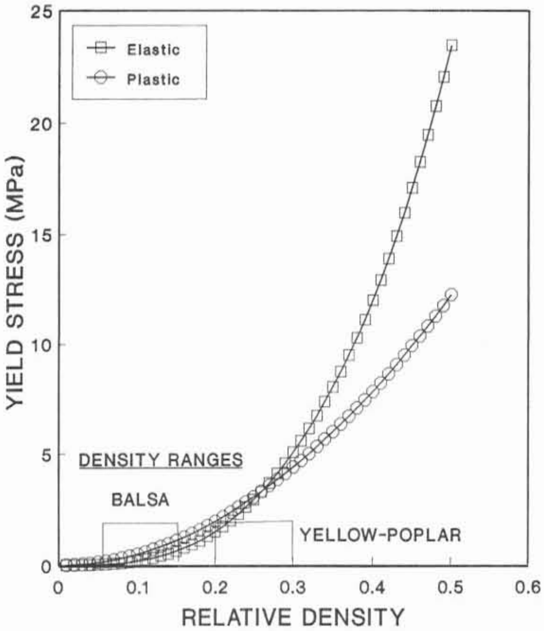

FIG. 5. Yield stress as a function of relative density calculated assuming elastic and plastic collapse mechanisms.

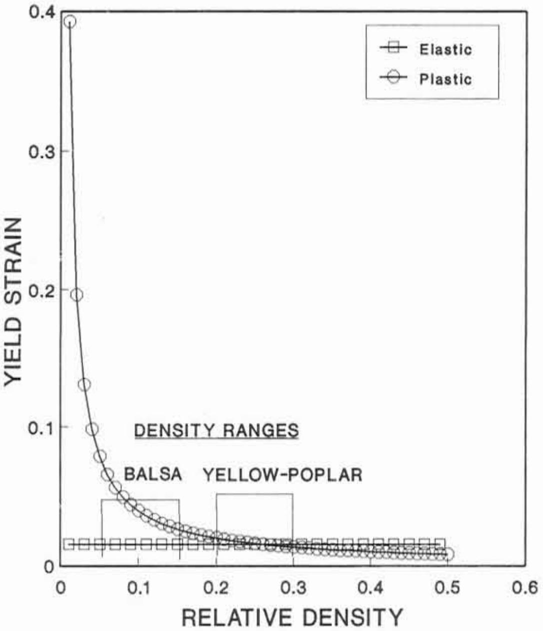

FIG. 6. Yield strain as a function of relative density calculated assuming elastic and plastic collapse mechanisms.

metric nonlinearities. It can be derived experimentally from the observed plastic strain in the stress-strain diagram (Rusch 1969). Maiti et al. (1984) derived a relation to describe the plateau and densification region of the stress-strain diagram. The longest cell walls were assumed to buckle first, resulting in the yield point. They then postulated that the length of the cell walls that will buckle with increasing compression strain could be described as a function of the longest cell-wall length and the increasing elative density with strain ($\delta(\epsilon)$). These equations can be rewritten allowing $\phi(\epsilon)$ to be described for strains greater than ϵ_y (Wolcott et al. 1989a):

$$\phi(\epsilon) = \frac{\epsilon_y}{\epsilon}\left[\frac{1-\delta}{1-\delta(\epsilon)^{1/3}}\right]^3 \qquad (6)$$

For strains less than ϵ_y, $\phi(\epsilon)$ is equal to unity. If $\delta(\epsilon)$ is calculated from the plastic strain, then $\phi(\epsilon)$ is equal to unity when $\epsilon = \epsilon_y$.

Dilatation (volume change) results from cellular collapse even in uniaxial loading. Therefore, $\delta(\epsilon)$ is a monitor of the cellular collapse. Little or no lateral expansion occurs in an elastomeric foam specimen within the nonlinear region of the stress-strain relationship. For this case, $\delta(\epsilon) = \delta/(1 - \epsilon)$. However, lateral expansion has been noted for wood (Wolcott et al. 1989a). When the loaded surfaces are restrained from frictional forces, the specimen exhibits barrelling and $\delta(\epsilon)$ can be determined by (Kasal 1989):

$$\delta(\epsilon) = \delta\left[1 - \epsilon_p + \frac{2}{3}\mu\epsilon_p - \mu\epsilon_p^2\right]^{-1} \qquad (7)$$

where: ϵ_p = plastic strain = $\epsilon - \epsilon_y$

The expansion coefficient (μ) is defined as the ratio of the lateral strain to compressive strain in the nonlinear stress-strain region. This parameter is influenced by specimen height because the surfaces are restrained by frictional forces between the test samples and compression platens. Characteristic values of μ for yellow-poplar at 30 C and 12% moisture content were between 0.1 and 0.15 for 20-mm cubes and approximately 0.05 for 0.9-mm-thick

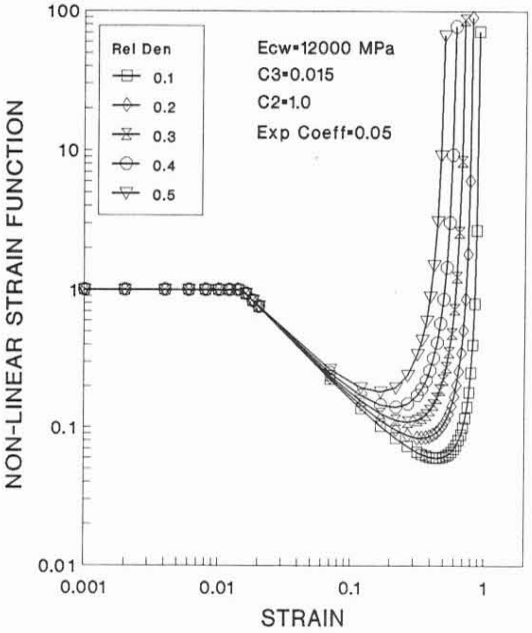

FIG. 7. Predicted nonlinear strain function ($\phi(\epsilon)$) by relative density.

flakes. A thorough discussion of this is presented elsewhere (Kasal 1989).

Whereas ϵ_y was found to be relatively invariant with material variables such as species, density, moisture content, and temperature, it has been found to be affected significantly by specimen geometry (Wolcott et al. 1989b; Menges and Knipschild 1982; Kunesh 1968; Bodig 1963). It has been found that the apparent E increases nonlinearly with specimen height, while ϵ_y varies with the reciprocal of E. A conclusive explanation of this behavior is lacking. However, the phenomenon has been related experimentally (Menges and Knipschild 1982) and theoretically (Wolcott et al. 1989b) to the excessive deformation of the loaded surfaces. Wolcott et al. (1989a) showed that surface asperities as small as 0.25 mm could significantly influence the measured E in wood. The relationship described above can be modeled through C_2 in Eq. [2] and should be viewed purely as a specimen effect (Wolcott et al. 1989a).

Experimental results.—The primary focus of the research presented here is to determine whether the nonlinear response of wood in transverse compression can be modeled using theories of cellular materials assuming elastic collapse mechanisms. To remove the variability introduced by the unknown cell-wall modulus, Young's modulus and ϵ_y for the radial direction were determined for every specimen. These values, along with the measured density, were used in Eqs. [5], [6], and [7] to calculate the predicted nonlinear response.

A comparison of experimental and predicted behavior for the plateau and densification regions was made using two distinct points: ϵ_{den}, ϵ_u. These points were chosen as a measure to test the theoretical behavior with experimental results because they can be easily identified both mathematically and visually through the nonlinear strain function: $\phi(\epsilon)$ (Fig. 7). The ϵ_{den} is defined here as the beginning of the densification region, which is denoted by a minimum in $\phi(\epsilon)$. The point where $\phi(\epsilon)$ equals unity after reaching ϵ_{den} is defined here as ϵ_u.

Experimental and predicted values for the stress and strain at ϵ_{den} and ϵ_u were used to quantitatively determine how well $\phi(\epsilon)$ predicted the collapse and densification regions of the stress-strain relation for flakes. Typical predicted and experimental curves are presented in Fig. 8. On average, predicted values of ϵ_{den} and ϵ_u are less than those measured experimentally. The mean errors in predicting ϵ_{den} are 11.1% for stress and 8.2% for strain. For predicting ϵ_u, the mean errors were 9.5% for stress and 9.9% for strain. Given these results, the theories regarding the nonlinear behavior of cellular materials presented here appear to represent wood in transverse compression.

Interaction of time, temperature, and moisture

Analytical development.—An important aspect of classical viscoelasticity theory in polymers is time-temperature equivalence. Originally, time-temperature superposition was used to shorten the time required to determine time-dependent mechanical properties. The power of this technique was not fully realized until the advent of free volume theories and their relation to the WLF equation. Ferry (1980) notes that criteria other than the smooth shifting of curves should be used to substantiate the validity of experimentally determined shift factors. Theory such as the WLF equation can be used to substantiate validity of the time-temperature equivalence when prior experience does not exist for shifting. Successful application of these theories also indicates that a material behaves viscoelastically, like other amorphous polymers. For wood, this conclusion has important implications both theoretically and experimentally.

This research involves the interaction of both temperature and moisture with time-dependent properties of wood. An equivalent approach to time-temperature superposition has been used with moisture (Maksimov et al. 1975, 1976). In time-temperature-moisture superposition, time-dependent properties are determined at different temperatures and moisture contents. These curves are then shifted along the log time axis to form the master curve in a manner analogous to time-temperature superposition. The shift factor for temperature and moisture (a[T,M]) is then defined using reduced time:

$$t' = \int_0^t \frac{d\xi}{a[T, M]} \tag{8}$$

Knauss and Emri (1981) proposed a free volume theory that accounts for changes in temperature, diluent concentration, and hydrostatic stress to predict nonlinear viscoelastic properties. Their model assumes that a change in free volume of a polymer depends on the dilatation from these three variables. The free volume of a polymer can then be expressed as:

$$f = f_o + \alpha(T - T_o) + \beta(C - C_o) + \delta\sigma \tag{9}$$

where:

f = fractional free volume
T = temperature
C = diluent concentration
σ = hydrostatic stress

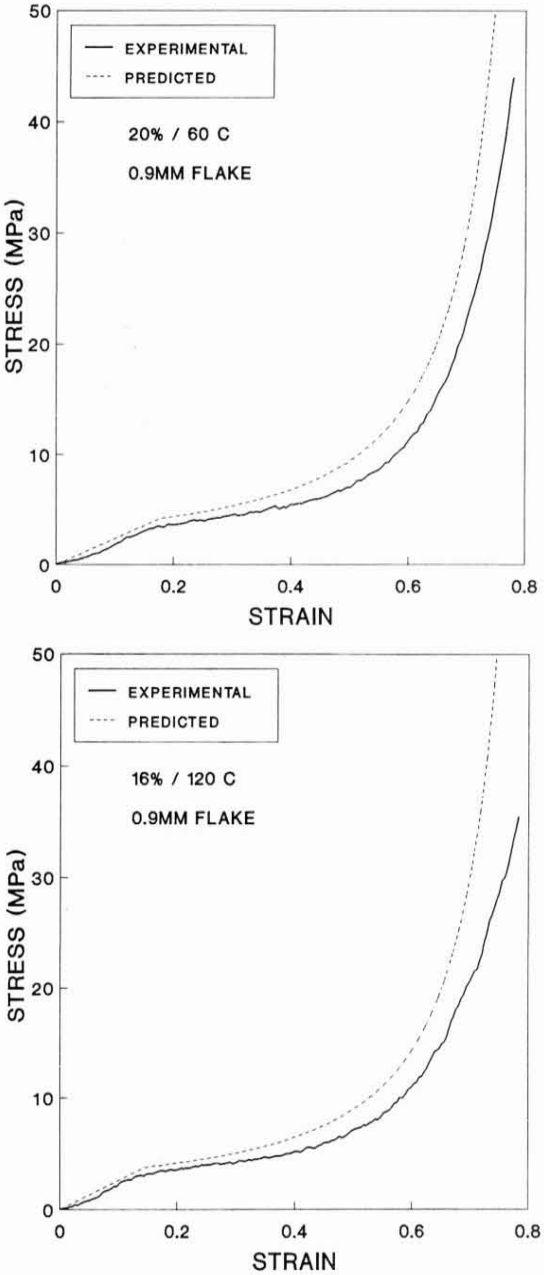

FIG. 8. Experimental and predicted stress-strain diagrams for 0.9-mm flakes.

α, β, δ are free volume expansion coefficients. Subscript *o* refers to the reference condition

The shift factor can be defined in terms of fractional free volume through the Dolittle equation (Ferry 1980):

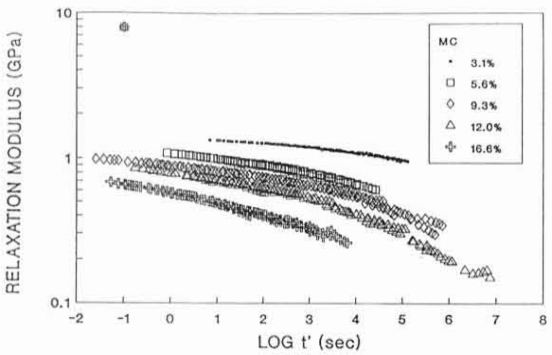

FIG. 9. Temperature-compensated relaxation modulus (E(t')) plotted against reduced time (t' = t/a[T,M]). The temperature-compensated curves are shifted to a reference temperature of 60 C.

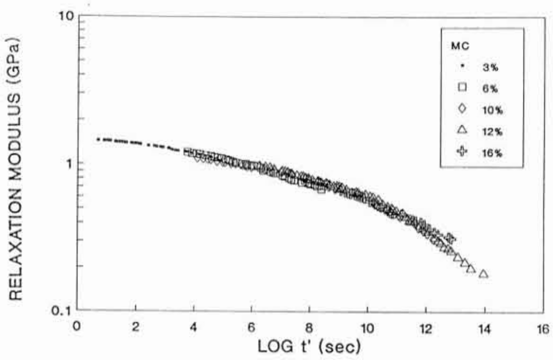

FIG. 10. Relaxation modulus (E(t')) plotted against reduced time (t' = t/a[T,M]). The master curve was developed by shifting the temperature-compensated curves in Fig. 9 to a reference moisture content of 3.1%.

$$\log a = \frac{B}{2.303}\left[\frac{1}{f} - \frac{1}{f_0}\right] \qquad (10)$$

where B = constant.

Combining Eqs. (9) and (10):

$$\log a[T, C, \sigma] = -\frac{B}{2.303}\left[\frac{\alpha(T - T_0) + \beta(C - C_0) + \delta\sigma}{f_0 + \alpha(T - T_0) + \beta(C - C_0) + \delta\sigma}\right] \qquad (11)$$

Equation [11] defines the behavior of the shift factor for amorphous polymers above the glass transition temperature. It is likely that a majority of the stress relaxation tests conducted under the conditions outlined in Table 1 will correspond with the glassy region of lignin. At temperatures below T_g, free volume does not expand in a linear manner. In the glassy region, the shift factor can be described using an Arrhenius-type equation [10], which is derived from entropy-based arguments (LeFebvre et al. 1989). Substituting this temperature dependence while neglecting stress effects that are difficult to evaluate on a cell-wall level, temperature and moisture dependence can be described through the shift factor as:

$$\log a[T, M] = \frac{\Delta H_a}{2.303RT} - \left[\frac{(B/2.303f_0)(C - C_0)}{(f_0/\beta) + (C - C_0)}\right] \qquad (12)$$

where:

ΔH_a = apparent activation energy
R = universal gas constant

Experimental results

The relaxation modulus (E[t]) determined for different temperatures at a common moisture content was shifted along the log time axis to a reference temperature of 60 C (Fig. 9). The temperature-compensated curves were then shifted along the time axis to construct a master curve for both temperature and moisture content (Fig. 10). This shifting process resulted in a smooth curve possessing a shape consistent with those displayed by amorphous polymers. The recorded shift factors were then evaluated using Eq. [12]. To solve for the four unknowns (ΔH_g, B, f_o, β), a two-step procedure was utilized. First, the temperature dependence of the relation was determined by differentiating Eq. [12] with respect to 1/T:

$$\frac{d(\log a[T, M])}{d(1/T)} = \frac{\Delta H_a}{2.303R} \qquad (13)$$

Using Eq. [13], a linear regression of d(log a[T,M]) versus d(1/T) will yield a slope value equal to $\Delta H_a/2.303R$. The differential values of log a[T,M] and 1/T are calculated from a

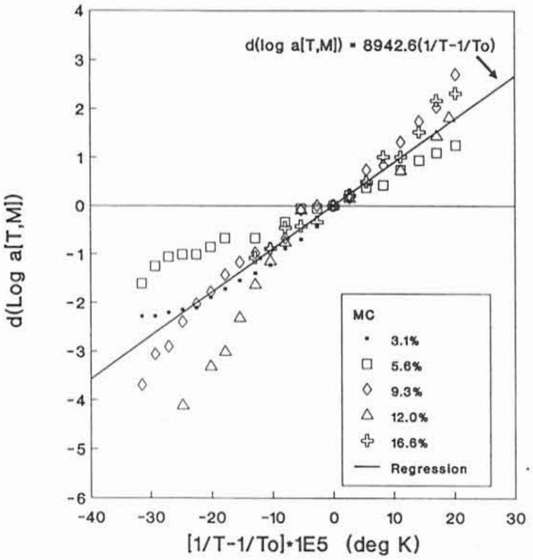

FIG. 11. Linear regression results of d(log a[T,M]) versus (1/T − 1/To). The reference temperature (To) equals 333.15 K. The $r^2 = 0.83$ for the regression analysis.

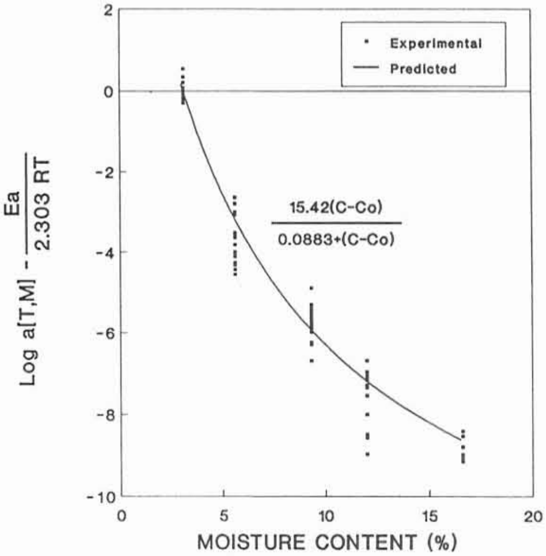

FIG. 12. Nonlinear regression results to determine moisture dependence at a[T,M].

reference value of 60 C (333 K). In the second step, the values of ($B/2.303f_o$) and (f_o/β) were determined using a nonlinear regression technique. The model used was:

$$\log a[T, M] - \frac{\Delta H_a}{2.303RT} = -\left[\frac{(B/2.303f_0)(C - C_0)}{(f_0/\beta) + (C - C_0)}\right] \qquad (14)$$

The results of this two-stage procedure are presented graphically in Figs. 11 and 12.

The linear regression analysis used to solve for the temperature dependence of the shift factor yielded the slope shown in Fig. 11. The intercept was not significant at the 0.05 level. This result is expected because the shift factor will equal zero at the reference temperature. The apparent activation energy calculated from the slope of this regression is found to be 171 kJ/mole. This value is nearly identical to the 172 kJ/mole determined by Caulfield (1984) for the viscoelastically dominated rate of loading and duration of loading effects for strength properties of wood. Van de Put (1987) determined an activation energy for short-term relaxation properties of wood as 209 kJ/mole. As Caulfield recognized, these apparent activation energies are approximately equivalent to that required to break nine hydrogen bonds. Nissan (1977) has developed a theory for creep of hydrogen-bonded materials that involves the simultaneous breaking of hydrogen bonds.

The results of the nonlinear regression analysis are shown in Fig. 12. To evaluate the meaning of these coefficients, they must be reduced to the fundamental variables of f_o and β. The constant B from the Dolittle equation is typically assumed to equal unity from previous experimental results (Ferry 1980). With this relation, the identity for ($B/2.303f_o$) = 15.4 and (f_o/β) = 0.083 can be solved simultaneously to yield $f_o = 0.028$ and $\beta = 0.33$.

The universal value for f_o is 0.025 at T_g (Ferry 1980). This value is remarkably consistent with 0.023 determined by Salmen (1984) and 0.0245 by Kelley et al. (1987) at T_g. Fractional free volume can be viewed as the fraction of micro-voids in the cell wall that is often referred to in the wood/moisture literature (Skaar 1972; Stamm 1964). Stamm (1964) calculated that the fraction of micro-voids in mercerized cellulose was equal to 0.023 using the difference between swelling volume and the volume of water adsorbed. This same technique was used independently by Adamson (1987) in determining a fractional free volume of 0.025 for neat epoxy. Certainly, the agreement of the results presented here with that of various researchers lends credibility to the general nature of free volume concepts.

Little experimental evidence is available to evaluate the expansion coefficient β. However, a mathematical relation is given for the fractional free volume of polymer/diluent systems by McKenna (1989):

$$f = v_p f_p + v_d f_d - k v_p v_d \qquad (15)$$

where:

f = fractional free volume
v = volume fraction
k = interaction parameter (ca. 10^{-2})
subscripts p and d denote polymer and diluent, respectively

The free volume expansion coefficient, β, is equivalent to the first derivative of Eq. [15] with respect to C. Because of the difficulty in converting from weight fraction (C) to volume fraction, this differential was evaluated numerically. For moisture contents ranging from 3% to 16%, the average value for $df/dC = 0.33$ for a $f_d = 0.28$. This value is within the range of 0.1 to 0.3 given for small-molecule diluents far above T_g (McKenna 1989). Given these agreements, the parameters determined for Eq. [12] are certainly within the range predicted by theory and determined experimentally for the general class of amorphous polymers.

CONCLUSIONS

Theories of mechanical properties of cellular materials and viscoelastic properties of amorphous polymers were presented and compared to the experimental behavior of wood. The objective of this research was to evaluate the conformity of wood behavior to these theories to provide a fundamental basis for understanding the deformation processes of wood during the pressing cycle of wood-based composites.

Wood was observed to display a stress-strain relationship in transverse compression that is characteristic of cellular materials. Comparison was made between the theoretical and experimental yield points assuming both plastic and elastic collapse mechanisms. It was concluded the cell walls in low density species of wood are likely to undergo elastic collapse. Only small errors are introduced by assuming elastic collapse for high density woods. An analysis of the collapse and densification regions of the stress-strain relationship for yellow-poplar flakes was found to be adequately predicted by theory assuming elastic collapse mechanisms.

The viscoelastic behavior of wood was compared to free volume theories for amorphous polymers. Specifically, the interactions of temperature and moisture content with the transverse relaxation modulus of wood were studied through time-temperature-diluent concentration superposition. Shift factors were evaluated using a modified version of the free volume theory presented by Knauss and Emri (1981). The analysis showed that temperature dependence is controlled by an Arrhenius-type behavior with an apparent activation energy of 171 kJ/mole. This value is consistent with findings by other researchers with wood. Moisture dependence was found to be controlled by changes in free volume with moisture content. The fractional free volume for the glassy region was determined to be 0.028. This value is remarkably consistent with the "universal" figure of 0.025 for amorphous polymers. The free volume expansion coefficient for moisture changes was calculated to be 0.33. This value is within the range predicted by theory.

There is a certain appeal to the fundamental materials engineering models, presented here, which can unify the interactions of time, temperature, and moisture with the nonlinear response of wood in compression. The polymers and cellular structure that dictate responses under these conditions are common to all species of wood. In addition, fiber and flake-based mats of wood-based composites can also be viewed as a cellular material. The conformity of wood with these general materials theories opens many avenues for research aimed at fully engineered wood-based composites. For instance, the predictive nature of these theories can be used to understand wood behavior under environmental conditions indicative of the pressing cycle that are extremely difficult to control experimentally. Also, wood mats can be experimentally modeled using mats composed of synthetic polymer fibers and films. These systems could provide stepping stones in our understanding of more complicated wood-based systems by eliminating experimental parameters such as moisture and natural variability. Finally, the demonstration that wood behavior can be studied as a function of the governing polymer behavior, coupled with the structural geometry of the cellular anatomy, has implications far beyond the scope of this particular problem.

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