MECHANICAL PROPERTIES OF INDIVIDUAL SOUTHERN PINE FIBERS. PART III: GLOBAL RELATIONSHIPS BETWEEN FIBER PROPERTIES AND FIBER LOCATION WITHIN AN INDIVIDUAL TREE

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ABSTRACT

This is the third and final paper in a three-part series investigating the effect of location within a tree on the mechanical properties of individual wood tracheids. This paper focuses on the definition of juvenile, transition, and mature zones as classified by fiber stiffness, strength, microfibril angle, and cross-sectional area. The average modulus of elasticity and ultimate tensile stress of all loblolly pine fibers were, in equal proportion of earlywood and latewood, 17.3 Gpa and 824 Mpa, respectively. The average microfibril angle was found to be 15.4 degrees, with rings 5 and 48 averaging 26.8 and 6.1 degrees, respectively. Normalization of all mechanical and physical properties showed that the juvenile zone is not cylindrical but rather biconical, tapering from stump to below the live crown and then again from the live crown to the bole tip. The transition zone parallels the juvenile zone, ranging in width from 3 to 15 rings. Fiber properties continued to improve slightly throughout the duration of the mature zone.

Keywords: Loblolly pine, wood fibers, tracheids, stiffness, strength, modulus of elasticity, ultimate tensile stress, microfibril angle, juvenile wood, mature wood, fiber quality index, whole tree variation.

INTRODUCTION

The continuing trend of increasing the efficiency of our renewable resources has resulted in the increased production of engineered wood-based composites that rely primarily on the smallest basic unit from a tree; the wood fiber. Although the wood fiber is the primary component in paper, paperboard, and structural fiberboard, very little fundamental information exists on the mechanical properties of individual wood fibers.

There exists a strong relationship between wood juvenility and the structural performance of wood-fiber-based paper and com-

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posites. This relationship has been shown by numerous researchers for paper (Semke and Corbi 1974; Cown and Kibblewhite 1980; Labosky and Ifju 1981) and structural fiberboard (McMillin 1969; Nelson 1973; Pugel et al. 1990). This relationship indirectly supports the thesis that fiber mechanical properties are directly linked to the mechanical properties of the composite. Page et al. (1972) have shown this thesis to be true for paper. Groom et al. (1999) have demonstrated that this is also valid for medium-density fiberboard.

The potential benefits of modeling wood-fiber-based composites based partially on fiber physical and mechanical properties are enormous. These models will allow us to determine the effect of silviculture and genetics on final composite performance. The previous two papers in this series (Groom et al. 2001; Mott et al. 2001) have evaluated fiber mechanical properties in a preliminary sense, focusing either on juvenility or the comparison between earlywood (EW) and latewood (LW) fibers. This paper combines the data from the previous two papers to thus outline the effect of tree location on the properties of loblolly pine fibers. It is with this in mind that the primary objective of this paper, the third in a three-part series, is to characterize the effect of within-tree location on fiber mechanical properties. A secondary objective is to identify the effect of within-tree location on fiber physical properties.

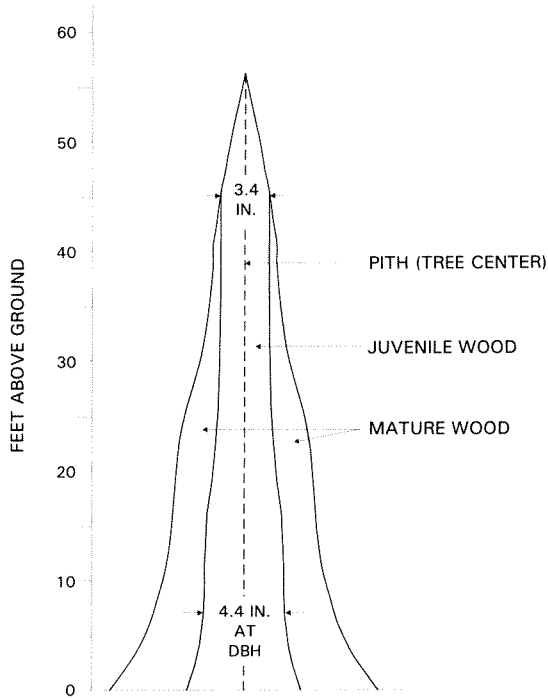

FIG. 1. Distribution of specific gravity for longleaf pine at various distances from the pith and heights from Fernow (1896).

LITERATURE REVIEW

Within-tree variability of wood samples

The effect of specimen location within a tree has been studied quite extensively as it relates to growth variables and specific gravity. One of the earliest studies on wood was such a case. Fernow (1896) investigated the effect of within-tree location on the specific gravity of longleaf pine and found that specific gravity is lowest near the pith and increases with cambial age. In addition, Fernow also found that specific gravity variability is greatest at the base of the tree and decreases with height.

The data supporting this relationship between distance from the pith and specific gravity have been strengthened over the past century by a myriad of researchers and have been one of the primary definers of juvenile, transition, and mature wood. Juvenile wood has been rather extensively categorized in loblolly pine (*Pinus taeda* L.) to be between 6 and 14 years (Zobel et al. 1959; Pearson and Gilmore 1980; Bendtsen and Senft 1986; Clark and Saucier 1989). Juvenile wood is directly related to cambial age (Pearson and Gilmore 1980) and has been shown to be controlled by auxin production in the tree crown (Larsen 1962).

The vast majority of studies focusing on the effect of juvenility are based on specimens at or near the breast height. However, there are a few notable exceptions. Figure 1 summarizes work by Zobel and Talbert (1984) that inves-

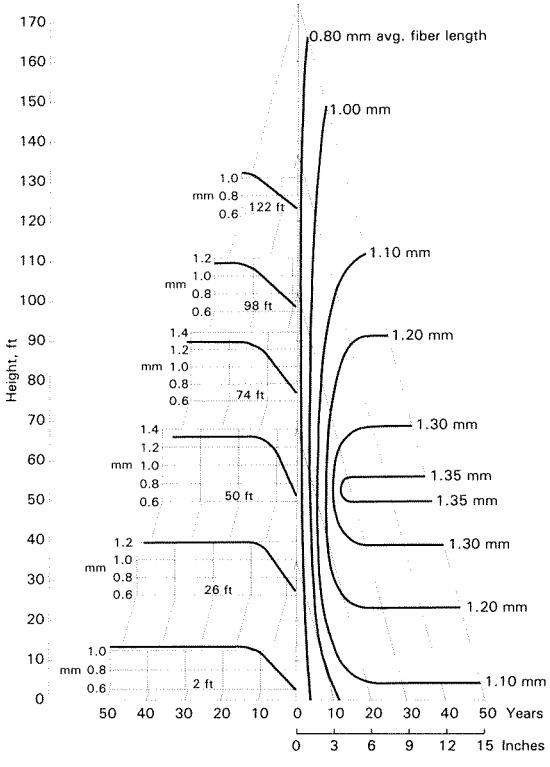

FIG. 2. Schematic diagram of radial and vertical fiber length variations for *Eucalyptus regnans* F.v.M. The left half of the image represents graphic curves at various heights. The right half of the image uses the same data but is shown as a series of isometric lines. (Adapted from I.J.W. Bisset and H.E. Dadswell 1949).

tigated the horizontal and vertical location of juvenility within several 17-year-old southern pines. The authors found that the juvenile zone is essentially a cylinder extending from the base to the tip of the bole. Megraw (1985) argues that although the concept of a cylinder is useful, there exist biological differences along the tree height that suggest juvenility is a function of cambial age and vertical position within the tree. This nonuniformity can be seen in a classic publication by Bisset and Dadswell (1949) in which the authors evaluated the effect of fiber length on location within a tree of *Eucalyptus regnans* F.v.M. (Fig. 2). Bisset and Dadswell found that fiber length increased with cambial age and reached a maximum near the bark approximately 1/3 total tree height. The deviation of physical properties from a cylindrical juvenile core has also been found by Yang and Benson (1997). Indefining juvenility, they found that cell length and growth ring width indicate a cylindrical juvenile wood core.

Within-tree variability of fiber mechanical properties

There currently exists no literature on the effect of location within a tree on wood fiber mechanical properties. Various researchers have studied the effect of location on mechanical properties of solid wood specimens. Pearson and Gilmore (1980) evaluated the effect of innerwood versus outerwood for loblolly pine specimens. The classification of innerwood specimens was determinant on log size, but included or was near the pith. The authors found that static bending modulus of rupture (MOR) increased by 25 to 50% from innerwood to outerwood, depending on stand type. Modulus of elasticity (MOE) was impacted more by stem maturation, increasing 35 to 90%. Similar results were also found by Barrett and Kellogg (1991) for second-growth Douglas-fir. Bendtsen and Senft (1986) also evaluated the effect of juvenility on wood mechanical properties by evaluating small, clear bending specimens in one-year increments from pith to bark. The authors found a large shift in mechanical properties: MOR and MOE increased approximately 200 and 500 % respectively, from pith to bark.

McKimmy (1986) evaluated the effect of juvenility and vertical location within boles of Douglas-fir. He found that near the stump and in upper logs, lumber from juvenile wood had lower MOR and MOE values than mature lumber. McKimmy also found that mature wood from lower and upper logs possessed similar MOR and MOE values. This was not the case with juvenile wood specimens. For juvenile wood, the MOR and MOE for lumber from top logs were higher than their stump counterparts, suggesting that juvenile wood is

not constant throughout the height of the tree. Similar results were also found by Senft et al. (1986) and Zhou and Smith (1991).

Although no literature exists on the effect of within-tree variation on fiber mechanical properties, a comprehensive study on effect of within-tree location on fiber physical properties was published by Megraw (1985). Megraw dealt primarily with changes in specific gravity from pith to bark and from stump to tip. However fiber length, in which a strong, inverse relationship with microfibril angle exists (Wardrop 1951), was also discussed. Megraw found that in loblolly pine, fiber length increases with cambial age and is at a minimum at the base of the tree. In subsequent publications (Megraw et al. 1998; Megraw et al. 1999), the data were expanded, focusing primarily on microfibril angle.

Megraw et al. (1998) found that microfibril angle in loblolly pine decreases with cambial age at all heights except at stump height. The microfibril angle increases from pith to growth ring 7 at which time the angle decreases to bark. They also found an increase from growth rings 15 to 20 in the live crown, but likened the increase to a thinning 4 years prior to sampling. In addition, Megraw et al. found that for a given growth ring, microfibril angle decreases with tree height. Megraw et al. (1999) evaluated the same trees as in Megraw et al. (1998) but with emphasis on clearwood stiffness. The authors found that MOE values in loblolly pine are very much a function of cambial age and vertical location in the tree. The most compliant samples were located at stump height. Stiffness increased with height up to 3 m, at which the differences were negligible. Stiffness also increased with cambial age, with the greatest increases evident in the juvenile portion of the bole.

A workshop conducted in New Zealand in 1998 focused primarily on the measurement of microfibril angle in wood and its effect on wood properties. The assembled proceedings (Butterfield 1998) are of significant importance in the understanding of development of wood mechanical properties. Most noteworthy papers in reference to wood stiffness are Megraw et al. (1998), Navi (1998), Donaldson (1998), Astley et al. (1998), Booker et al. (1998), Tsehaye et al. (1998), Groom et al. (1998), and Kopenen (1998). Navi (1998) did discuss the effect of microfibril angle on the tensile properties of individual wood fibers. However, the focus was primarily on modeling the behavior of the stress-strain curve and thus no data were presented on the effect of within-tree location on fiber properties. Theoretical papers such as that given by Navi (1998), married with the information presented by Megraw et al. (1999) outlining microfibril angle in loblolly pine, El-Hosseiny and Page (1975) relating fiber strength to microfibril angle, and Page and El-Hosseiny (1983) discussing the effect of microfibril angle on fiber stiffness provide us with the best estimate of fiber mechanical properties regarding within-tree location.

EXPERIMENTAL PROCEDURES

The first two phases of this study involved the mechanical properties of EW and LW loblolly pine fibers, with a detailed description of the experimental procedures found in the corresponding papers (Groom et al. 2001; Mott et al. 2001). The following section is an abbreviated description of the material selection, material preparation, and experimental procedures.

Material selection

A 48-year-old loblolly pine (*Pinus taeda* L.) tree was selected and felled from a conventional plantation stand. The tree was straight in form and had a diameter at breast height of 42.2 cm, live crown height of 21.2 m, and an overall height of 30.3 m. Immediately upon felling, a disk approximately 2.5 cm thick was removed every 3 m in height, starting from the stump and proceeding to a 10-cm top. Several slivers measuring approximately 2 by 2 by 25 mm were removed from each disk at growth rings 5, 10, 20, 30, 40, and 48. The number of growth rings analyzed for each disk varied as a function of tree

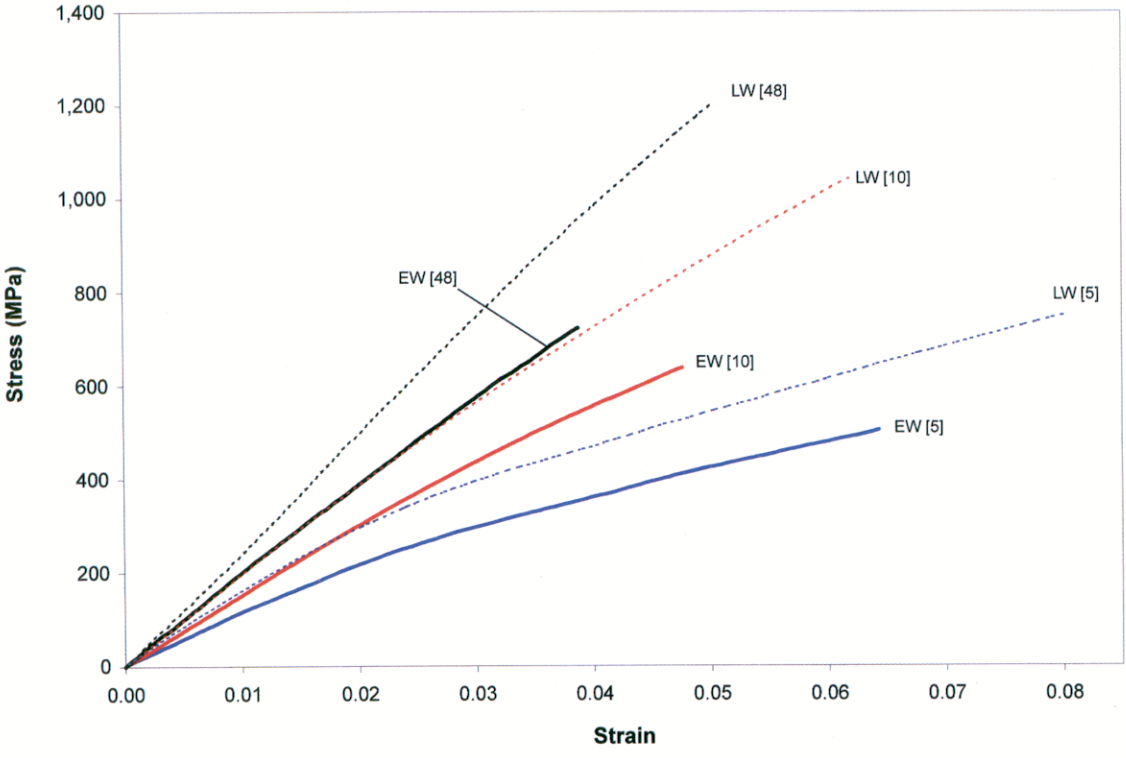

FIG. 3. Average stress-strain curves for latewood (dashed line) and earlywood (solid line) fibers from growth rings 5, 10, and 48. Each curve includes all specimens from a particular growth ring regardless of vertical location within the tree. Sample size for the 48th ring is based on 30 fibers; sample sizes for the 5th and 10th growth ring are based on 240 fibers.

height, with the uppermost disk consisting of only growth rings 5 and 10. The slivers were taken from the north compass dierction and separated into EW and LW fractions. Approximately three slivers per growth ring, tree height, and EW/LW fraction were macerated separately in a solution comprised of 1 part 30% hydrogen peroxide, 4 parts distilled water, and 5 parts glacial acetic acid. Macerated fibers were washed with distilled water and placed between glass slides to minimize twisting during drying. Two epoxy droplets were placed in the center portion of each fiber via forceps with an approximate spacing of 1 mm. The epoxy was allowed to cure at 60°C for 24 h followed by a minimum of an additional 24 h at 22°C.

Tensile tests

Tensile testing of individual fibers was conducted with a custom gripping assembly attached to a miniature materials tester. A dissecting microscope was used to place specimens in the gripping assembly as well as to remove them upon fiber failure. The crosshead rate of elongation was 80 microns per minute. Thirty each of EW and LW fibers were tested for each growth ring and height, for a total of 2,160 fibers. Span length was determined with a micrometer embedded in an ocular of the dissecting microscope. Fibers were removed from the tensile apparatus immediately upon failure and stored for subsequent cross-sectional analysis with the confocal scanning laser microscope (CSLM).

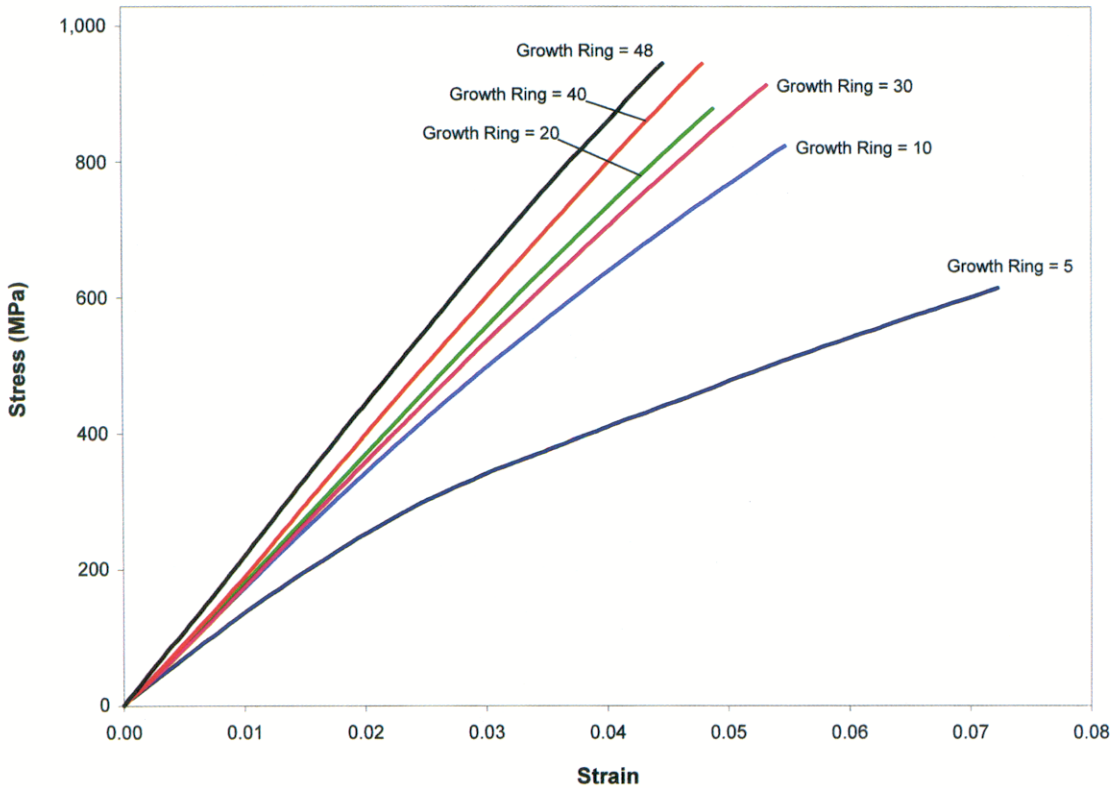

FIG. 4. Average stress-strain curves for all fibers from growth rings 5, 10, 20, 30, 40, and 48. Each curve includes all specimens from a particular growth ring regardless of vertical location within the tree and regardless of earlywood and latewood.

Confocal scanning laser microscopy

Failed fibers were stained with a dilute concentration of acridine orange (10 mg/300ml of distilled water) and mounted to a glass slide with the aid of tissue tack. Epoxy droplets were then removed with micro-scissors and discarded. The remaining fiber segments were mounted with permount and covered with a number 1 cover slip. Fiber cross-sectional (XS) areas were imaged with a Biorad Model 600 CSLM and a 100X oil-immersion lens. A standard image analysis software program was used to quantify the XS area from each reconstructed vertical line scan image. The XS area and span length were then used to convert the load-elongation curves into stress-strain curves. In addition to XS area, the CSLM was used to ascertain microfibril angle (MFA) on 10 of the failed fiber specimens per sample. The MFA was determined on the failed fiber ends (Groom et al. 2001).

RESULTS AND DISCUSSION

Mechanical properties

The average tensile span for all fibers tested in this study was 1.14 mm. Due to the shortness of the juvenile fibers, tensile spans for growth rings 5 averaged less than 1 mm. All other fibers were tested at a span of slightly greater than 1.2 mm. Stress-strain curves for all EW and LW fibers tested at growth rings 5, 10, and 48 averaged for all heights are shown in Fig. 3. The compliance associated with EW fibers was examined in the second paper of this series (Mott et al.

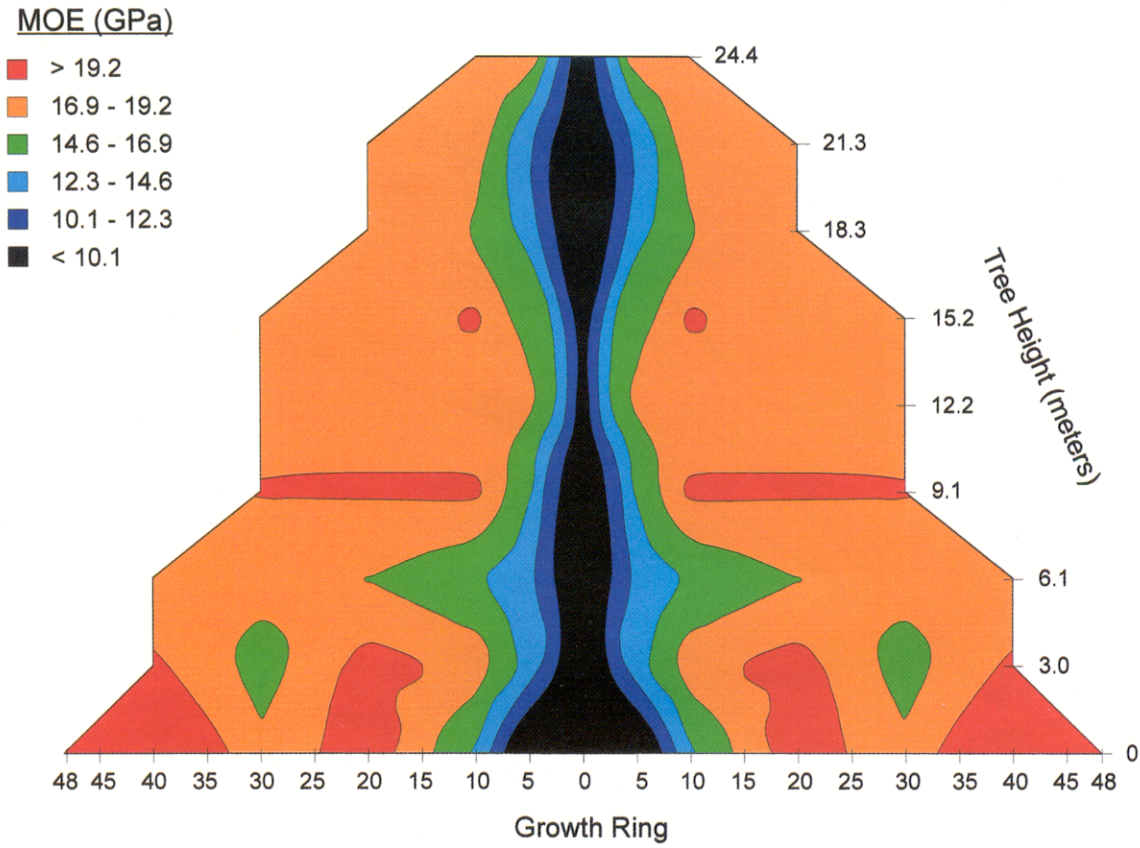

FIG. 5. Map of fiber modulus of elasticity shown as function of location within a tree.

2001) and is a result of the larger microfibril angles and pitting structure associated with EW fibers. The other growth rings showed similar trends.

Combined EW and LW data for each growth ring are shown in Fig. 4. The juvenility of growth ring 5 is evident by the shallow slope of the stress-strain curve. The stress-strain curve for growth ring 5 is different in shape from the rest of the growth rings; the curve is linear below 30% and above 60% of the ultimate tensile stress (UTS). The nonlinear portion of the stress-strain curve between 30 and 60% of the UTS is similar to that demonstrated by Page and El-Hosseiny (1983) and was postulated by Navi (1998) to be the series-coupling of localized damage. These small regions of localized damage are most likely the result of heavy pitting associated with juvenile fibers. Strains in these pit-associated regions have been calculated to be 300 times greater than in non-pit-associated regions (Mott 1996). This damage behavior is also evident to a lesser extent in the stress-strain curve of growth ring 10. Growth rings 20 and above demonstrate similar behavior; proportionality up to approximately 80% of UTS followed by nonlinearity to failure.

Tabular summaries of the engineering properties of fibers tested are presented in Tables 1 and 2. The average MOE and UTS for all fibers tested were, respectively, 17.3 Gpa and 824 MPa. The MOE of fibers from growth ring 5 were approximately 35% less than that of all other fibers. The UTS was similarly 40% less for fiber from growth ring 5 as compared to all other fibers. Stiffness

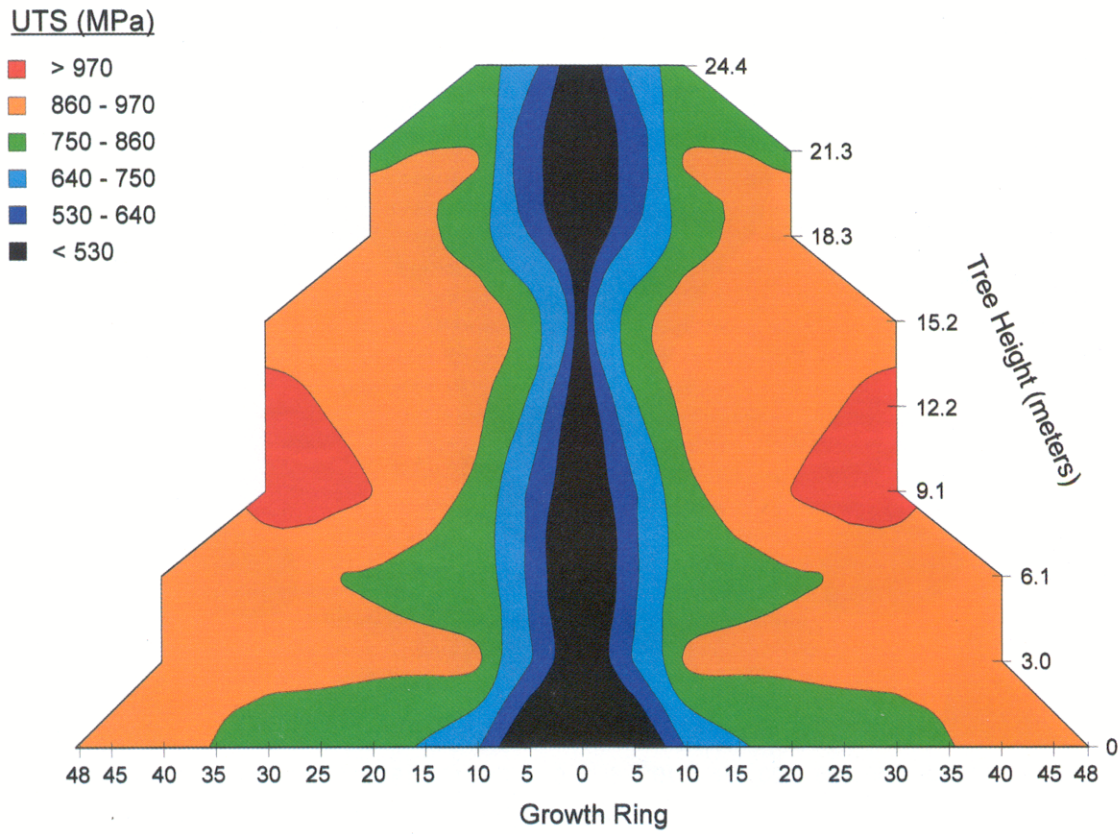

FIG. 6. Map of fiber ultimate tensile stress shown as function of location within a tree.

tended to increase with cambial age. In all growth rings studied, the average fiber strength increased with cambial age, reaching a peak near 965 MPa.

The variability of fiber MOE and UTS can be seen in Figs. 5 and 6, respectively. Data between the pith and growth rings 5 were extrapolated from existing data. The most compliant and weak regions of the tree were generally restricted to the first five to ten growth rings. The size of this juvenile region varied with height and reached a minimum approximately 40 to 50% of the total tree height. The size of the juvenile zone as characterized by fiber stiffness and strength increased near the base of the live crown. The transition zone between compliant and stiff fibers, as well as for weak versus strong fibers, is a very narrow band generally extending less than five growth rings.

Cross-sectional area

The average cross-sectional area for all fibers tested was 297 microns^2. A summary of cross-sectional area by growth ring and height is shown in Table 3. Cross-sectional area ranged from 184 microns^2 at growth ring 5, stump height to 380 microns^2 at growth ring 20, and a height of 3 m. The average cross-sectional area for fibers from growth ring 5 was no greater than 260 microns^2. With the exception of growth ring 10 at stump height, the average of all other growth rings was over 260 microns^2 and generally greater than 300 microns^2.

Figure 7 shows that cross-sectional area is linearly associated with cambial age. Although the variability is exceptional in the live crown and at stump height, the cross-sectional area

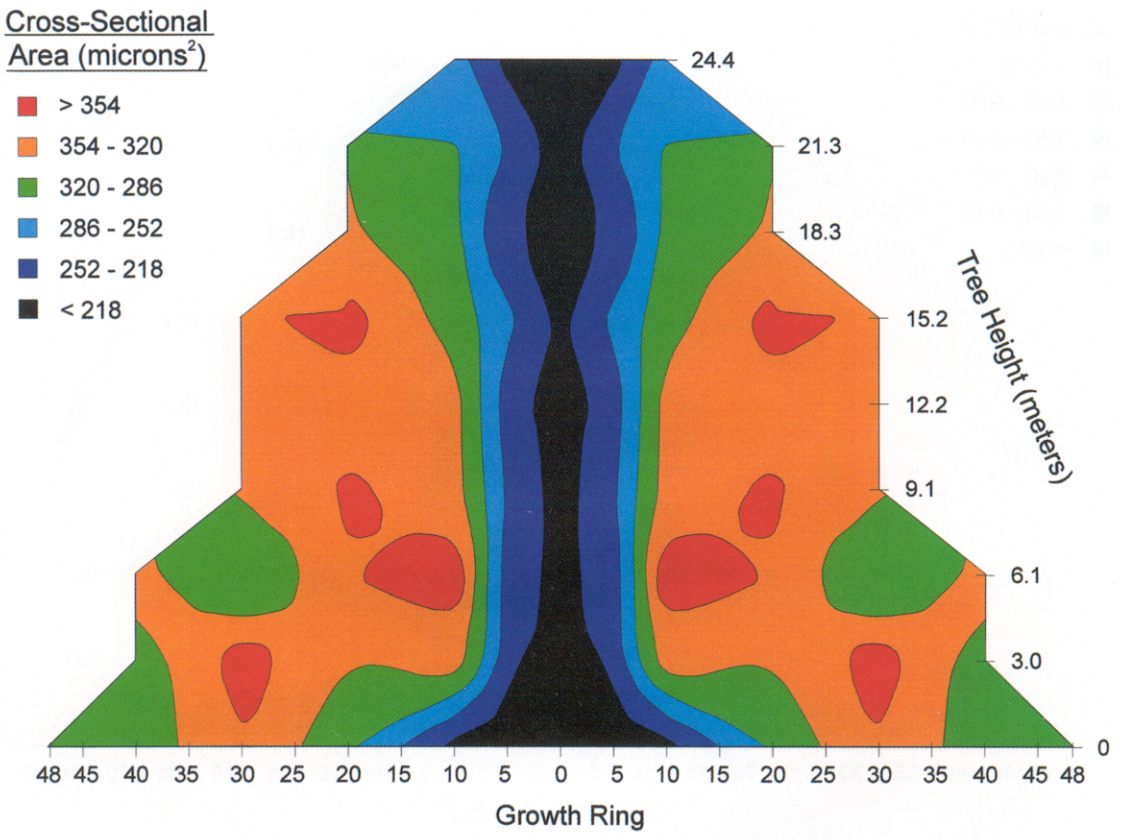

FIG. 7. Map of fiber cross-sectional area shown as a function of location within a tree.

of fibers is greatest beyond growth ring 10. There are minor deviations in cross-sectional area in the mature region of the tree, but Fig. 7 reflects a similar pattern as was found for fiber length by Wardrop (1951) (Fig. 2).

Microfibril angle

The average microfibril angle for all fibers tested was 15.4 degrees. Microfibril angles decreased from an average of 26.8 degrees for growth ring 5 down to 6.1 degrees for growth ring 48 (Table 4). The distribution of microfibril angles, summarized in Fig. 8, shows a pattern similar to fiber MOE (Fig. 3) and UTS (Fig. 4): Microfibril angle diminishes with cambial age and tree height up to approximately one half of overall tree height. The microfibril angle increases with height in the vicinity of the live crown, followed eventually by a decrease to a 10-cm top. A similar pattern has recently been observed by Megraw et al. (1999) for loblolly pine latewood fibers.

Overall normalized properties

The variability of data for fiber MOE, UTS, cross-sectional area, and microfibril angle is sufficient that characterization of specific zones is difficult. This variability can be minimized by merging the data for all four variables by some means of normalization. The normalized variable, referred to in this study as the Fiber Quality Index (FQI), will allow for quantifiable segregation into juvenile, transition, and mature zones. The technique of normalized weighted averages used in this study is summarized as:

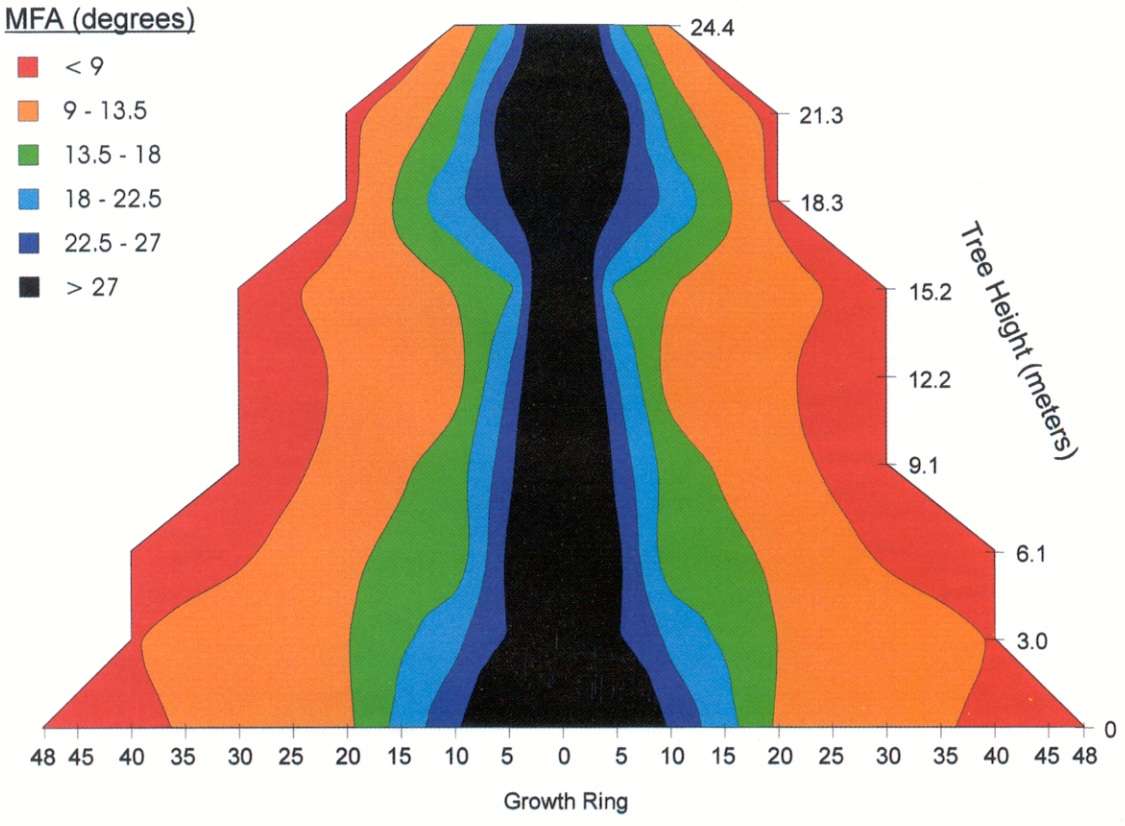

FIG. 8. Map of fiber microfibril angle shown as function of location within a tree.

$$FQI = \frac{MOE_i}{MOE_{Max}} \times \frac{UTS_i}{UTS_{Max}} \times \frac{XS_i}{XS_{Max}} \times \frac{MFA_{Min}}{MFA_i} \quad (1)$$

where: FQI = normalized value for a given growth ring and height; MOE_i = MOE for a given growth ring and height; MOE_{Max} = maximum MOE for all growth rings and heights; UTS_i = UTS for a given growth ring and height; UTS_{Max} = maximum UTS for all growth rings and heights; XS_i = cross-sectional area for a given growth ring and height; XS_{Max} = maximum cross-sectional area for all growth rings and heights; MFA_i = microfibril angle for a given growth ring and height; and MFA_{Min} = minimum microfibril angle for all growth rings and heights. Theoretically, values for FQI can range between 0 for fibers with no desirable properties to 1 for 'superior' fibers. The average value for FQI for all fibers tested was 0.71. The minimum and maximum average FQI values for each growth ring and height were found at stump height, respectively at growth ring 5, FQI = 0.31 and growth ring 48, FQI = 0.95. Average FQI values for all heights at growth rings 5, 10, 20, 30, 40, and 48 were respectively, 0.51, 0.69, 0.79, 0.82, 0.85, 0.95.

Graphical representation of weighted, overall normalized properties (Fig. 9) shows that the juvenile core is not vertically distributed as was previously reported by Bisset and Dadswell (1949); Zobel et al. (1959); and Zobel and Talbert (1984). There were shifts in FQI at approximately 0.6 and 0.7; thus those values were chosen to demarcate the zones of juvenility, transition, and maturity in Fig. 9. There

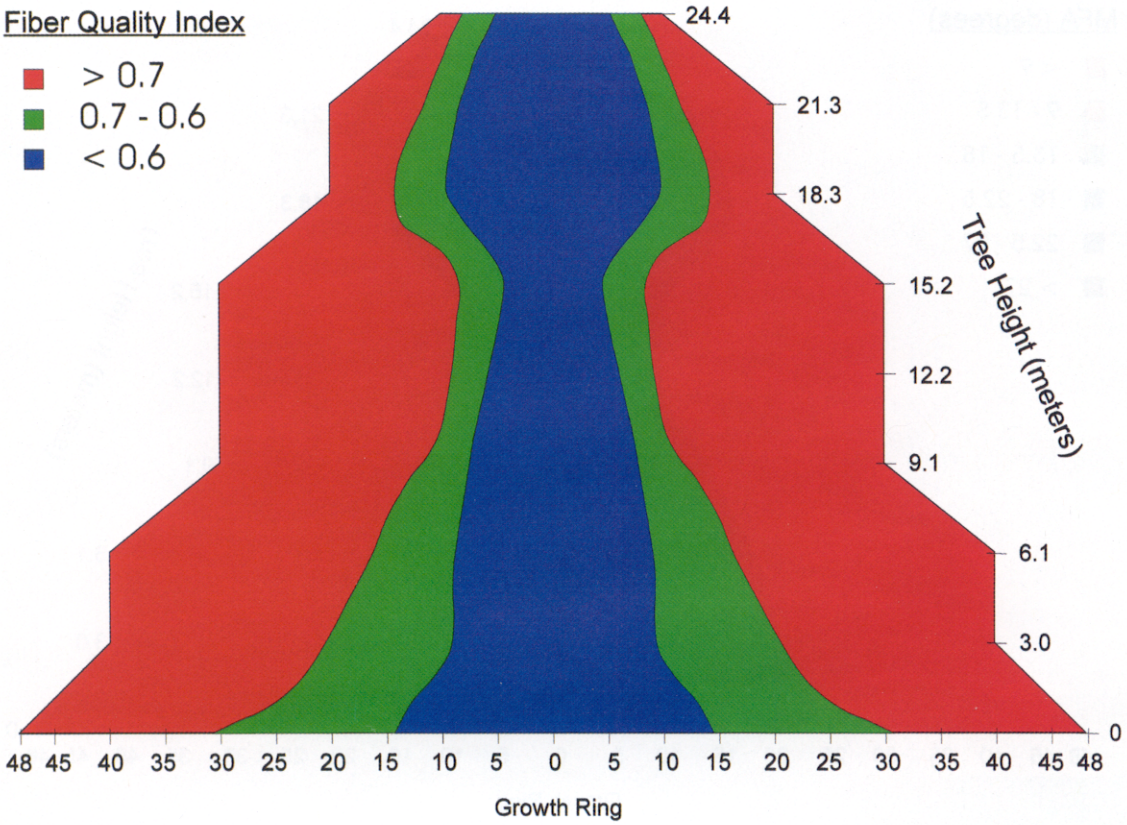

FIG. 9. Graphical representation of good, medium, and poor zones of fiber physical and mechanical properties. These zones were based on equally weighted and normalized values of modulus of elasticity, ultimate tensile stress, microfibril angle, and cross-sectional area.

exists a physiological response of fiber physical, and thus mechanical, properties of growth response factors associated with initial tree growth and with the development of stem wood. The juvenile core varied widely in this study, ranging from a maximum of growth ring 14 at stump height to less than 5 growth rings slightly below the live crown. There are two regions along the main stem that promote juvenility: stump height and the live crown. The result is a juvenile zone that is bioconical; tapering from stump to the live crown and a similar taper from the crown to the tip of the main bole.

The transition zone in Fig. 9 also is not a cylindrically-shaped core from stump to tip, but closely parallels the response of juvenility. The cambial age representing the shift from transition wood to mature wood is approximately twice that of the outer edge of the juvenile zone. This is true regardless of vertical location of fibers within the tree.

The greatest proportional volume of mature fibers exists between 30 and 50% of total tree height (Fig. 9). Megraw et al. (1999) also showed a similar pattern with clearwood MOE, with 30-year-old loblolly pine trees reaching maximum stiffness furthest from the pith and 9 feet from the stump. This maximal region of maturity lies equidistant from the stump and live crown, both of which have been shown to have poor mechanical properties (Megraw et al. 1999; Mott et al. 2001).

SUMMARY AND CONCLUSIONS

The mechanical properties of individual earlywood and latewood loblolly pine fibers were determined at various locations within a tree. These results, in conjunction with physical properties, were discussed in detail and then classified into juvenile, transition, and mature zones.

The stress-strain curves of juvenile fibers show proportionality between 30 and 70% of the ultimate tensile stress of the fiber and are otherwise curvilinear. Fully mature fibers are linear throughout the entire stress-strain curve. The average modulus of elasticity and ultimate tensile stress of all loblolly pine fibers, in equal proportion of earlywood and latewood, was 17.3 GPa and 824 MPa, respectively. Fiber stiffness and strength increase with cambial age. The average cross-sectional area for all fibers was 297 microns^2, ranging between 184 and 380 microns^2. The overall average microfibril angle was 15.4 degrees. Average microfibril angles for growth rings 5 and 48 were, respectively, 26.8 and 6.1 degrees.

Normalization of all properties by means of a Fiber Quality Index shows that 3 distinct zones occur within the main stem of the tree: juvenile, transition, and mature. The juvenile zone is comprised of fibers that are thin-walled, possess high MFAs and minimal stiffness and strength. This juvenile zone is not cylindrical as is generally reported but is biconical; tapering from stump to below the live crown and then again from the crown to the tip. This response is most likely a result of physiological responses to tree growth. The transition zone parallels in shape the juvenile zone and is between 3 to 15 growth rings in width, with the width of the transition zone dependent on vertical location. The mature zone radiates outward at various distances from the pith, reaching a maximum width between the stump and live crown.

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