Chapter 12

Selected Mesostructure Properties in Loblolly Pine from Arkansas Plantations

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Abstract

Design properties of wood are currently established at the macroscale, assuming wood to be a homogeneous orthotropic material. The resulting variability from the use of such a simplified assumption has been handled by designing with lower percentile values and applying a number of factors to account for the wide statistical variation in properties. With managed commercial forests geared toward rapid growth and shorter rotation harvests, wood products now contain significantly fewer and more widely spaced growth rings, further stretching the validity of the assumption of homogeneous behavior. This chapter reports on preliminary results of a study on measuring the property differences and variability of earlywood and latewood (mesostructure) samples from a commercial loblolly pine plantation. Novel testing procedures were developed to measure properties from 1- by 1- by 30-mm mesostructure specimens. Properties measured included longitudinal modulus of elasticity, shear modulus, specific gravity, and microfibril angle. The test results showed dramatic differences in the properties of adjacent earlywood and latewood, differences that are believed to influence product performance. As important as the data that documents the property differences is the information on the variability of these properties.

Keywords: juvenile wood, southern pine, modulus of elasticity, shear modulus, microfibril angle, early wood properties, latewood properties, micro-testing

Introduction

We are living in a time of human-made materials that are designed or engineered at the micro or nano level for consistency and performance. Wood, however, is a material that has been "designed" and manufactured at the micro level by biological processes for performance as a tree and not as a board. Growing, harvesting, sawing, and grading technologies work together to render wood material readily usable by others, but in these processes wood is largely considered to be homogeneous and uniform. Converting a material optimized for use as a tree into a board results in widely variable values of stiffness, strength, and dimensional stability. To improve the utilization of wood products,

it is necessary to study and understand this variability. With this understanding we will be able to model wood from a mechanic's point of view and optimize its use in products.

A visual feature of wood structure that contributes to variability in properties in many coniferous species, especially pines, is the presence of annual growth rings. These rings are not completely uniform in width and often provide a record of annual and seasonal weather- and climate-based events that affect growth and the formation of earlywood and latewood bands. The earlywood band, a tapered cylindrical layer, is formed in the early part of the growing season; the latewood band is formed later in the season (Larson 1969). Silviculture practices also affect the formation of growth rings. The trend toward managed tree plantations has generally resulted in wider rings with greater proportions of earlywood. These are expected outcomes from strategies that include thinning, pruning, and fertilization.

Earlywood and latewood bands represent the *mesostructure* of wood. The *microstructure* consists of individual cells. Typical *macrostructure* assumptions in which wood is assumed to be a homogeneous, orthotropic continuum ignore the growth rings. As rapidly grown plantation wood becomes an increasing part of the wood resource for the United States, a greater proportion of juvenile wood (crown-formed wood near the pith) with fewer rings per inch raises new challenges for producing the highest quality wood products. Variability in performance and properties has become a much larger issue for less tolerant customers who have more choices of competing materials.

The mechanical properties of earlywood are significantly different than those of latewood. The variations in earlywood and latewood mesostructure properties have not been extensively determined and the resulting impact on product performance has not been defined. Measurement of mesostructure properties and development of a means to predict their values can lead to an understanding of the role of these properties in wood product quality and performance.

The immediate objective of the study reported here is to measure the individual elastic properties of matched earlywood and latewood specimens. The longer-range objective is to develop a foundation for property predictions and mechanical modeling. This chapter reports on a selected portion of the work completed to date. More statistically rigorous articles are planned for future publication (Cramer et al. 2005). In this chapter, we present data defining modulus of elasticity, shear modulus, and related properties of earlywood and latewood in loblolly pine (*Pinus taeda* L.) from a plantation in Arkansas.

Background

Earlywood formation tends to begin abruptly in the cambium, prompted by bud activity in the spring and proximity to foliage organs (Larson 1969). In the early part of the growing season, cells are formed rapidly; these cells, which have large lumens and small cell walls, form the earlywood portion of the growth ring. Once activated, cambial activity continues through the growing season. The transition from earlywood to latewood is gradual, whereas the transition from latewood of the previous season to earlywood is very abrupt. The width of the latewood portion of a ring tapers upward in the stem, reaching a point of extinction at the apex.

Radial diameter and secondary wall thickness are the main characteristics that distinguish earlywood from latewood. These two characteristics can be altered independently. Although there is a general understanding of the difference between earlywood and latewood, there is no definition of latewood tracheids that satisfies all conditions. Some definitions of latewood tracheids do not apply to juvenile wood.

The existing literature lacks data on variability and changes in specific gravity, modulus of elasticity, shear modulus, and microfibril angle of earlywood and latewood around the stem of the tree. Considerable work has focused on the specific gravity of earlywood and latewood (Pew and Knechtges 1939; Paul 1958; Goggans 1964; Megraw 1985; Hodge and Purnell 1993; Ying et al. 1994). Biblis (1969) found considerable variability in specific gravity and modulus of elasticity of latewood. Biblis (1969) and Megraw (1985) both discussed a transitional zone between earlywood and latewood zones. They found that properties within this zone showed a gradual change from typical earlywood values to typical latewood values. Recently, the specific gravity and modulus of elasticity of individual earlywood and latewood fibers bas been measured (Groom, Mott and Shaler 2002; Groom, Shaler and Mott 2002; Mott et al. 2002). Literature on earlywood and latewood research is described in detail in Larson et al. (2001) and Cramer et al. (2005).

Methods

Specimen preparation

Samples were taken from loblolly pine (*Pinus taeda* L.) trees on approximately 32 ha (80 acres) of commercial plantation in Arkansas. The fertilization and pruning history of the plantation was recorded as well as the location and orientation of each stem. Twenty bolts were taken from 10 trees. Two 1.5-m (5-ft) bolts were collected from each tree, one at breast height (1.2 m, or 4 ft) and the other approximately 6 m (20 ft) from the ground (Figure 12.1). The bolts were shipped to the Forest Products Laboratory (FPL) in Madison, Wisconsin.

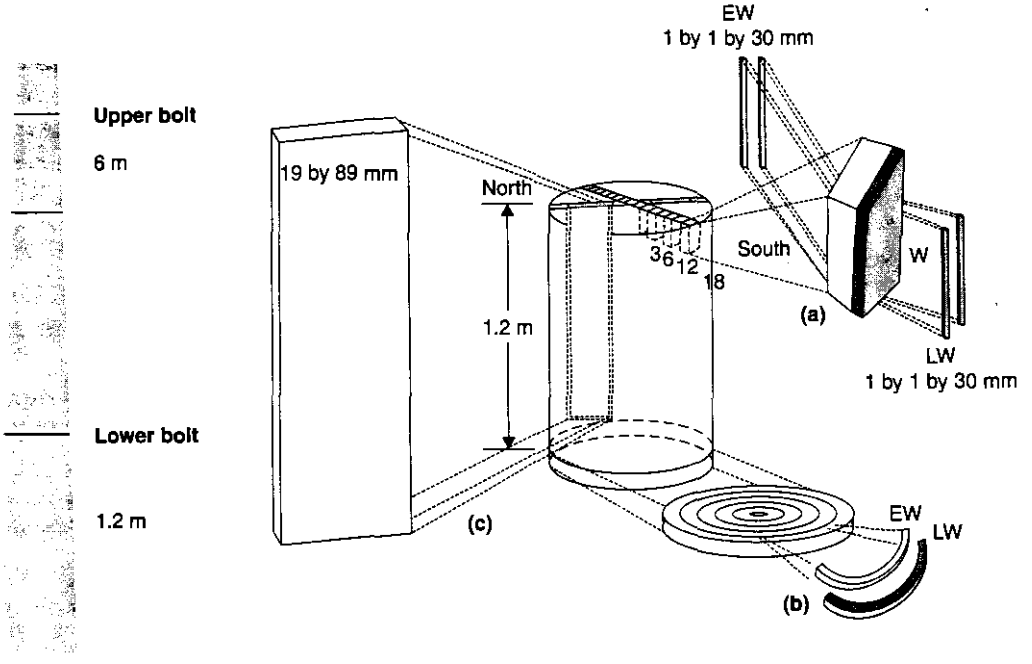


Fig. 12.1 Pattern for cutting specimens from 1.5-m (5-ft) loblolly pine bolt: (*a*) Set 1 : small rectangular earlywood and latewood specimens for longitudinal modulus of elasticity (E) and shear modulus (G); (*b*) Set 2: arcs of earlywood and latewood from disk for tangential E; (*c*) 19- by 89-mm (nominal 1- by 4-in.) board for full-size stability measurements.

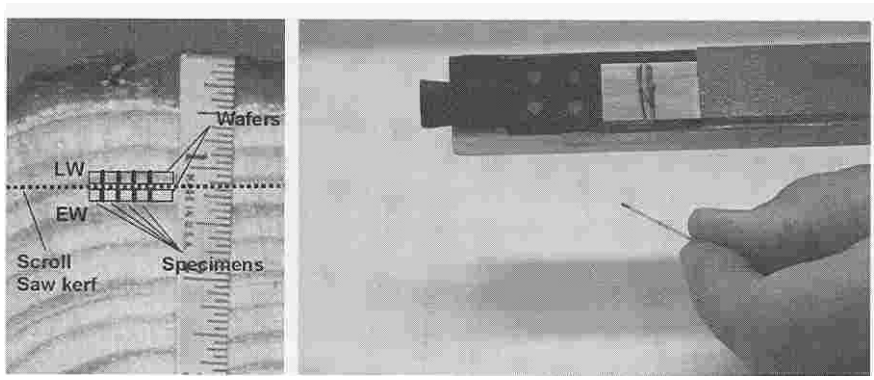


Fig. 12.2 *Left,* adjacent latewood (*LW*) and earlywood (*EW*) specimens were obtained for modulus of elasticity (E) and shrinkage evaluations. *Right* specimens were cut using a 1-mm-thick wafer held in place by a vacuum block. (This figure also is in the color section.)

Two disks were removed from the top and bottom of the bolts. The disks were cut into straight specimens and arcs. The remaining portion of the bolts was cut into 1/4-circle wedges and full-size boards (Figure 12.1). The specimens were stored under controlled environmental conditions. Samples from 10 bolts derived from 6 trees are reported here.

The straight toothpick-size (1- by 1- by 30-mm, or 0.039- by 0.039- by 1.18-in.) specimens were cut from adjacent earlywood and latewood (Figure 12.2). The earlywood and latewood bands were separated into wafers for each growth ring by cutting along a line with a scroll saw. The kerf of the saw blade essentially eliminated the transition zone between the earlywood and latewood zones. The kerf was initially 3.2 mm (0.125 in.) and later reduced to 0.5 mm (0.02 in.). Excess material was removed until the wafer appeared to be composed completely of a light-colored band of earlywood or dark-colored band of latewood.

Specimens were manufactured from individual earlywood and latewood bands of rings 3, 6, 12, and (where possible) 18. Four sets of earlywood and latewood specimens were prepared corresponding to the north, south, east, and west sides of the bolt.

Testing methods

The straight specimens were tested to determine modulus of elasticity (MOE) and shear modulus (G) by using a unique micromechanical testing device. A broadband viscoelastic spectroscopy (BVS) instrument, previously developed to study other viscoelastic materials like bone and tin, was used to determine moduli and loss tangent values (Brody et al. 1995; Chen and Lakes 1989). This instrument was chosen because of its capacity for small-dimension specimens and its capability of measuring very small strains, on the order of 10^{-5}. A simplified schematic of the BVS device is shown in Figure 12.3.

Each specimen was glued with cyanoacrylate to a brass support rod on one end and a magnet on the other, forming a fixed–freecantilevered beam with the magnet on the free end. The magnet was centered between two pairs of Helmholtz coils, one pair for bending and the other for torsion. The coils were excited by a function generator with a known sinusoidal voltage producing an electric field that caused the magnet and thus the specimen to cyclically deflect. Deflection was measured by reflecting a laser beam off a mirror, which was glued to the magnet, onto a light detector. Knowing

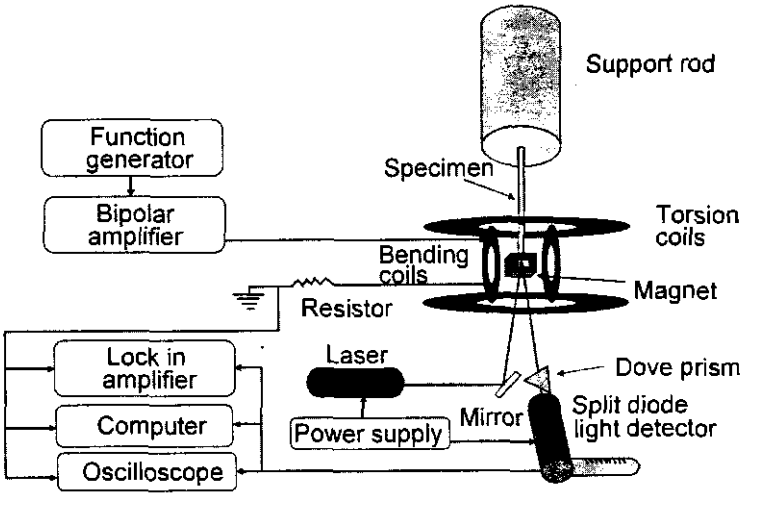


Fig. 12.3 Schematic of broadband viscoelastic spectometry device. (This figure also is in the color section.)

the force and the amount of maximum deflection the moduli could be calculated using equations developed for a fixed–freecantilevered beam.

The elastic properties were determined by collecting information on specimen dimensions, calibration constants for split diode angle detector, magnet calibration, distance between specimen and detector, feedback resistance, torque or bending signal (in volts), and deflection angle signal (in volts), and by using basic elastic theory describing the deflection of a fixed–freerod.

The mounted specimens were stored in an environmental chamber until testing. Relative humidity was controlled by using an evaporating salt bath of +99% sodium bromide in water. This maintained a stable relative humidity of 55% and was used as the target condition during testing to help reduce the amount of drift caused by specimen shrinkage or swelling.

Needle values were used to control the mixture of dry and humid air in the chamber to within ±10% of 50% relative humidity. Pressurized air was sent through a cylinder of gypsum (anhydrous calcium sulfate) desiccants to create dry air or through a 500-mL flask of water to create saturated air. To monitor conditions within the test chamber, temperature and relative humidity sensors were placed next to the test specimen. A series of preliminary tests determined that the change in measured modulus of elasticity resulting from a 10% change in relative humidity was small, and more precise controls were not deemed necessary.

Specimen dimensions were established using an optical stereomicroscope, at 64× magnification, which featured a moveable stage linked to a digital display with accuracy to 2.54×10^{-4} mm (10^{-5} in.). The width of the radial and tangential faces was measured at 5-mm (0.20-in.) intervals along the length of the specimen. The average width of each face of the cross section was used in the equations for modulus of elasticity and shear modulus.

Each specimen was subjected to longitudinal modulus of elasticity (MOE_L) tests three times to minimize test-induced variability. For example, this resulted in 48 separate tests of earlywood MOE_L for bolt 1. A similar sequence was used to establish shear modulus ($G_{L\wedge}$). Specific gravity and microfibril angle were also measured. Over 3000 individual tests were conducted.

Specific gravity was measured using oven-dry weight and green volume. Specimens were dried for 24 hours at 40°C (105°F). Specimens were spread out evenly in the oven to allow for sufficient airflow

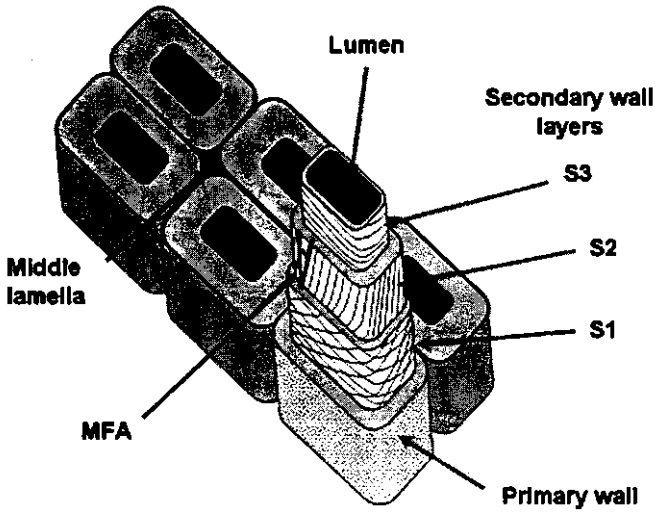


Fig. 12.4 Anatomy of a wood cell. Gray lines in secondary wall layers represent idealized cellulose microfibrils. The angle formed by microfibrils in the S_2 layer, called the microfibril angle (MFA), plays a crucial role in determining wood stiffness. (This figure also is in the color section.)

around them. A forceps was used to remove the specimens from the oven and onto an electronic balance with a resolution of ±0.00001*g*. To obtain green volume, specimens were stored in water for 24 hours to assure complete saturation. Volume was measured by stereomicroscope, as previously described.

Wood exhibits hierarchical structure. It is a layered composite of polymeric cellulose microfibrils embedded in a matrix of hemicelluloses and lignin. The stiffness of wood is derived from semi-crystalline cellulose microfibrils wound in a left-handed helix around the lumen, the center of each tube-shaped wood cell. Wood cells, or tracheids, consist of multiple layers: a primary wall (the most external layer) and three secondary layers (S_1, S_2, and S_3), which are successively positioned toward the lumen (Figure 12.4). Cells are connected to each other by the middle lamella. The thickest and most critical of the secondary layers is the S_2 layer. The microfibril angle (MFA), the angular deviation of microfibrils in the S_2 layer relative to the longitudinal cell axis, plays a crucial role in determining the mechanical behavior of wood (Bendsten and Senft 1986; Walker and Butterfield 1995).

The MFA was measured using X-ray diffraction. Fibers contained in the straight specimens were irradiated perpendicular to the fiber length by a narrow, monochromatic X-ray beam. The method used to translate the X-ray diffraction data to MFA measurements was previously developed by Kretschmann et al. (1998) and Verrill et al. (2001). A diffraction pattern was produced by the crystalline cellulose structure and recorded by an electronic detector. This pattern consisted of a series of arcs that were spaced apart by a number of well-defined concentric circles with bright spots. The diameter of each concentric circle indicated the spacing of the crystalline planes within the cellulose crystalline fibrils. The position of the bright spots and intensity of these concentric circles were used to estimate MFA.

Results and discussion

Because of biological activities in the tree, the properties of wood at higher portions (upper bolts) of the stem are different than those of wood located near the base (lower bolts). Consequently, all

data presented is separated into two categories, lower and upper bolts. Lower bolts were taken near the base at a height of approximately 1.2 m (4 ft). Upper bolts were taken approximately 6 m (20 ft) from the base; an intermediate height of 3.4 m (11 ft) was included in this category.

Modulus of elasticity

The summary box plots for earlywood and latewood modulus of elasticity (MOE) are shown in Figure 12.5. These and similar box plots (Figures 12.6–12.10) show the outlying data ("outliers"),

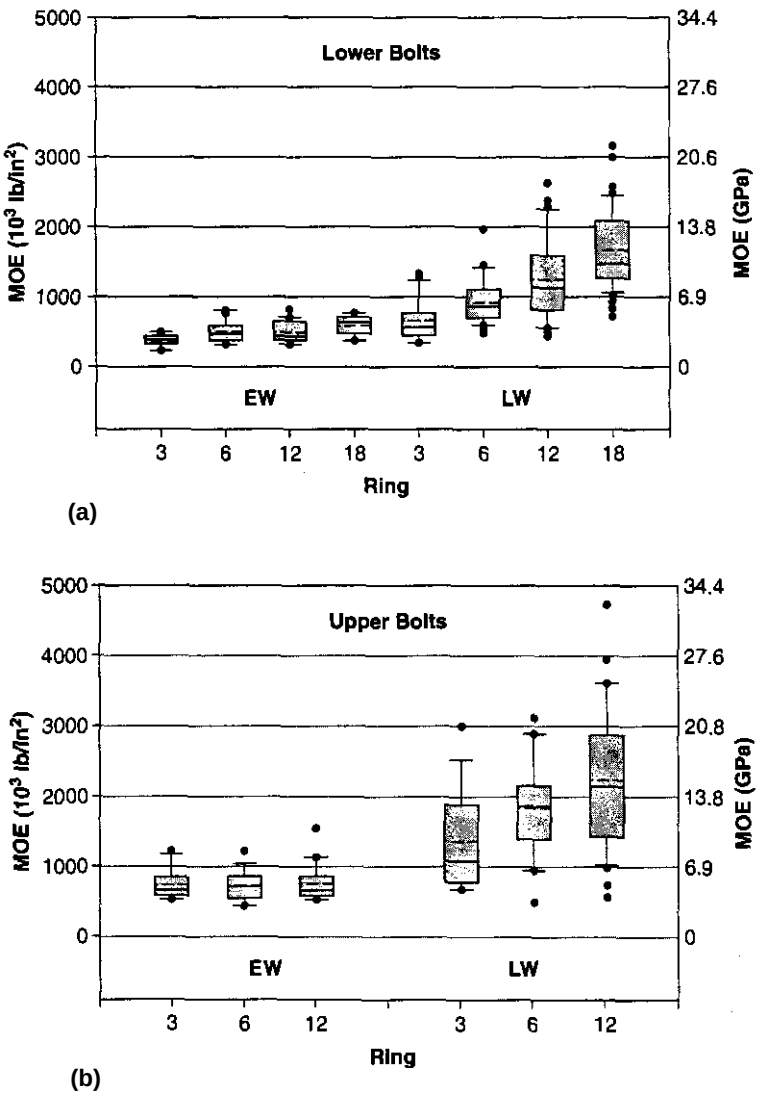


Fig. 12.5 Earlywood and latewood modulus of elasticity (MOE) for lower and upper bolts. Box plots show outlying data *(dots);* 5th, 25th, 50th, 75th, and 95th percentiles *(solid lines);* and mean values *(dashed lines).*

the 5th, 25th, 50th, 75th, and 95th percentiles (solid lines), and the mean (dashed lines). The data show that earlywood MOE increased slightly from pith to bark. Average earlywood MOE for all specimens was 4.2 GPa (608×10^3 lb/in^2) with a coefficient of variation (COV, standard deviation divided by mean) of 35%. Earlywood MOE also increased with bolt height. Average MOE was 5.1 GPa (737×10^3 lb/in^2) with a COV of 29% for the upper bolts and 3.5 GPa (511×10^3 lb/in^2) with a COV of 29% for the lower bolts.

Over twice as many tests of latewood were conducted to substantiate trends, because the variability in the latewood was considerably greater than that in the earlywood. The latewood MOE values showed a much more pronounced trend of increasing MOE with increasing distance from the pith. The MOE of the outer growth ring was almost 2.5 times greater than that of other selected rings in the lower bolts and over 60% greater in the upper bolts. For all specimens, average latewood MOE was 9.9 GPa (1.433×10^6 lb/in^2) with a COV of 53%. Average MOE was 13.0 GPa (1.887×10^6 lb/in^2) with a COV of 43% for the upper bolts and 8.1 GPa (1.176×10^6 lb/in^2) with a COV of 50% for the lower bolts.

Shear modulus

Average earlywood shear modulus (G) for all specimens was 0.8 GPa (114×10^3 lb/in^2) with a COV of 29% (Figure 12.6). The values for G remained rather constant with increasing distance from the pith. Earlywood specimens taken from upper bolts usually had smaller G values than specimens taken from similar ring positions in lower bolts. Average G was 0.7 GPa (97×10^3 lb/in^2) with a COV of 21% for the upper bolts and 0.9 GPa (125×10^3 lb/in^2) with a COV of 28% for the lower bolts.

Average latewood G for all specimens was 1.6 GPa (237×10^3 lb/in^2) with a COV of 31%. Bolt height also influenced latewood G, but again the trend was opposite that for MOE. Average G was 1.6 GPa (229×10^3 lb/in^2) with a COV of 31% for the upper bolts and 1.7 GPa (242×10^3 lb/in^2) with a COV of 31% for the lower bolts. As with MOE, latewood showed a more pronounced trend of increasing G with increasing distance from the pith than did earlywood. The increase in G was relatively greater in the lower bolts (60%) than in the upper bolts (35%).

Specific gravity

Specific gravity values for earlywood were remarkably consistent, averaging 0.30 with a COV of 20% (Figure 12.7). For latewood, overall average specific gravity was 0.56 with a COV of 19%. Specific gravity values were much more variable for latewood rings than for earlywood rings; specific gravity increased 30% to 50% with increase in distance from the pith.

Microfibril angle

Microfibril angle (MFA) showed considerable variability at all levels (Figure 12.8). The lower bolts exhibited lower variability than did the upper bolts. Height of bolt had a significant effect on MFA: upper bolts had considerably lower MFA values. Average MFA for upper bolts was 19° with a COV of 35%, whereas that for lower bolts was 34° with a COV of 19%. The MFA decreased from 10% to 25% with increasing distance from the pith for both earlywood and latewood and upper and lower bolts. For both earlywood and latewood, the cell structure seemed to he mature by ring 18, which had a considerably lower MFA compared to that of the other rings.

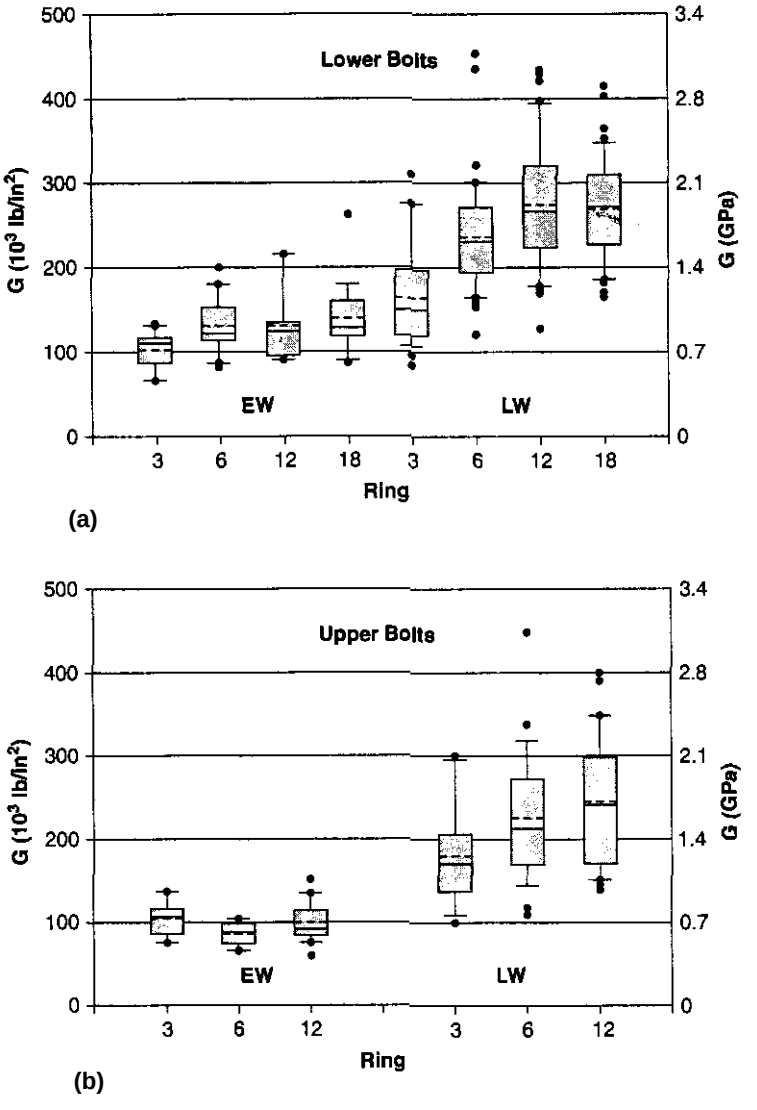


Fig. 12.6 Earlywood and latewood shear modulus (G) for lower and upper bolts.

Ratio of latewood to earlywood MOE

Conifers with a pronounced annual ring mesostructure can be thought of, in the extreme, as rigid latewood cylinders spaced apart by low density, low stiffness earlywood foam. Mechanically such a structure would resist loads much differently than would the assumed homogeneous material. Our test results confirmed that the elastic properties of earlywood and latewood in loblolly pine are significantly different. Figure 12.9 shows box plot representations of the MOE and G ratios of each set of adjacent latewood to earlywood specimens. These ratios for all specimens ranged from 0.8 to 6.5, with an average of 2.3 and a COV of 51%.

The average ratio of latewood to earlywood MOE (2.7) was greater in the upper bolts than in the lower bolts (2.1). The ratio of latewood to earlywood also increased from the pith outward from an

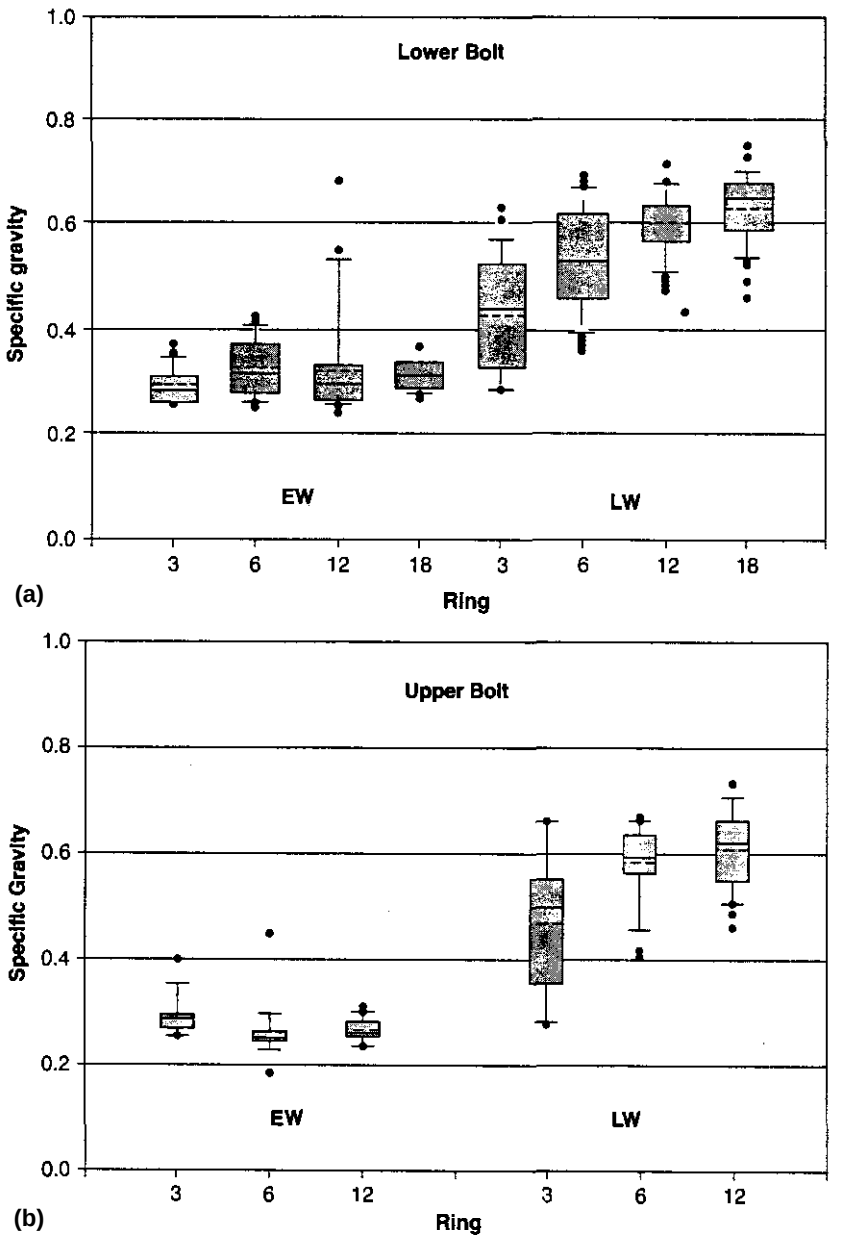
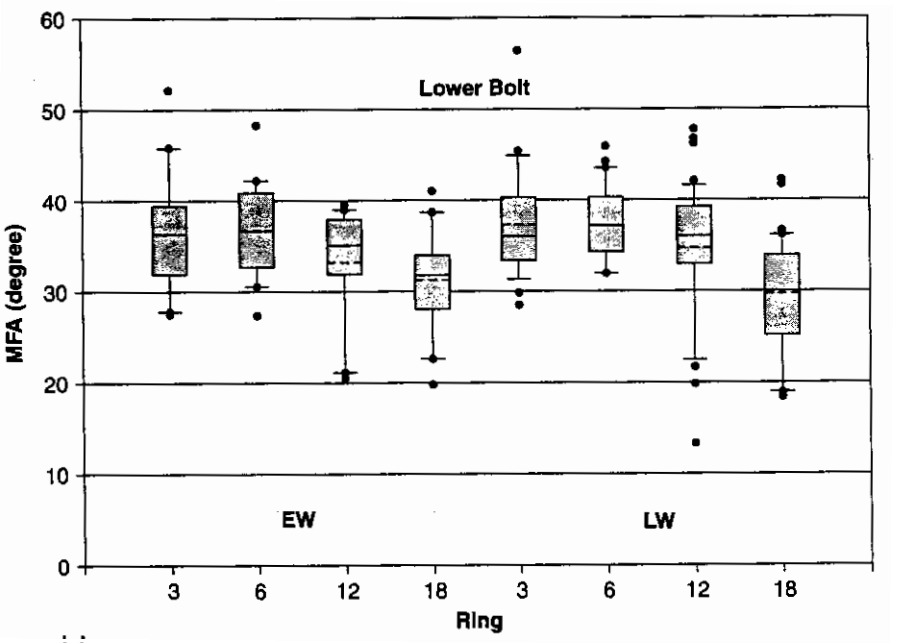


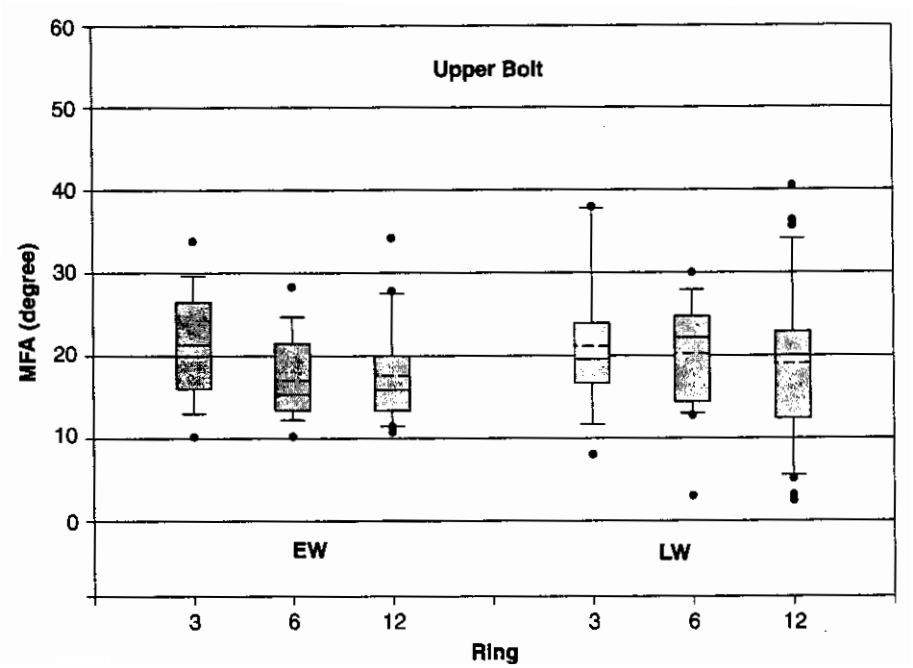
Fig. 12.7 Earlywood and latewood specific gravity for lower and upper bolts.

average value of 1.6 in ring 3 to 2.7 in ring 18. Latewood represented 27% of the cross-sectional area for rings 3, 6, 12 and 18 in the bolts tested.

While the difference between earlywood and latewood is not rigorously defined, latewood is generally described as having thicker cell walls and smaller lumens. Although the MOE values of latewood were several multiples greater than those of earlywood, the larger cross-sectional area occupied by earlywood in the mesostructure suggests that its mechanical role relative to latewood should be considered.

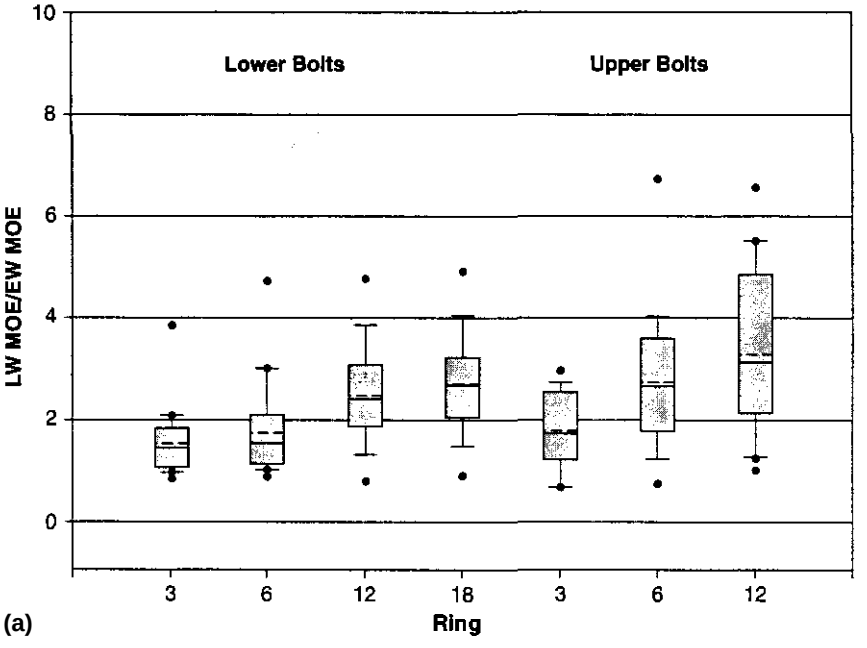


(a)



(b)

Fig. 12.8 Earlywood and latewood microfibril angles (MFA) for lower and upper bolts.



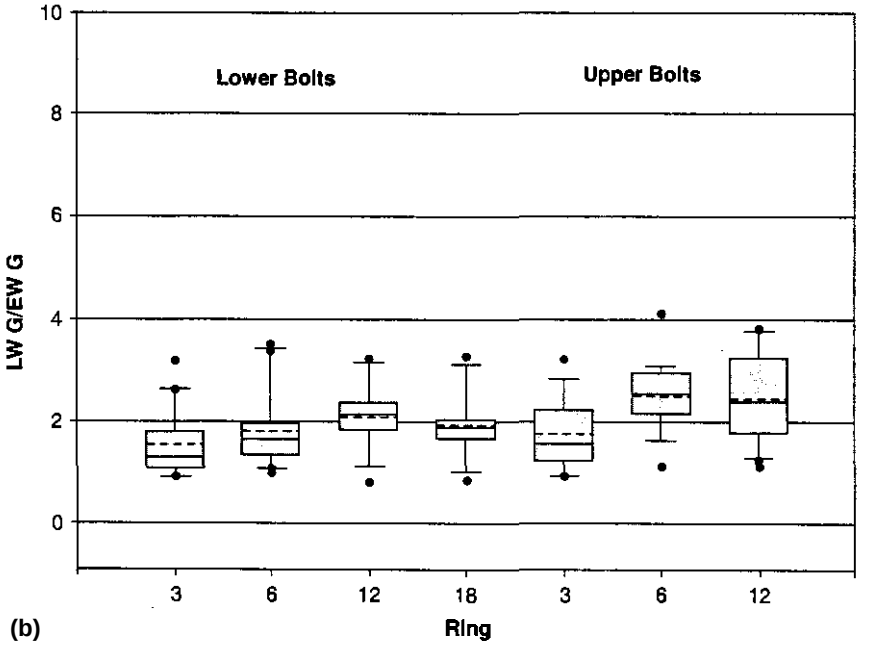


Fig. 12.9 Ratio of latewood to earlywood MOE and latewood to earlywood G.

Ratio of MOE to shear modulus

The averages and trends for the ratio of MOE to shear modulus (G) were similar for earlywood and latewood; therefore the earlywood and latewood data were combined. The ratio of MOE to G for all samples tested was smaller in the lower bolts (Figure 12.10). The MOE/G ratio averaged from 4 to

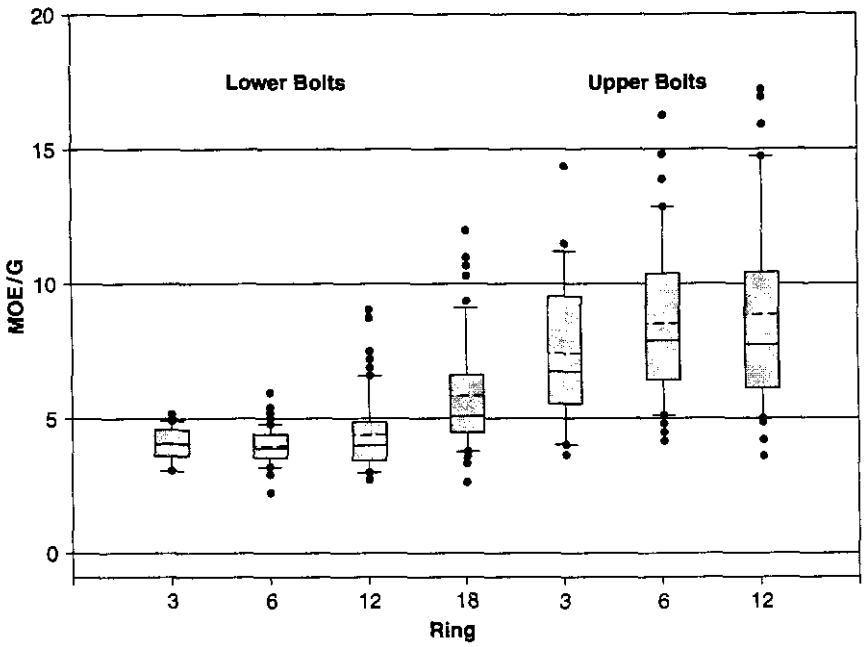


Fig. 12.10 Ratio of MOE to G for all samples tested.

6 in the lower bolts compared to 7.5 to 9 in the upper bolts. These values are considerably different than the MOE/G ratio of 16 specified in ASTM D 2915 (ASTM 2004).

Earlywood elastic properties

Earlywood shear modulus (G) appears to have a linear relationship with earlywood MOE (Figure 12.11). hut the relationship depends upon the height of the wood in the stem. The scale for Figure 11 is set for ease of comparison with latewood results. For a given MOE value, G was much greater for the lower bolts than for the upper bolts. There was considerable variability in the relationship between MOE and G within bolts and from bolt to bolt.

Earlywood specific gravity was not a good predictor of earlywood MOE, as shown in Figure 12.12; a similar lack in trend was observed for specific gravity and G (not shown). No meaningful trends in the relationship of MOE to specific gravity were identified in lower bolts compared to upper bolts.

Microfibril angle appeared to be a better predictor of earlywood MOE MFA followed the same general trend in lower and upper bolts, despite considerable variability in values (Figure 12.13). Microfibril angle by itself could not be considered an accurate predictor of earlywood MOE. There was no clear trend in the relationship between earlywood G and corresponding MFA, as shown in Figure 12.14. Individual bolts did not follow the overall trend, as indicated by bolt 18 (Figure 12.14).

Latewood elastic properties

Latewood showed a confused relationship between G and MOE. The slope of this relationship was clearly different for lower and upper bolts (Figure 12.15); results for lower bolts were more variable

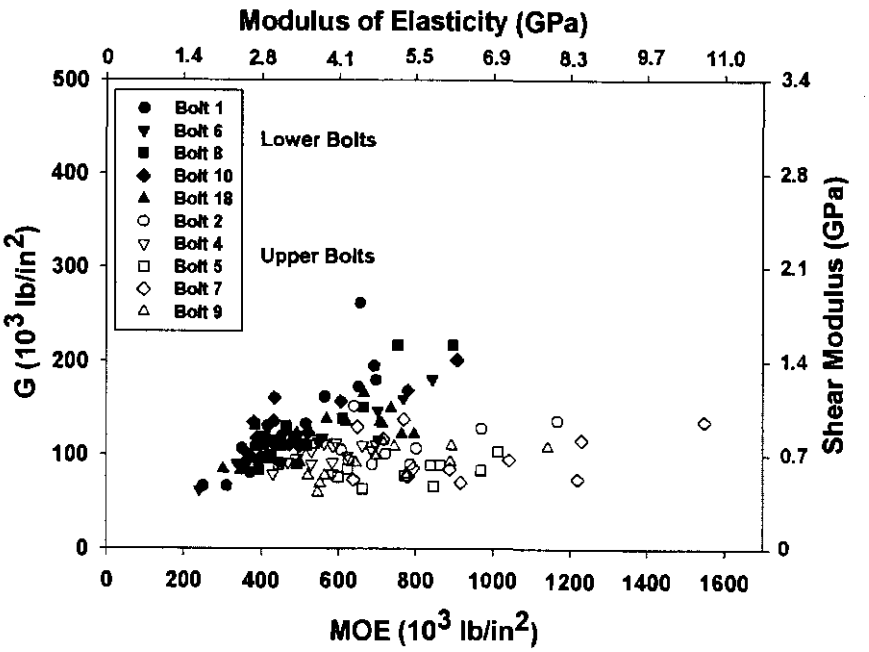


Fig. 12.11 Relationship of earlywood G to MOE.

than those for upper bolts. Latewood MOE showed a slightly stronger relationship to specific gravity (Figure 12.16) than that observed for earlywood. Nonetheless, there was considerable scatter. Close examination of Figure 12.16 reveals that some bolts showed no trend between latewood MOE and specific gravity. Figure 12.17 suggests a weak but slightly increasing relationship between specific gravity and G. Both upper and lower bolts seemed to follow the same trend.

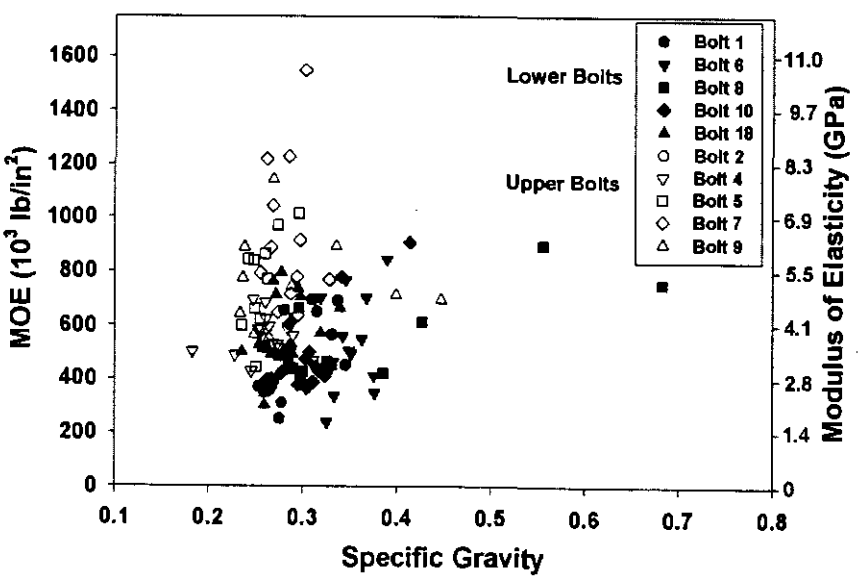


Fig. 12.12 Relationship of earlywood MOE to specific gravity.

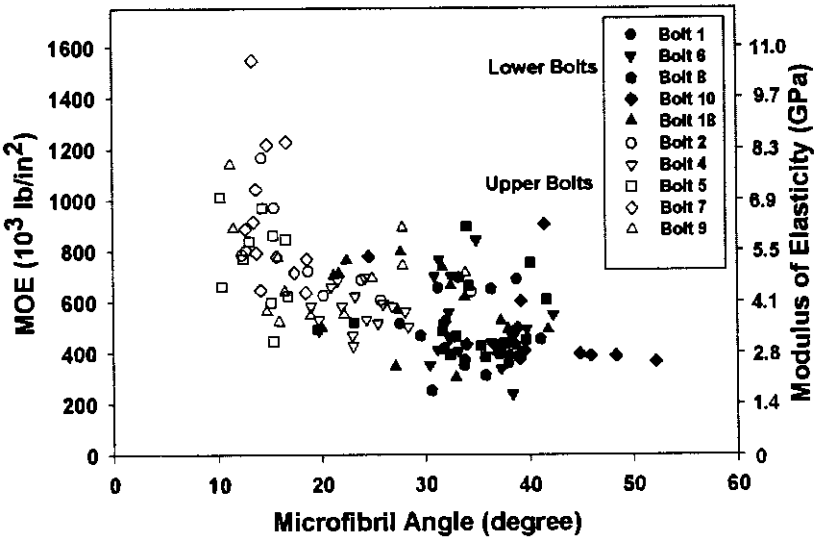


Fig. 12.13 Relationship of earlywood MOE to MFA.

A stronger trend was apparent between latewood MOE and MFA, as shown in Figure 12.18. The plot also shows that as MFA decreased, variability increased. As in earlywood, latewood MFA by itself did not accurately predict MOE, but it is clear that MOE increased with a decrease in MFA. No trend was observed between latewood G and corresponding MFA (Figure 12.19). Lower and upper bolts seem to be segregated by MFA.

Variation of MOE around growth ring

The test results indicated considerable variability in latewood MOE for a given growth ring. We were interested in whether the property variations around the ring were governed by cardinal direction

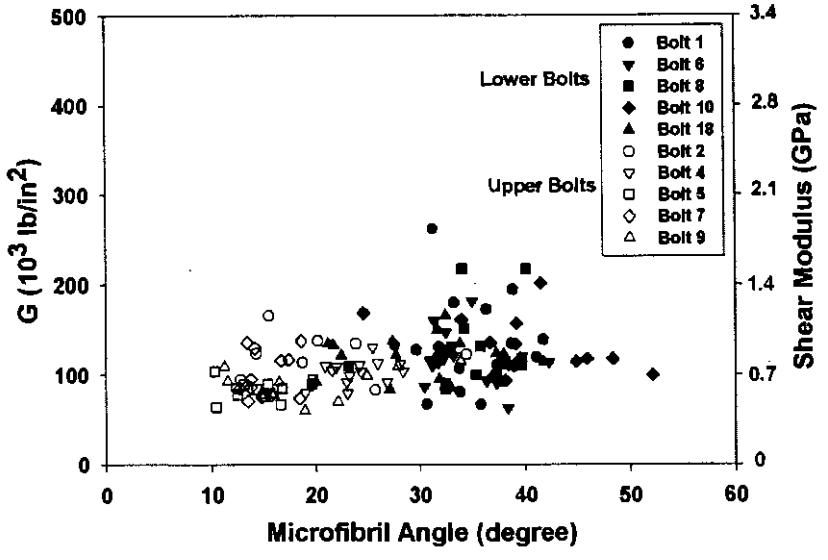


Fig. 12.14 Relationship of earlywood G to MFA.

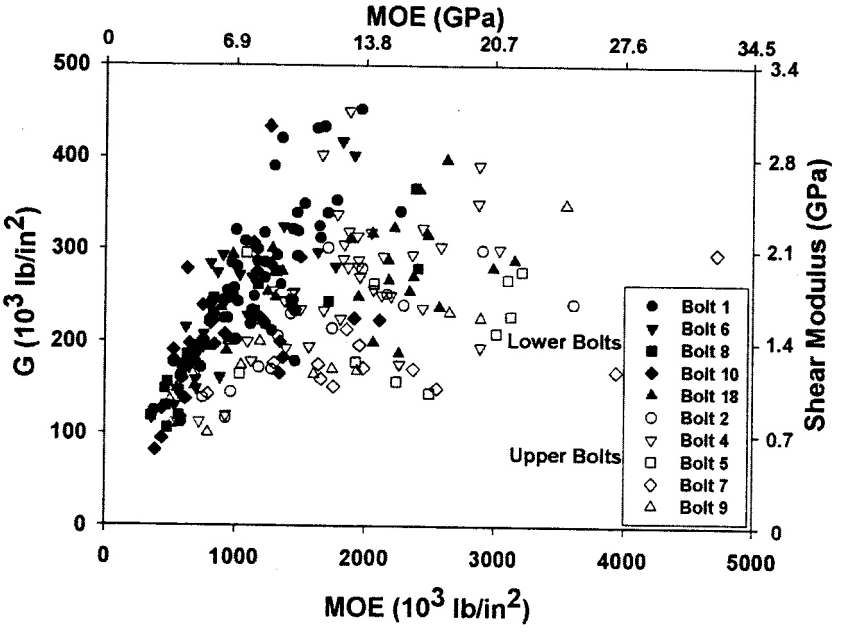


Fig. 12.15 Relationship of latewood G to MOE.

(north, east, south, and west). For the logs harvested, the location of the pith was offset to the west or northwest (Figure 12.20a). The relationship of earlywood and late wood MOE to cardinal direction for bolt 10 is shown in Figure 12.20b. earlywood properties were very consistent around the stem for bolt 10; latewood properties were apparently higher for the south and west compared to the other directions. This pattern, however, was not repeated consistently in the other bolts.

The test results showed no consistent pattern for mechanical properties around the stem based on the distance of the ring from the pith. Three-dimensional plots of MOE and G data for all lower bolts

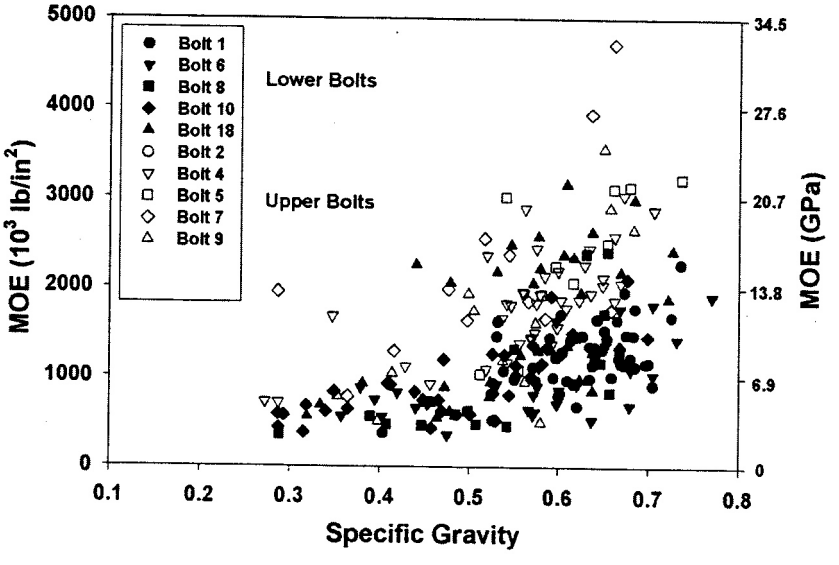


Fig. 12.16 Relationship of latewood MOE to specific gravity.

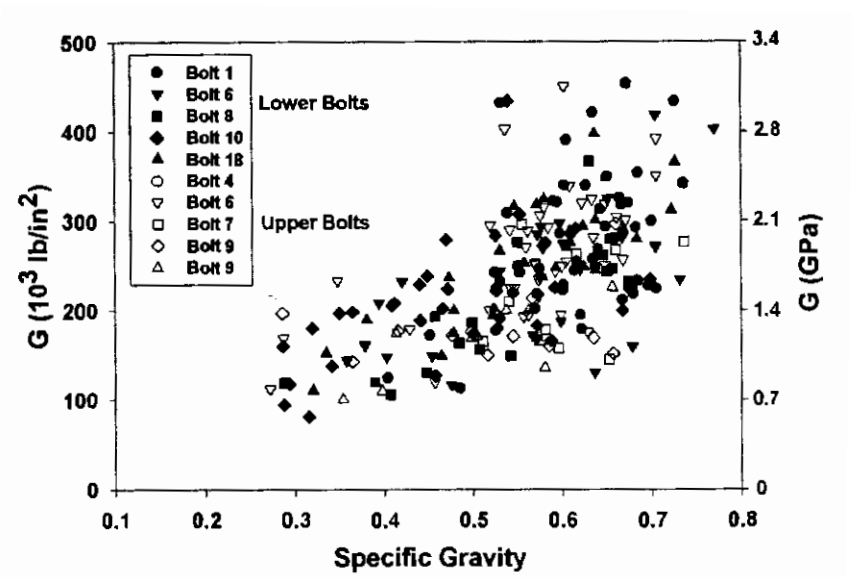


Fig. 12.17 Relationship of latewood G to specific gravity

are shown in Figure 12.21. Close examination of these plots suggests that earlywood properties are remarkably consistent with respect to cardinal direction and distance of growth ring from pith. For latewood, properties were affected by distance from the pith but not by cardinal direction.

Conclusions

The data presented here reveal that earlywood and latewood mechanical properties behave differently, even when the specimens are essentially adjacent to each other in the same growth ring and the same tree. Latewood MOE and shear modulus (G) values are two to three times higher than earlywood

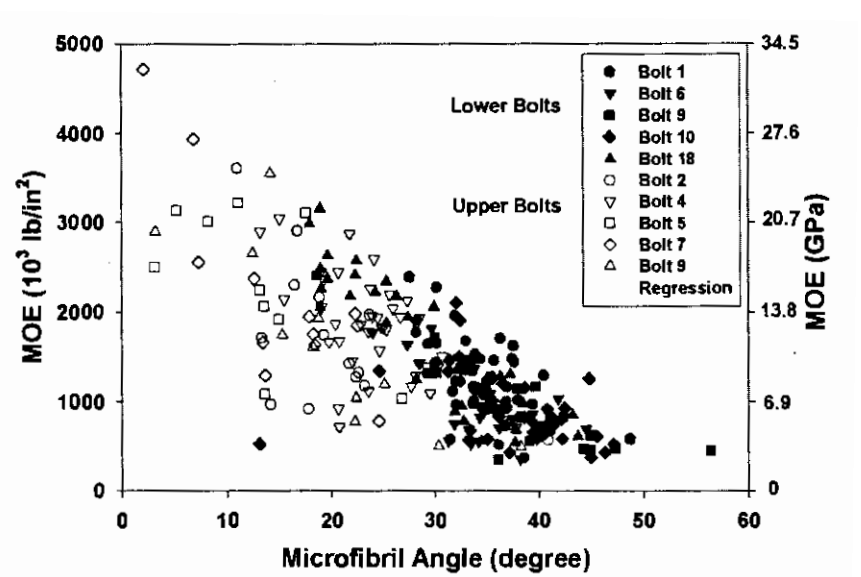


Fig. 12.18 Relationship of latewood MOE to MFA.

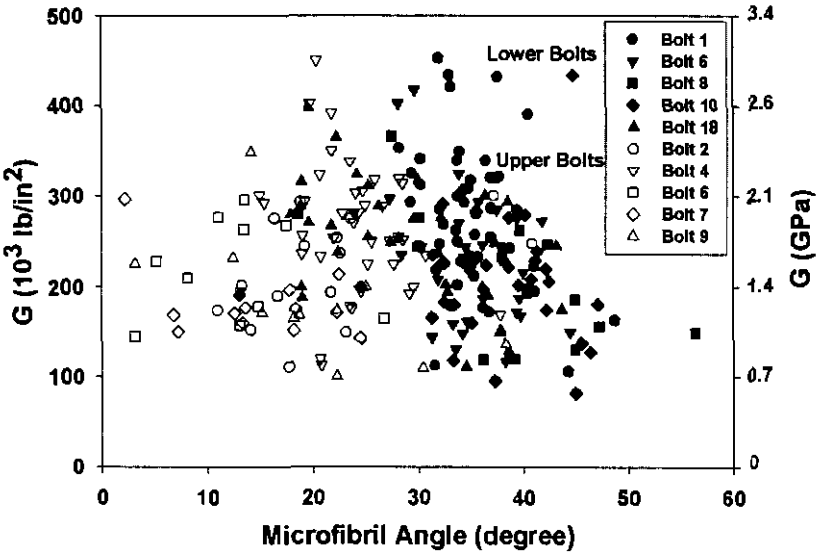


Fig. 12.19 Relationship of latewood G to MFA.

values. In addition, earlywood and latewood properties do not follow similar trends, and they do not show the same relationships with the same parameters. The relationships between mechanical properties and indicator properties differ for earlywood and latewood, MOE and shear modulus, and lower and upper bolts. The relationships that do exist are weak and significant variability persists, especially from tree to tree.

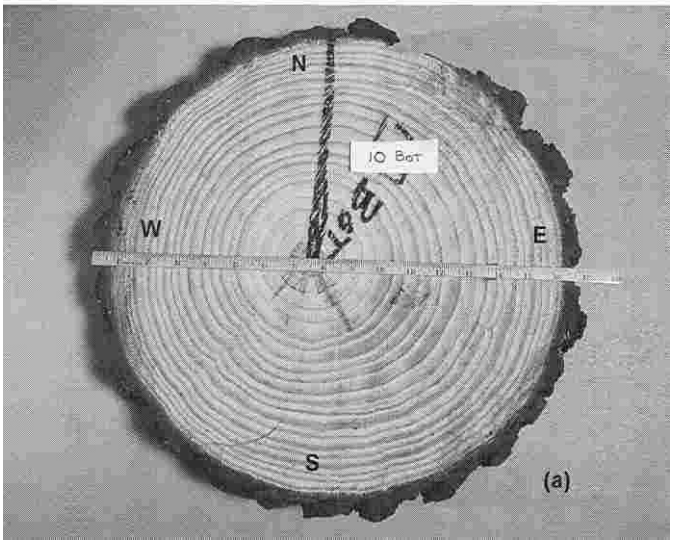
Variability in earlywood properties tends to be low, but the material follows few of the accepted rules in the interrelationship between properties (for example, relationship of MOE to specific gravity). Earlywood properties seem to be constant, regardless of ring position or distance from the pith.

Variability in latewood properties, on the other hand, tends to be high and the relationships between other properties are stronger than those with earlywood. Nevertheless, the relationships are not strong enough to fully account for the variation observed. The relationships between MOE and specific gravity and between MOE and microfibril angle need further analysis. Latewood shear modulus showed no meaningful trend with specific gravity or MFA. Although MOE was a marginal predictor of shear modulus for earlywood, this relationship was strong for latewood only in the lower bolts.

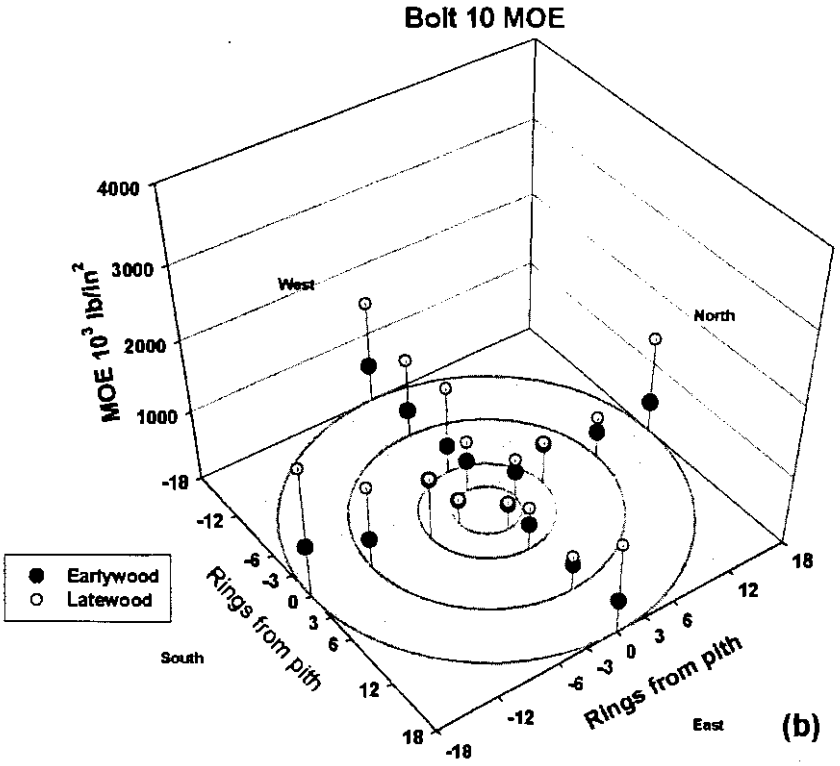
Moving from macrostructure-scale measurements to mesostructure-scale measurements accentuates rather than reduces material variability. The property variation appears to be magnified at the smaller scale. Variation around an individual ring is nearly as large as that from ring to ring and bolt to bolt. This variability is not explained very well by specific gravity, although MFA shows a helpful correlation. One possible explanation is that the variation observed may be a result of biological input and responses that are not reflected in the typical indicator properties. By closely examining biological activity such as branch and crown development, perhaps a linkage to resulting mechanical properties can be established.

Application

It is not just low properties that lead to low wood product quality but also the inconsistency of properties within a line of wood products or within an individual wood product unit. Thus, anticipating,

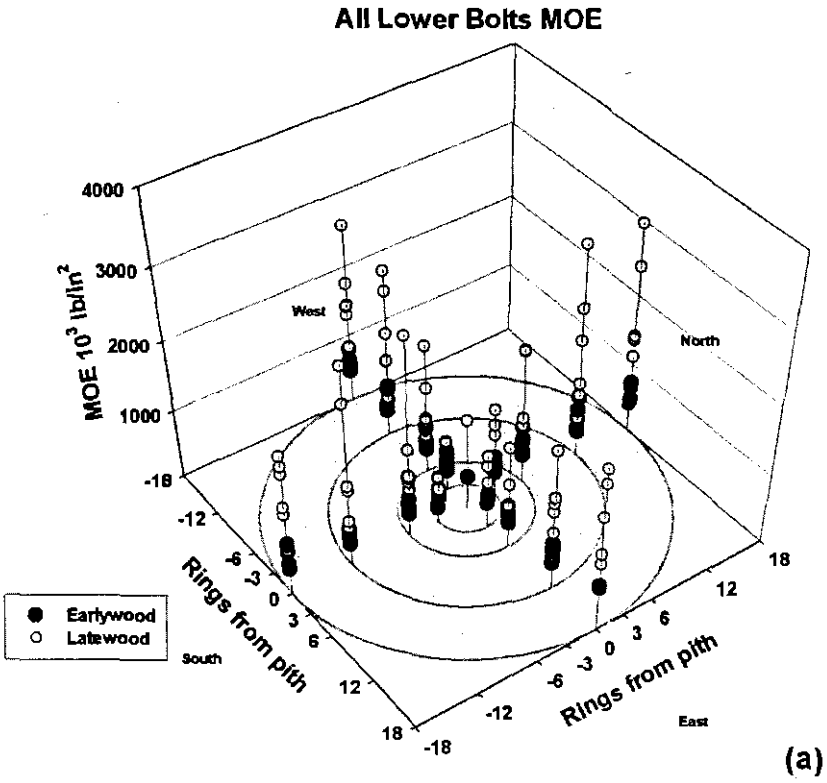


(a)



(b)

Fig. 12.20 (a) The location of the pith in bolt 10. (This figure also is in the color section.) (b) Earlywood and latewood MOE properties for bolt 10.



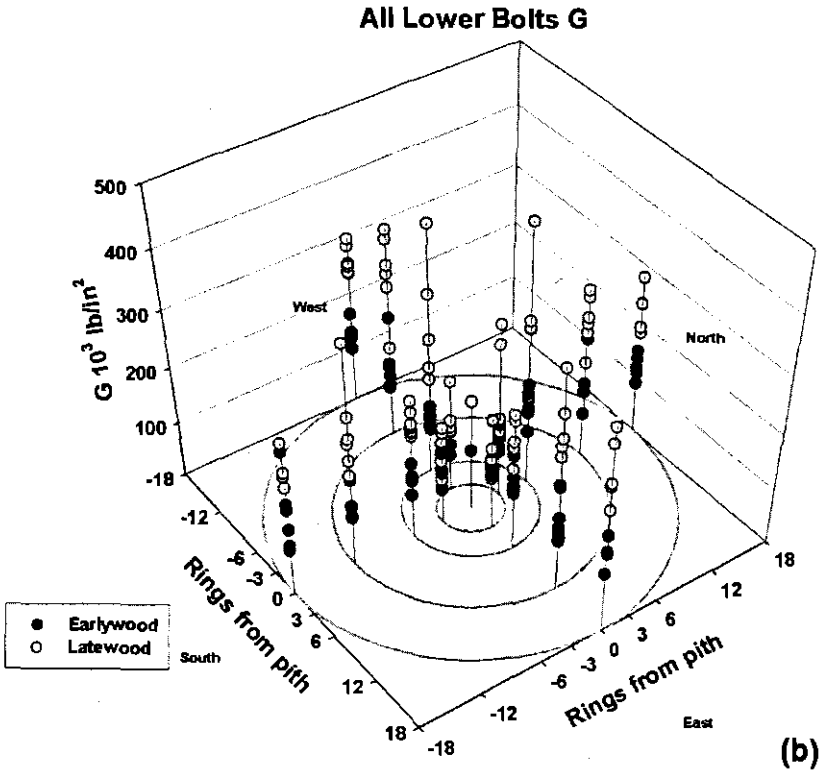


Fig. 12.21 Cardinal direction plots (a) for MOE and (b) for G of all lower bolts

tracking, and controlling property variability is essential to producing the highest quality wood products. The long-range goal of this research is to develop a foundation for property predictions and mechanical modeling. This will allow for a better assessment of resource potential and improved stand management.

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Characterization of the Cellulosic Cell Wall

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