MECHANICAL PROPERTIES OF INDIVIDUAL SOUTHERN PINE FIBERS. PART I. DETERMINATION AND VARIABILITY OF STRESS-STRAIN CURVES WITH RESPECT TO TREE HEIGHT AND JUVENILITY

Les Groom?
Research Forest Products Technologist
USDA Forest Service
Southern Research Station
2500 Shreveport Hwy
Pineville, LA 71360

Laurence Mott†
Research Manager
Perstorp AB
Perstorp, Sweden

and

Stephen Shaler†
Professor
Advanced Composites Engineering Laboratory
University of Maine
Orono, ME 04469

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ABSTRACT

This paper is the first in a three-part series investigating the mechanical properties of loblolly pine fibers. This paper outlines the experimental method and subsequent variation of latewood fiber mechanical properties in relation to tree position. Subsequent papers will deal with differences between earlywood and latewood fibers and effect of juvenility and tree height on global fiber properties. In this paper, the mechanical properties were determined on individual wood fiber with a user-built tensile testing apparatus. Cross-sectional areas of post-tested fibers were determined with a confocal scanning laser microscope and used to convert acquired load-elongation curves into stress-strain curves. The modulus of elasticity and ultimate tensile stress of loblolly pine latewood fibers tested in this study ranged from 6.55 to 27.5 GPa and 410 to 1,422 MPa, respectively. Fibers from the juvenile core of the main stem were on the low end of the mechanical property scale, whereas fibers beyond the twentieth growth ring were near the high end of the scale. Coefficient of variation for fiber stiffness and strength averaged around 20 to 25%. The shape of the fiber stress-strain curves is dependent on their growth ring origins: Mature fibers were linear from initial loading until failure, whereas juvenile fibers demonstrated curvilinearity until about 60% of maximum load followed by linear behavior to failure.

Keywords: Modulus of elasticity, ultimate tensile stress, juvenility, confocal scanning laser microscope, cross-sectional area, microfibril angle.

INTRODUCTION

Individual wood fibers are the primary constituent of fiberboard and paperboard composites. Global pulp requirements for the paper industry alone are expected to almost double from the estimated 1995 level of 191 million metric tons to a 2010 level of 370 million metric tons (Kaldor 1992). The rapid increase in the manufacture of structural fiberboard panels such as medium density fiberboard will compound the situation. Substitution of alternate
fibers and increasing the efficiency of the engineered fiber products are necessary to help meet this demand.

Knowledgeable engineering and subsequent production of fiber-based composite products that meet structural and dimensional stability requirements are difficult at best due to the lack of fundamental information regarding the physical and mechanical properties of the fibers. Researchers have attempted to produce basic engineering data for various virgin fibers (Page et al. 1977; Cave 1969; Jayne 1959; Hartler et al. 1963). However, the reported properties are incomplete due to small sample sizes, inconsistent sampling, and the likely influence of stress concentrations resulting from the gripping assembly (Hartler et al. 1963; Kersavage 1973).

The overall objective of this three-part series will be to determine the mechanical properties of southern pine fibers. The objective of this research is the development of an experimental technique that is rapid, invariable, and accurate. The technique will be used to ascertain the stiffness and strength of latewood (LW) fibers in relation to vertical location within a tree as well as variation from juvenile wood to mature wood. Subsequent papers in this series will evaluate the differences in earlywood (EW) and LW fiber properties and global summation of fiber mechanical properties in relation to position within a tree.

LITERATURE REVIEW

Single fiber testing methodologies

Jayne (1959) was among the first of several researchers to develop methodologies for evaluating the mechanical properties of individual wood fibers. Jayne tested fibers to failure in an Instron tensile tester, with fibers mechanically gripped with small jewelers’ vises. Fiber cross-sectional areas of tensile-failed fibers were determined by visual observation under a compound microscope. A modified version of this system was used subsequently by Kellogg and Wangaard (1964); Tamolang et al. (1967); and by McIntosh and Uhrig (1968). Jayne (1960) reported that a high variation existed in ultimate tensile stress (UTS) and elastic modulus (MOE) both between and within the respective species tested. No values for coefficient of variation or standard deviation were quoted in Jayne’s paper. However, it was speculated that variation may have been attributable not only to fiber geometry and inherent structural differences, but perhaps also to aspects of the testing method employed and specimen selection. Further research by Hartler et al. (1963) concluded that mechanical clamping led to fiber slippage under load. A further major disadvantage of the mechanical restraint method is the unquantified effect of cell-wall compression at the fiber ends, likely contributing to premature fiber failure. Mechanically gripping single fibers often results in more than 50% of the specimens failing at the grips. Hardacker (1963) confirmed this, associating the crushed areas at the fiber tips with low tensile strength values.

The problems cited in previous single fiber axial tensile tests spawned the development of a new fiber gripping method. This involved gluing the fibers to various materials, such as paper tabs, and then clamping these paper tabs within the jaws of a tensile tester. Other adhesive gripping methods involved gluing the single fibers to plastic or metal tabs (Klauditz et al. 1947; Van den Akker et al. 1958; McIntosh 1963; Luner et al. 1967; Duncker and Nordman 1965; Leopold and Thorpe 1968). These authors viewed the adhesive method of gripping as a great improvement over mechanical gripping systems. However, problems have also been encountered using this technique. Manipulating the fibers and gluing them is a tedious and time-consuming operation and it can be difficult to obtain an adhesive with suitable properties. The adhesive should not penetrate or flow along the fiber cell wall, but must develop enough adhesion to prevent fiber slippage or pull-through.

Using the fiber and adhesive system, Hartler et al. (1963) reported fiber misalignment and handling difficulties, both potentially leading to unintentional fiber damage. Testing of a
misaligned glued fiber would lead to deformation at the grips and to stress concentrations. The result leads to 40–50% fiber failure rate at the point of gripping (Leopold and McIntosh 1961; Hartler et al. 1963). Ehrnrooth and Kolseth (1984) analyzed fiber misalignment and concluded it to be a major contributor to both the development of premature failure at the grips and to the development of unrepresentative load-elongation curves.

In a series of experiments conducted at the Pulp and Paper Research Institute of Canada (Page et al. 1972; El-Hosseiny and Page 1975; Kim et al. 1975; Page and El-Hosseiny 1976; Page et al. 1977), fibers were first dried into a flat ribbon shape between a glass slide and a glass micro-cover plate. This prevented the tracheid cell wall from twisting and further facilitated ease of gluing and microanalysis. Improved alignment was noted, and a reduced number of fibers failed prematurely at the end grips. The Fiber Instron developed by Page et al. (1972) also employed a photonic cell to sense fiber elongation, as opposed to gathering data directly from the moving crosshead. This greatly improved the precision in which single fiber strain could be measured. Other unique features of this apparatus included a 16-mm color cine camera attached to a polarizing light microscope. The microscope was mounted in such a fashion as to permit microanalysis of the fiber as the tensile test progressed. This methodology allowed the researchers to establish the relationship between microfibril angle (MFA) and fiber mechanical properties.

Kersavage (1973) modified the adhesive gripping method in an effort to further reduce fiber misalignment stress concentrations and associated premature cell-wall failure. Drops of epoxy resin were applied near the ends of various softwood pulp types. The cured epoxy droplets served as ball joints that were permitted to rotate freely in a ball-and-socket type restraint. Tension was applied to the fiber via the ball-and-socket type assembly. Kersavage (1973) pointed out that fiber tensile strengths obtained by this method were higher than any comparable published values, suggesting the system causes little strength reducing damage to the individual test specimens. This technique was also adapted to investigate load-carrying capacities of fibers from various locations within a tree (Groom et al. 1995, 1996) as well as various agricultural fibers (Groom et al. 1996). Mott et al. (1996) and Shaler et al. (1996) also used the ball-and-socket type assembly for investigations of failure mechanisms of individual fibers monitored in an environmental scanning electron microscope.

Single fiber properties

Jayne (1959) was among the first to develop a database on individual wood fiber mechanical properties. He evaluated the UTS and MOE for 10 softwood species, segregating fiber properties by EW and LW. Results indicated that fibers are generally Hookean in nature, displaying a proportional stress-strain relationship. A limit was found to exist for this region; evidence that wood fibers themselves are viscoelastic in nature. It was conceded that proportional limits varied considerably both between and within each species group. No attempts were made by Jayne (1959) to explain or develop the stress-strain relationships beyond simple speculation, leading Kersavage (1973) to propose that the curvilinear portions of Jayne’s (1959) load elongation curves (LEC) were due to slippage of the grips. Many authors carried out similar tests very often employing only minor changes in testing procedure in efforts to improve accuracy. Understandably this resulted in similar findings to Jayne’s (1959) tests. However, most investigators realized that the shape of the LEC curve was highly dependent upon test conditions and methods.

A more controlled experiment was conducted by Kersavage (1973). The effects of fiber misalignment were considered more carefully, and testing was developed to all but eliminate this problem. Kersavage (1973) was able to report improved accuracy by carefully controlling environmental conditions. Findings suggest that freely dried wood fibers of a
low MFA stressed uniaxially in tension exhibit purely linear LECs, any nonlinearity or curvilinearity (even extreme terminal nonlinearity) being due to the experimental methodology employed.

Kersavage (1973) was also able to confirm the findings of Hardacker (1963), namely that increasing gauge length has a significant negative effect on ultimate fiber stress and that grip failure was more likely to occur in single delignified specimens than individual lignified test specimens. It was also reconfirmed that an increased moisture content reduces fiber strength. The average UTS of fibers taken from a Douglas-fir specimen was reported to be from 853–912 MPa and the MOE in the order of 23.5–24.5 GPa.

Problems associated with small fiber tensile testing have led to the fact that only a handful of species have been tested in the fashion described (Bohalek and Chaturvedi 1988; Jones 1989a; Klungness 1974; McKee 1971; Oye 1985; Pycraft and Howarth 1980; Van Wyk and Gerischer 1982). Difficulty in handling such small samples and the time required to conduct microtensile testing have resulted in a lack of data that reflects only a tiny portion of commercially available species. More impor-
tantly, though, is that these data are most likely skewed, and probably for two reasons. Fiber length limitations precipitated the exclusion of juvenile fibers, thus skewing the data towards cell maturity. In addition, previous researchers were unaware or ignored the aspect of juvenility that affects physical and mechanical properties of wood and wood fiber.

EXPERIMENTAL PROCEDURES

Material selection

A loblolly pine (Pinus taeda L.) tree was selected and felled from a conventional plantation stand located at the Crossett Experimental Forest, Crossett, Arkansas. The selected tree, which was 48 years old was straight in form to minimize the presence of reaction wood. The diameter at breast height was 42.2 cm, overall height equaled 30.3 m, and height to live crown was 21.2 m. Immediately upon felling, a disk approximately 2.5 cm thick was removed every 3.05 m in height, starting from the stump and proceeding to a 10-cm top.

Several LW slivers were removed from each disk at growth rings 5, 10, 20, 30, 40, and 48. The slivers measured approximately 2 by 2 by 25 mm. The number of growth rings analyzed for each disk varied as a function of tree height, with the uppermost disk consisting of only growth rings 5 and 10. In an attempt to minimize variability, all slivers were removed from the north compass heading. Approximately three slivers per growth ring and tree height were macerated in a solution comprised of 1 part 30% hydrogen peroxide, 4 parts distilled water, and 5 parts glacial acetic acid. A typical maceration time for LW slivers was approximately 24 h. Macerated fibers were washed several times with distilled water. Dilute fiber slurries were placed between glass slides, thus allowing the fibers to dry without twisting.

Dried fibers were placed over a 2.5-mm channel; fiber ends were attached to the channel plate via double-sticky tape, with the center portion of the fibers suspended over the channel. Two epoxy droplets were placed in the center portion of each fiber via forceps with an approximate spacing of 1 mm. An expanded description of the epoxy placement technique can be found in Mott (1995). The epoxy used in this study was Devcon 2-ton, a slow-cure, high-strength, 2-part adhesive. An epoxy:hardener ratio of 56:44 was found to release the most energy during cure as determined with differential scanning calorimetry tests (Mott 1995), and thus that ratio was used throughout this study. 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 (Fig. 1) attached to a miniature materials tester. The gripping assembly allows for free fiber rotation during tensile testing and allows for rapid replacement of replicates during testing. A dissecting microscope was used to place specimens in the gripping assembly as well as subsequent removal upon fiber failure. Load was acquired with a 5-N capacity load cell. Elongation was taken as crosshead movement, set at a constant strain rate of 80 microns per minute. 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). Thirty fibers were tested for each growth ring and tree height.

Confocal scanning laser microscopy

Failed fibers were stained with a dilute concentration of acridine orange (10 mg/300 ml of distilled water), thus enabling the macerated fibers to fluoresce when subjected to an excitation laser source. The failed fiber segments were attached to a glass slide with the aid of tissue tack. Epoxy droplets were then removed under a dissecting microscope with Vannas-type 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, with techniques and settings similar to Jang et al. (1991). Fibers were imaged with a 100X oil-immersion objective. The XS images were constructed from a series of vertical line scans adjacent to the failure location. Although exact settings varied due to a great degree of variability between fiber types, generally the step size was 0.1 micron, and approximately 40 images were averaged per scan to increase the signal-to-noise ratio. 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 MFA on 10 of the failed fiber specimens per growth ring and tree height. The MFA was determined on the failed fiber ends (Fig. 2) with the aid of an angular micrometer on the margins of a rotating stage. No attempt was made to differentiate between radial and tangential walls.

RESULTS AND DISCUSSION

Mechanical properties

Load-elongation curves for LW fibers tested at heights 0, 12.2, and 24.4 m are shown in Fig. 3. The average tensile span length for the LW fibers was 1.3 mm. Failure occurred pri-
FIG. 3.  Load-elongation traces for various growth rings of individual wood fibers located at: (a) stump height, (b) 12.2 m from stump height, and (c) 24.4 m from stump height. Every tensile test (n = 30) for each growth ring and vertical location is shown.

marily in the center span, with failures occurring at the grips in 18% of the tests. Thus, the ball-and-socket type grip assembly used in this study reduced the rate of grip failures by one-third as compared to previous mechanical clamping studies (Hardacker 1963).

It is evident that there exists a large difference in load-elongation response in relation to distance from the pith. This is especially evident at stump height: load-carrying capacity and slope of rings 5 and 10 displayed compliant behavior with a slight increase in load-carrying capacity and slope with an increase in age. This behavior is present regardless of tree height, although this effect decreases with tree height. However, the LECs are of limited use due to varying cross-sectional areas. It is for that reason that stress-strain curves are more representative of engineering properties.

Cross-sectional areas in conjunction with tensile span length values were used to convert LECs to stress-strain curves. Cross-sectional area and tensile span length are summarized in Tables 1 and 2. The stress-strain curves of all LW fibers tested at heights 0, 12.2, and 24.4 m are shown in Fig. 4. Modulus of elasticity and UTS values for all fiber groups tested were taken from the corresponding stress-strain curves and are summarized in Tables 3 and 4, respectively. The average MOE and
UTS values for all LW fibers tested were 19.7 GPa and 1,040 MPa, respectively. It should be noted that these averages are not representative of the entire tree. The whole tree average of MOE and UTS would actually be higher due to the large volume of mature fibers as compared to juvenile fibers. The coefficient of variation (COV) for the MOE and UTS values were 20.7 and 23.5%, respectively.

The stress-strain curves of LW fiber show a similar trend as was observed in the LECs; the stiffness and strength of LW fibers are directly related to the degree of juvenility. This effect is not as pronounced for the stress-strain curves as it was for the LECs. This is due primarily to the change in cross-sectional areas with cell-wall maturation. Juvenile fibers have thinner cell walls than the corresponding mature counterparts (McMillan 1968) and as such converted stress-strain curves near the pith have slopes more comparable with those beyond growth ring 20 when compared to the LECs.

The effect of juvenility with regard to fiber mechanical properties diminishes with vertical location within the tree. At stump height, the fiber stiffness and strength increase by a factor of approximately three from the fifth growth ring when compared to rings 20 and beyond. The increase in mechanical properties is only about 35% for ring 5 versus rings 20 and beyond at a tree height of 12.2 m. This difference is no doubt due to the variability of MFA in an individual tree and the relationship be-

Table 1. Average cross-sectional area and coefficient of variation of fibers as a function of tree height and growth ring.

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<th>Disk height (m)</th>
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<th>Coefficient of variation (percent)</th>
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Table 2. Average tensile span and coefficient of variation of fibers tested in tension to failure.

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Fig. 4. Twenty-five confocal scanning laser microscope images of latewood fiber cross sections from the 20th growth ring at a tree height of 9.1 m.

tween MFA and fiber mechanical properties. Megraw et al. (1999) found that the greatest values and variability of MFA for N-year-old loblolly pine occur at the tree base and decrease with tree height. Van Den Akker (1970) and Page and El-Hosseiny (1976) also found that fiber stiffness is correlated strongly with MFA. Thus, it is justifiable that the effect of

Table 3. Average modulus of elasticity and coefficient of variation of fibers as a function of tree height and growth ring.

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juvenility on the stiffness and strength of loblolly pine LW fibers is greatest near the pith at the base of loblolly pine. This effect is minimized with increased vertical location within the tree but still exists.

Figure 5 shows that the shapes of the stress-strain curves are also a function of juvenility. Juvenile fibers demonstrated an initial curvilinear region followed by linearity to failure. The curvilinear region became linear at approx-
proximately 60% of the UTS. The degree of curvilinearity was proportional to the MFA and appeared to be nonexistent for fibers with an MFA less than 20 degrees. This is most likely related to the sliding of microfibrils in the secondary cell walls during tensile loading. The shift from curvilinearity to linearity coincided with the occurrence of tension buckling. This phenomenon, first described in wood fibers by Page et al. (1971), OCCURS when the tensile force exceeds the lateral resistance of the cell wall.

Physical properties

Measured cross-sectional areas of loblolly pine LW fibers averaged 350 μm², with an overall COV of 19.2%. Cross-sectional images obtained with the CSLM are shown in Fig. 6. Figure 6 demonstrates the wide variety of fiber shapes encountered within each data set. Fiber collapse occurred in 27% of all fibers tested. The frequency of fiber collapse occurred consistently in all data sets, regardless of vertical or horizontal position within the tree. The mechanical properties of the fibers were unaffected by collapse.

The need for physical measurement of cross-sectional area is evident for fibers tested and measured in the manner outlined in this study. Approximately ten percent of fibers exhibited fuzzy lumen margins. The actual lumen could be seen and measured manually, but were not easily measured precisely with thresholding. A second problem arose with missing cell-wall material, also present in about ten percent of measured fibers. Cross-sectional areas are most commonly measured immediately adjacent to the failure site and thus may have portions of the cell wall missing as a result of tensile failure. These missing cell-wall areas are ignored during manual measurement of cross-sectional area but would...
be measured as lumens with thresholding techniques.

The cross-sectional areas follow trends that are consistent with published literature on juvenility, specifically that cross-sectional area is at a minimum near the pith. The cross-sectional area increases steadily until rings 20-30 and then levels off (Megraw 1985). However, the maximal cross-sectional area in this study was located approximately 10 to 15 rings inside the cambium and below the live crown (Table 1).

Microfibril angle

The average MFA and corresponding COVs for all loblolly pine LW fibers examined in this study (Table 5) were 14.3 degrees and 34.6%, respectively. There existed a great disparity between juvenile and mature fibers. Growth ring 5 had an average MFA of 25.7 degrees as compared to an average MFA of 8.2 degrees for growth rings 20 and beyond. The trends of MFA are inversely related to fiber MOE and UTS. This relationship between MFA and fiber mechanical properties has been previously reported by Van Den Akker (1970) and Page and El-Hosseiny (1976) and will be explored in detail in an upcoming publication.

SUMMARY AND CONCLUSIONS

The mechanical properties of individual LW loblolly pine fibers were ascertained with a material tensile testing apparatus outfitted with a ball-and-socket type grip assembly. Cross-sectional areas were determined with a CSLM. The mechanical properties of LW loblolly pine fibers are a function of location within a tree. Latewood fiber stiffness and strength are relatively constant with respect to vertical location within an individual tree but increase with increased distance from the pith. Fiber MOE and UTS increased by a factor of approximately 3 from the juvenile to the mature portion of the tree at stump level and markedly less for distances further up the tree. Average LW fiber MOE values ranged from 6.6 GPa at the pith to as high as 27.5 GPa in the mature portion of the tree. Average fiber UTS values ranged from 410 MPa at the pith to 1,420 MPa in the mature portion of the tree. The effects of juvenility on the stiffness and strength of LW fibers are consistent with changes in the MFA.

Stress-strain curves of the individual fibers are of two distinct types. Fibers with MFAs greater than 20 degrees exhibited curvilinear behavior up to 60% of the load-carrying capacity of the fibers at which the stress-strain behavior becomes linear to failure. Fibers with narrow MFAs exhibited linear stress-strain curves throughout the duration of the test.

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