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The Longitudinal Young's Modulus of *Pinus Radiata*

By I. D. CAVE, Lower Hutt

Summary

The variation of the longitudinal YOUNG's modulus with mean cellulose microfibril angle of the wood substance of the earlywood of a softwood has been determined from small clear samples.

The longitudinal YOUNG's modulus falls steeply as the angle between the longitudinal axis and the mean microfibril direction in the cell walls increases. The variation has been explained in both form and magnitude by applying the elastic theory of a fibre composite material with distributed fibre directions to a model of the experimental material. It confirms the two phase concepts of the plant cell wall, as far as the elastic properties are concerned, of rigid crystalline microfibrils embedded in an isotropic matrix of amorphous and paracrystalline materials.

Zusammenfassung

Die Änderung des YOUNG-Moduls in longitudinaler Richtung mit dem mittleren Winkel der Cellulose-Mikrofibrillen in der Frühholzsubstanz von Nadelholz wurde an kleinen, fehlerfreien Proben bestimmt.

Der longitudinale YOUNG-Modul fällt mit zunehmendem Winkel zwischen Längsachse und mittlerer Mikrofibrillen-Richtung in der Zellwand steil ab.

Die Änderung wurde nach Form und Größe durch Anwendung der Elastizitätstheorie für ein faseriges Material mit wechselnden Faserrichtungen auf ein Modell aus dem Versuchsmaterial erklärt. Das Zwei-Phasen-Modell der Pflanzenzellwand wird damit bestätigt, soweit es sich um die elastischen Eigenschaften von starren, kristallinen Cellulose-Mikrofibrillen handelt, die in einer isotropen Matrix aus amorphem, parakristallinen Material eingebettet sind.

Introduction

In recent years several accounts of work relating the mechanical properties of plant cell walls to their structure have been published. [MEREDITH 1946; HEARLE 1963; PROBINE 1963; BARBER and MEYLAN 1964; COWDREY and PRESTON 1966; MARK 1967]. CAVE [1968] has published a theory of the elasticity of plant cell walls which includes consideration of the dispersion of the cellulose microfibril directions about the mean direction. The present paper extends this theory by consideration of not only the wall structure but also the cell configuration of the earlywood of softwood, from which the longitudinal YOUNG's modulus is computed. The results are compared with those found experimentally in a set of *Pinus radiata* earlywood specimens.

The essential structure of softwood as far as the present study is concerned, is the same as that given by COWDREY and PRESTON [1966] in their study of the elasticity of Sitka spruce; they state, "Conifer wood consists in the main of a series of elongated cells—the tracheids—long hollow thick wall tubes with ends tapering to a blunt chisel shape lying parallel to each other and to the length of the trunk cemented together mainly by lignin into a tissue. The tissue is traversed horizontally from pith to bark by a number of 'ribbons'—rays—consisting of shorter cells whose lengths are oriented radially along the ribbons. The walls of

the tracheids, like the walls of most plant cells, consist physically of a two phase system where the phases interpenetrate to some extent. One phase consists of cellulose in the form of long thin rods, the microfibrils, some 100 Å wide and perhaps half as thick and apparently endless, of which the central core is crystalline. These lie embedded in the other phase, an amorphous or paracrystalline complex of polysaccharides, polysaccharide derivatives and lignin. The microfibrils are densely, though not closely packed, fairly parallel to each other in a direction inclined to cell length and therefore winding helically round the cell. In terms of this helical winding three lamellae can be distinguished in the wall. In a thin outermost lamella (S1) the microfibrils lie in a slow helix. In a thicker central lamella (S2) in a steep helix and in a third innermost lamella, again in a slow helix (S3)."

The model used by the author for the cell wall itself differs from that of COWDREY and PRESTON in that a balanced crossed-structure of the microfibrils about the axial direction has been postulated in order to account for the lack of torsion in the transverse plane associated with an axial tension acting on whole wood specimens, and which would ordinarily take place in a free single tracheid under axial tension. If the cell walls are thin this crossed-structure represents the balanced laminate of the bonded wall pairs of adjacent cells.

The Theoretical Model

The theoretically expected relation between microfibril angle and longitudinal YOUNG's modulus is obtained by applying the results of the elastic theory of the plant cell wall [CAVE 1968] to a model of the experimental material.

The model assumes that the tracheids comprise 95% of the earlywood in *Pinus radiata* (2 ... 7% of coniferous wood is ray tissue, KELSEY [1963]) and that it is these fibres which provide the longitudinal rigidity of the wood. All tracheids are assumed to have the same microfibril angle distribution, and features such as the pits and tapered ends of the tracheids are ignored so that the tracheids can be considered to be long smooth tubes. The tracheid walls of the earlywood are thin and justify the use of the crossed laminate concept in the structure of the main load carrying layer (S2) of the tracheid. MARK [1967, p. 111] gives estimates of the area proportions of the various regions of the earlywood cell wall of softwoods derived from information from various sources and species. He found that the area external to the secondary wall (corrected for microvoids), i.e. the middle lamella or intercellular bond region plus the primary cell wall (M + P), amounts to 11.2% of the whole and consists of 10.1% cellulose, while the area proportion of the S1, S2, S3 secondary wall layers are 17.5%, 61.1%, 19.2% where the volume ratio of fibre to matrix is the same in each layer. These figures can be regarded only as estimates for softwoods in general so that it is proposed to take *Pinus radiata* earlywood tracheid area ratios M + P: S1 + S3 : S2 to be 10 : 30 : 60 and assume all the cellulose is confined to the secondary wall. The cellulose in the S1 and S3 layers lies at large angles to the axis and so can contribute little to the stiffness in the longitudinal direction. Accordingly, it is assumed that the S2 layer alone bears the load in axial tension. Thus two-thirds of the cellulose of the tracheid is effective in axial tension. Overall, then, the longitudinal YOUNG's modulus of the earlywood model is $\frac{2}{3} \times .9 \times .95$ that, (E_L say), of a piece of the S2

layer. For the purposes of comparison with experiment this value would provide a lower limit.

The value of E_L has been determined from the theory discussed in CAVE [1968]. A gaussian with standard deviation of 10° about the mean angle has been assumed for the fibre distribution of the S2 layer, and the elastic constants of the composite have been obtained by inserting the values for the elastic constants of cellulose and matrix and the volume ratio of the two phases given in Table 1 into the formulae provided by HILL [1965] in the self-consistent model of the fibre composite.

Table 1. *Data for the Computation of the Elastic Constants of the Composite*

	Elastic constant stated by MARK (1965)		Derived stiffnesses	Source
Cellulose	longitudinal YOUNG's modulus	$E_L = 1.37 \times 10^4$ kp/mm²	$c_{33} = 13{,}730$ kp/mm²	SAKURADA et al. [1962]. Experimentally determined by X-ray technique
	transverse YOUNG's modulus	$E_T = 0.157 \times 10^4$ kp/mm²	$c_{11} = 1{,}572$ kp/mm²	MARK [1965]. Theoretical computation
			$c_{12} = 12$ kp/mm²	Obtained by setting $s_{12} = 0$
	POISSON's ratio of contraction transverse to longitudinal extension under longitudinal stress	$\nu_{LT} = 0.1$	$c_{13} = 157$ kp/mm²	MARK [1965]. Theoretical computation
	Shear modulus of rigidity average for 101 and $10\bar{1}$ planes	$G_{LT} = 380$ kp/mm²	$c_{44} = 380$ kp/mm²	MARK [1965]. Theoretical computation
Matrix	YOUNG's modulus	$E = 204$ kp/mm²	$c_{11} = 275$ kp/mm²	Estimate based on SRINIVASAN's [1941] value for lignin
	POISSON's ratio	$\nu = 0.3$	$c_{12} = 118$ kp/mm²	Estimate based on POISSON's ratio for a rigid laminating resin

Variation of cellulose volume ratio with mean microfibril angle M

M	0	10	20	30	40
Cellulose volume ratio	.58	.58	.57	.51	.46

HILL's formulae for the elastic constants of a composite are the following:

c_1, c_2 are the volume fractions of the two phases, 1 and 2 and m, k, l, n, μ are elastic constants of the composite.

$$\frac{c_1 k_1}{k_1 + m} + \frac{c_2 k_2}{k_2 + m} = 2\left(\frac{c_1 m_2}{m_2 - m} + \frac{c_2 m_1}{m_1 - m}\right)$$

$$k = \left(\frac{c_1}{k_1 + m} + \frac{c_2}{k_2 + m}\right)^{-1} - m$$

$$l = \frac{l_1 - l_2}{k_1 - k_2}(k - c_1 k_1 - c_2 k_2) + c_1 l_1 + c_2 l_2$$

$$n = \left(\frac{l_1 - l_2}{k_1 - k_2}\right)^2 (k - c_1 k_1 - c_2 k_2) + c_1 n_1 + c_2 n_2$$

$$\frac{c_1}{\mu - \mu_2} + \frac{c_2}{\mu - \mu_1} = \frac{1}{2\mu}$$

where $m = \frac{1}{2}(c_{11} - c_{12})$, $k = \frac{1}{2}(c_{11} + c_{12})$, $l = c_{13}$, $n = c_{33}$, $\mu = c_{44}$ in each phase and in the composite.

The constants of cellulose and matrix quoted are the estimates provided by MARK [1965] while the volume ratios are derived from the determination of the cellulose weight percentage of the whole wood across the fifth internode disc of the tree under study. The cellulose determination shows that the cellulose-matrix volume ratio varies with the mean microfibril angle in the S2 layer, as illustrated by the dashed lines in Fig. 5.

The variation of the longitudinal YOUNG's modulus with mean microfibril angle of this model of *Pinus radiata* is shown by the solid line in Fig. 4.

Experimental Materials and Methods

Sixty specimens were taken from the earlywood of two discs, one at breast height and the other from the fifth internode of a tree of *Pinus radiata*. The material was regarded purely as a tissue which could be used to test the hypothesis that the microstructure of the cell wall plays a predominant role in the elasticity of plant tissues. As such species and sample size are immaterial to the argument. Sampling as indicated by a survey of mean microfibril angle across the discs was made to ensure an even distribution of samples over the range of microfibril angle available (approximately $10° \ldots 40°$).

The samples were first roughed out from the earlywood by splitting down the grain of the discs, which were 70 ... 80 mm thick, and then machined to final size in three stages. A reference surface was prepared by hand from a carefully split surface by rubbing the specimen on fine glass paper. In the first machine stage the sample was cut down to about double final size on a band saw. In the next two stages the specimen was held in a specially constructed high flow vacuum chuck on a milling machine. On the mill the specimens were first machined to approximate size and then trimmed in one thousandths of an inch cuts by an extra sharp $\frac{1}{8}''$ spiral end mill to a final size of 2 mm × 2 mm × 60 mm. It is estimated that the cross-section over the centre portion between the gauge marks in each specimen was uniform to within 2% at the time of preparation. The ends of each specimen were potted in epoxy resin using 5 mm diameter gelatin capsules as moulds. The specimens were mounted in a brass and perspex jig before the filling of the capsules to provide accurate centring of the specimen in the mould. The resin was cured at room temperature to partly hard consistency and the capsules

removed. The specimens were gripped in self-aligning grips by the plastic coated ends and were strained at constant cross head speed in a standard Instron testing machine.

Measurement of Strain

The use of the relative crosshead motion as a measure of specimen extension was avoided because of the possibility of slip of the specimen in the grips and instead the extension was measured by photographing the specimen at approximately 10 second intervals throughout the period of strain. The crosshead speed was 0.5 mm per minute so that the period of extension usually lasted 2 minutes or about 12 photographic cycles. The camera was fired by an electronic timing device which simultaneously set off an electronic flash illuminator and put a marker on the time-load plotter of the Instron control console. The camera system consisted of a 35 mm M2 Leica body with mirror reflex attachment. The lens, a high quality process lens, was mounted to give a magnification at the focal plane of approximately 1.7. A narrow-band green filter to optimise performance of the lens and a fine grain 35 mm document copying film (Duplopan) were used. The aerial image of the specimen was focused on the lower surface of the clear glass screen of the mirror reflex attachment by means of a 50× microscope.

When viewed in a 50× microscope the negative image on the film showed a large number of small well-defined points of specular reflection, especially where soft pencil lead had been applied to the specimen surface and a pair of these at suitable separation was selected as reference marks for the extension process. The relative positions of each pair of spots was recorded with respect to the specimen axis to the nearest 0.00002 in. on a GSIP Trioptic measuring machine. The gauge length on the film was selected to be about 1 in. and typically the strain to break was about 1% so that overall extensions were measured to better than 1% accuracy. Film stability is entirely adequate in this application.

Measurement of Load

The load corresponding to the extension at the instants recorded on the film was measured at the time marker pips on the time-load record of the Instron machine. The chart speed was 10 cm per minute. A load cell of 500 kg was used with the load selector either on the 50 or 100 kg full scale settings. Load to break ranged from 20 ... 80 kg.

Measurement of Cell Wall Area

YOUNG's modulus has been expressed in terms of the cell wall area rather than in terms of whole wood which includes lumen spaces, to give the YOUNG's modulus of the wood material. The cell wall area was obtained from the vacuum dry weight per unit length of the central 20 ... 30 mm portion of each specimen after the tensile test was concluded, assuming the density of wood substance to be 1.46 g/cm³ [STAMM 1964].

The YOUNG's modulus test was carried out after conditioning overnight at 50% r.h. so that the YOUNG's moduli in Figs. 2 and 3 refer to a 10% moisture content corresponding to equilibrium at 50% r.h. and based on wall area determined in the vacuum dry condition. The reasons for this procedure are that the computations based on theory have used the dry weight basis for the cellulose-matrix composition ratio of the cell wall and the elastic constants of the matrix at normal

equilibrium moisture contents. It is implicit in the theory that the properties of cellulose are unaffected by moisture content changes in the cell wall [BARBER and MEYLAN 1964]. Thus the YOUNG's modulus at 10% moisture content is

$$E = \frac{0.0146}{m} \times \frac{P}{\varepsilon} \text{ kp/mm}^2$$

where m is the vacuum dried mass per unit length (g/cm), P is the load in kg, ε the strain at 10% moisture content.

The largest source of error in the determination of the YOUNG's modulus was in the strain. The elastic load/strain ratio of each sample was determined by plotting corresponding strain and load values from the photographic and Instron chart records and drawing the best fit line through the points. Usually when well-defined gauge markers were available on the images the maximum error of measurement of the load/strain ratio was of the order of 1%, but in less favourable cases when the specimen moved after the focussing procedures were completed and the image became slightly out of focus as the load was applied, the maximum likely error could have been 5%. This trouble was later eliminated by loading the specimen to about 5 kg before focussing the camera on the specimen. The errors in the other parameters involved in the determination of the YOUNG's modulus, the mass per unit length of specimen, and the load are substantially less than 1%. In the case of the mean microfibril angle, for specimens of the size involved in this study, it is believed that the method devised by MEYLAN [1967] has an accuracy of about $1\frac{1}{2}°$ over the range of angles available (10° ... 40°).

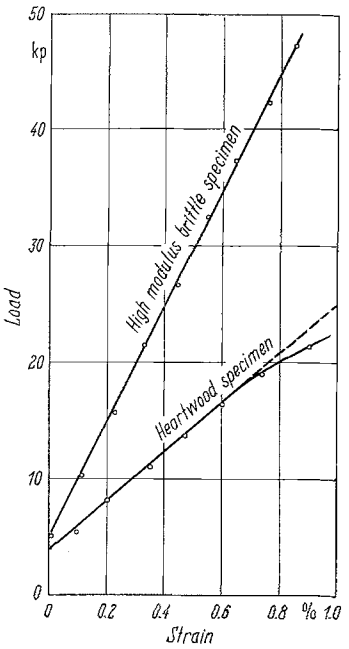


Fig. 1. Typical stress-strain diagram of high modulus brittle specimen and of heartwood specimen.

Experimental Results

The majority of the wood samples showed typical brittle behaviour, the load-strain curves following a straight line to breaking point (Fig. 1). Those specimens taken from within the heartwood region had two straight sections in the load-strain curve with a knee occurring at strains of 0.4 ... 0.8%. The initial gradient was used for the YOUNG's modulus in these cases. Failure occurred usually at either of the inner epoxy margins with strains ranging from 0.6% ... 2.0% with most in the 0.8% ... 1.1% range. The breaking strengths are plotted against mean microfibril angle in Fig. 2 and longitudinal YOUNG's modulus in Fig. 3.

Longitudinal YOUNG's modulus is plotted against mean microfibril angle in Fig. 4. The strengths and longitudinal YOUNG's modulus are stated for equilibrium at 50% r.h. and room temperature based on the wall area determined in the vacuum dry condition. The variation of the mean microfibril angle for both discs with annual ring number and the percentage cellulose on the dry weight

basis determined for five annual rings steps of the fifth internode disc with annual ring number is shown in Fig. 5. For the purposes of computation in the model the scatter between the pairs of variables has been smoothed as shown by the dashed lines, and a one to one relationship between the three variables assumed, to derive the variation between percentage cellulose and mean microfibril angle given at the bottom of Table 1.

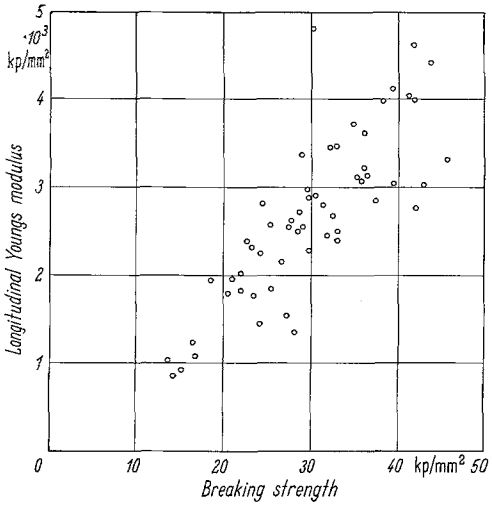


Fig. 2. Relation between mean microfibril angle and breaking strength.

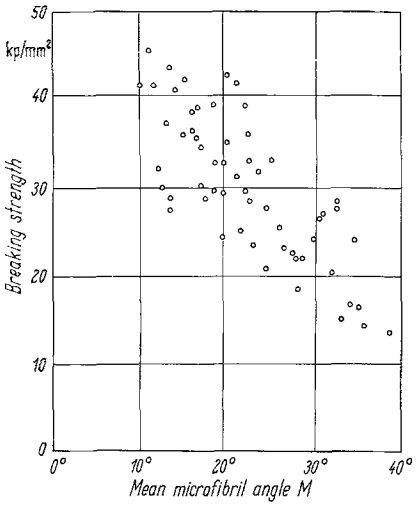
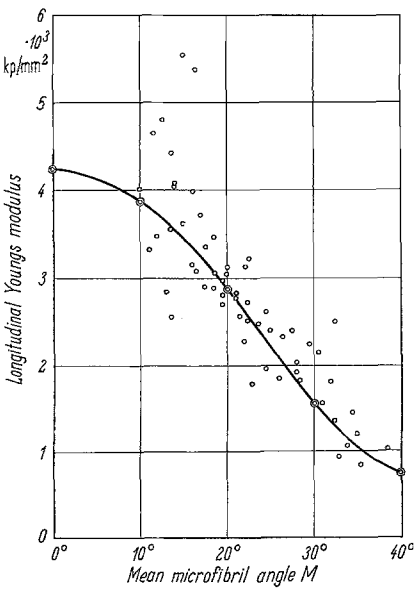


Fig. 3. Relation between breaking strength and longitudinal YOUNG's modulus.



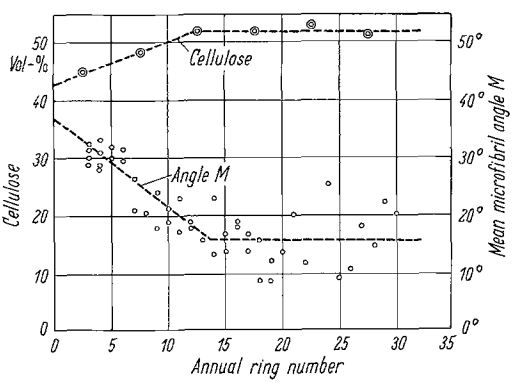


Fig. 5. Relation between annual ring number and volume percentage of cellulose ⊙; annual ring number and mean microfibril angle O; postulated one to one correspondence between the variables annual ring number, volume percentage of cellulose and mean microfibril angle, dashed lines.

Fig. 4. Relation between mean microfibril angle and longitudinal YOUNG's modulus. Experimental points O, theoretical relation = full line.

Discussion

In most previous work relating mechanical properties of wood to its microstructure attention has been focused directly on the properties of the tracheid and the problem of measuring the cross-sectional area of the cell walls [e.g. MARK 1967]. To reduce the labour involved in measuring cross-sectional area by conventional microscopic techniques and at the same time to eliminate all tissue other than tracheids from the sample very small microtome samples have been used. In MARK's work the extremum is reached in that samples containing but a single layered ribbon of 10 or fewer complete tracheids are used. His specimen size clearly makes any theoretical model of the specimen simple in that only tracheids need be considered, but great difficulty is introduced into the interpretation of the experimental results because of the unknown contribution of cut and incomplete cells, which constitute a considerable proportion of the sample. SADOH and CHRISTENSEN [1967] have shown that thin sections less than 80 μm thick distort during microtoming depending on the inclination of the microtome knife and that subsequent mechanical behaviour as evinced by shrinkage and swelling is affected. Because of their small size specimen gripping and the measurement of extension also become problems and it would seem that these difficulties coupled with the unknown effects of a large surface to volume ratio are responsible for the divergence between the strain behaviour in COWDREY and PRESTON's [1966] experiment and the present one where linear strains 100 times greater and longitudinal YOUNG's moduli (on wood substance basis) approximately 3 times greater are observed.

The cell wall cross-section area for the 2 mm × 2 mm specimens used here has been deduced by assuming a value for the density of wood substance (for which good values are now emerging [MARK 1967]) and measuring the mass per unit length of the sample. The cross-sectional area is then simply given by

$$\frac{\text{mass/unit length}}{\text{density of wood substance}}.$$

The method is free from the usual thin section technique objections of swelling of the sections due either to embedment and/or to release of restraint on microtoming.

Thus the uncertainties in the determination and interpretation of the stress-strain behaviour previously experienced by other workers have been eliminated in the present study by using larger specimens at the expense of complicating the theoretical model of the specimens. However, the model adopted readily provides a lower limit to the longitudinal YOUNG's modulus and in fact can be seen to produce extremely good agreement with the experimental data (Fig. 4).

It has already been mentioned that COWDREY and PRESTON's model of wood is fundamentally different from the author's. While their model is valid for an isolated piece of cell wall, it cannot describe the behaviour of a tissue such as wood composed of square or circular section tubes of the same material rigidly bonded together. Their model predicts that a rectangular sectioned element of wood material having sides parallel to the cell axis, and in which the mean microfibril angle is at an angle to the cell axis will distort into a rhomb under axial tension, as described by their function ψ. However, this type of distortion cannot take place in a tissue of identical cells made of this material because the bonded walls

of adjacent cells will tend to shear in opposite directions, as their microfibril angles are equally and oppositely disposed about the cell axis. Further, in axial tension the crossed structure provides a distinct stiffening effect; the value of the longitudinal YOUNG's modulus for COWDREY and PRESTON's model being only 63% that of an equivalent crossed structure of the same density at a mean angle of $M = 40°$ ($s = 10°$, $c_1 = 0.5$, on the basis of MARK's constants and HILL's theory). Thus the variation of the equivalent longitudinal YOUNG's modulus with mean microfibril angle for a single laminate falls more steeply (from identical values at $M = 0°$) than the curve given in Fig. 4, and so fits the experimental data less well.

Since both the crossed structure and the single laminate lead to quartic functions in sin M and cos M for the elastic constants, the curve fitting procedure used by COWDREY and PRESTON would conceal any deficiency of this nature in their model when comparing the shape of the experimental and theoretical curves. The theoretical parameters used in the author's model are on the other hand independent of the experimental data and so the predictions can provide a more definite test of the theoretical model. Fig. 4 shows that the model closely predicts in both form and magnitude the relation between longitudinal YOUNG's modulus and mean microfibril angle.

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I. D. CAVE, Physics and Engineering Laboratory,
Department of Scientific and Industrial Research, Lower Hutt, New Zealand.