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Longitudinal Shrinkage of Wood — Part I:

Longitudinal Shrinkage of Thin Sections

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Summary

The possibility of using microtomed longitudinal sections of wood for the study of longitudinal shrinkage has been investigated using hoop pine (*Araucaria cunninghamii* Ait.). Cutting of the sections was shown to distort them and to affect their subsequent shrinkage. However, by increasing the inclination angle of the microtome knife, sections could be cut without change in longitudinal dimensions. The longitudinal shrinkage behaviour of such sections more than 80 μm thick was little different from that of thicker sawn specimens.

Measurements of longitudinal shrinkage using this technique showed that very thin sections (40 μm) had greater longitudinal shrinkage than sawn wood. Partial delignification with sodium chlorite also increased longitudinal shrinkage. The longitudinal shrinkage was reproducible over successive cycles of moisture content change but not reversible, the length of a specimen at a given moisture content being less during desorption than at the same moisture content during adsorption. It is considered likely that the longitudinal shrinkage of wood is markedly affected by stresses which develop within the cell wall as the wood dries.

Zusammenfassung

Die Möglichkeit, Mikrotom-Längsschnitte zur Prüfung der Längsschwindung zu verwenden, wurde an Hoop Pine (*Araucaria cunninghamii* Ait.) geprüft. Dabei zeigte sich, daß die Längsschnitte beim Schneiden verschoben werden, was die nachfolgende Schwindung beeinflußt. Nachdem der Schnittwinkel des Mikrotommessers vergrößert worden war, konnten die Schnitte ohne Veränderung der Längsabmessungen abgetrennt werden. Das Längsschwindungs-Verhalten solcher Schnitte von über 80 μm Dicke unterschied sich nur wenig von demjenigen dickerer, gesägter Proben.

Messungen der Längsschwindung mit Hilfe des beschriebenen Verfahrens zeigten, daß sehr dünne Schnitte (40 μm) eine größere Längsschwindung aufweisen als gesägte Schnitte. Die teilweise Delignifizierung mit Natrium Chlorit erhöhte ebenfalls die Längsschwindung. Die Längsschwindung blieb während aufeinanderfolgender zyklischer Feuchtigkeitsänderungen reproduzierbar, war aber nicht umkehrbar. Die Länge einer Probe war, bei gleichem Feuchtigkeitsgehalt während der Desorption geringer als während der Adsorption. Es darf als wahrscheinlich angesehen werden, daß die Längsschwindung des Holzes deutlich durch Spannungen beeinflußt wird, die sich beim Trocknen des Holzes innerhalb der Zellwand entwickeln.

Introduction

This investigation is presented in two parts. Part I is concerned with a technique for the measurement of longitudinal shrinkage of wood and the use of this technique in studying factors affecting longitudinal shrinkage. Part II embodies suggestions for a model capable of accounting for the experimental observations.

Although the shrinking and swelling of wood have been studied extensively, the exact nature of the phenomena is not fully understood. This is particularly true of the manner in which shrinking and swelling are related to wood structure, a subject reviewed recently by K. E. KELSEY [1963]. The shrinkage of wood is highly anisotropic, the greatest difference occurring between directions perpendicular (transverse) and parallel (longitudinal) to the axis of the tree. The trans-

verse shrinkage has received much more attention for several reasons. It is of greater practical significance in wood utilization and is easier to study experimentally. However, longitudinal shrinkage also is of importance in some aspects of wood technology and must be considered in relation to the accompanying transverse shrinkage before a clear understanding of any of these dimensional changes is possible.

In the light of K. E. KELSEY's paper, it is not necessary to review fully the present state of knowledge regarding longitudinal shrinkage. There are, however, one or two features additional to those discussed in detail by K. E. KELSEY, which are of some relevance to the present paper.

It was apparent from the results of A. KOEHLER [1930, 1946] that the manner in which longitudinal shrinkage changes as wood is dried differs significantly from that of transverse shrinkage. Transverse shrinkage commences at a moisture content in the vicinity of 30% (in the absence of collapse) and increases linearly with decreasing moisture content over a considerable moisture content range. By contrast, longitudinal shrinkage is usually negligible or may even be negative in drying from the green condition to approximately 12% moisture content and then increases with further drying.

Unpublished results by K. E. KELSEY for small specimens of both softwood and hardwood have confirmed this pattern of longitudinal shrinkage, the moisture content often dropping to 8% before significant longitudinal shrinkage commenced. Tests on a number of species in the form of veneer have also shown that between 45% and 85% of the total longitudinal shrinkage occurred below 8% moisture content [GOTTSTEIN, STASHEVSKI and MOGLIA, unpublished data].

A further unreported observation of K. E. KELSEY's was that longitudinal shrinkage exhibited an unusual form of hysteresis with moisture content, in that the length of a wood sample at a given equilibrium moisture content during drying was less than that at the same moisture content during swelling. That is to say, the dimensional change tended to precede the moisture content change producing it.

Because longitudinal shrinkage is small (typically 0.4% of the green dimension compared with 6 to 12% for transverse shrinkage), much more accurate measurements are necessary than when studying transverse shrinkage. Apart from improving the mechanics of marking and measuring specimens, other spurious effects, which may be ignored in transverse measurement, must be recognized and reduced to negligible proportions.

In drying blocks of wood, any moisture gradients present will produce drying stresses which may affect the total shrinkage. In some species collapse also may occur. These effects will be relatively more important in the measurement of longitudinal than of transverse shrinkage [KÜHNE and VODOZ 1951; VODOZ 1953].

The use of sufficiently thin longitudinal sections should eliminate the problems associated with both moisture gradients and collapse. Additional advantages, which might be expected from the use of thin sections, are increased speed of moisture content equilibration and greater latitude in selection and matching of test material.

At the same time, however, the use of thin sections may introduce new difficulties. As pointed out by W. L. GREENHILL [1936], boiling a block in water prior

to sectioning may lead directly to changes in dimensions, an observation which has been amply confirmed since [e.g. MACLEAN 1952; KÜBLER 1959; YOKOTA and TARKOW 1961, 1962; GRZECZYŃSKI 1962]. Species which may be sectioned green without heating, e.g. species of low density, might therefore be preferred on these grounds. A further difficulty is the distortion of the wood that must occur during the cutting process. This may lead to the section having final dimensions which are different from those of the parent block. T. KISHIMA and S. HAYASHI [1959] observed tangential elongation and radial contraction in cross-sections after cutting, and suggested that release from restraint by rays, cutting force and clamp pressure were responsible. Release of growth stresses could produce a similar effect.

The extent to which the subsequent longitudinal shrinkage of sections might be affected by the sectioning process is not known. Effects have certainly been observed with transverse sections. Thus S. H. CLARKE [1930], and M. LAWNICZAK and co-workers [1956] contended that transverse shrinkage and swelling of such sections were greater than those of the parent blocks. On the other hand, F. W. LINDSAY and L. CHALK [1954] found tangential shrinkage was reduced.

The possible advantages to be gained by the use of thin sections, however, appeared to be such as to warrant a study of these, at least for comparative measurements of longitudinal shrinkage of wood. The first part of this paper is therefore concerned with examining the shrinkage behaviour of microtomed longitudinal sections in comparison with that of thicker sawn specimens.

Apparatus and Techniques

Test Material and Specimen Preparation

All measurements in Part I of this investigation were made on hoop pine (*Araucaria cunninghamii* Ait.), a softwood of comparatively uniform structure with only narrow rays. The mean basic density of the material used (weight of wood substance per unit green volume) was 0.45 g/cm³ and the average fibre daimeter (radial direction) approx. 40 μm.

Suitable blocks free from compression wood were prepared having dimensions 1.3 cm (tangential) × 5.0 cm (radial) × 7.5 cm (longitudinal) with the longitudinal direction as nearly parallel to the fibre axis as possible. Specimens were cut from the tangential face of these blocks with a sliding microtome in the case of the thin sections (40 μm ... 200μm) and a smooth-cutting circular saw for the thicker samples (1 mm). In cutting the thin sections, the knife movement was parallel to the longitudinal direction of the wood but the horizontal angle between the knife edge and the normal to this direction (inclination angle) was varied as described below. It was ultimately found that the best results were obtained when the inclination angle was a maximum, namely 82°. All specimens were cut from unheated green wood and placed immediately in distilled water.

To prepare specimens for shrinkage measurement, each 7.5 cm × 1.3 cm longitudinal-tangential section was halved lengthwise, but the thicker sawn specimens were measured without further cutting. To each end of each thin section a small tag of brass shim was attached with wax by local heating. A hole through each tag provided means for holding and supporting the specimen in the measuring chamber (see below).

Some difficulty was experienced in making suitable reference marks on the specimens, but this was resolved finally by attaching a very fine filament of black wax. A short length (1/4 mm) of filament was placed on the surface of the damp wood section and covered with a small grain of low melting-point paraffin wax. The latter was then melted with a hot wire, thus fixing the unmelted black filament in position. The area of wood affected did not exceed a square millimetre. Three such marks were placed across the width at each end of the length to be measured (4 cm), allowing three separate estimates of longitudinal shrinkage to be made on each specimen. The presence of any edge effects could therefore be detected. The full length of the specimen was not included in the measured distance in case restraints caused by the brass tags affected the longitudinal shrinkage near the ends of the specimen.

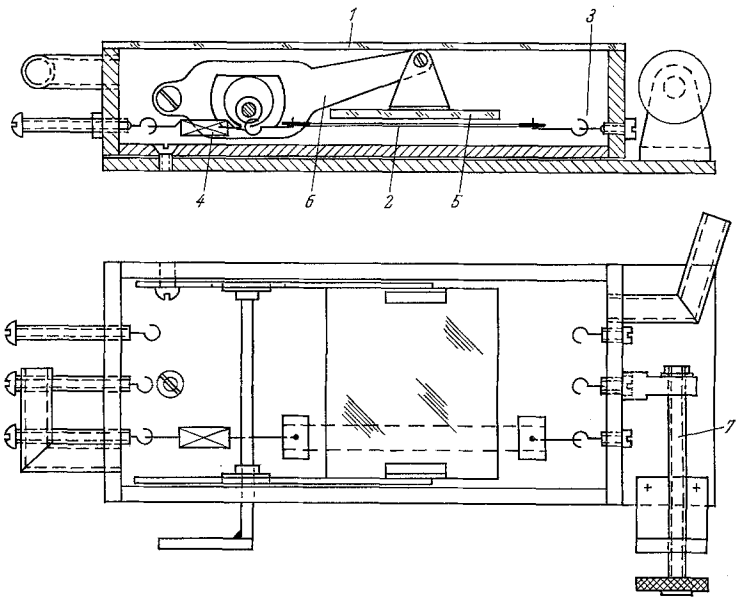

Fig. 1. Chamber holding specimens for conditioning and measuring.
1 Glass plate, *2* Microtomed section, *3* Hooks, *4* Spring, *5* Glass plate, *6* Lever, *7* Screw.

Shrinkage Measurements

The test specimens were placed in a small chamber through which air of controlled relative humidity could be circulated, and their shrinkage was measured with a travelling microscope.

The chamber consisted of a shallow rectangular brass box which could be covered and sealed with a flat glass plate (*1*) (Fig. 1). It held three specimens simultaneously. Each of the thin microtomed sections (*2*) was suspended horizontally in the chamber from two hooks (*3*) with a very light spring (*4*) included to accommodate length changes. The maximum tension in the spring, equivalent to 0.5 g weight was negligible compared with the strength of even the thinnest section. The reproducibility of successive cycles of shrinking and swelling finally obtained (see below) confirmed that no significant creep was produced by the very slight tension. By suspending the specimen slightly above the base of the

chamber during conditioning, moisture content gradients were minimized. During measurement, however, the specimens were pressed lightly on to the flat base of the container by a small glass plate having smoothed edges (*5*) which covered the measured length. This operation was controlled externally by a lever (*6*). The 1 mm thick specimens were measured in a similar chamber but were supported by three horizontal bars rather than hooks and springs.

Measurements were made with a travelling microscope capable of being read by drum and vernier to 0.00025 mm but the final accuracy of measurement was probably not better than $\pm$ 0.001 mm. The test chamber was clamped on the moving stage of the microscope but could be rotated horizontally by means of a screw (*7*). In this way, each pair of reference marks could be aligned separately with the direction of travel of the stage. There were no consistent differences between the shrinkages as measured between the outer and inner pairs of marks; that is, no edge effects were apparent. The shrinkage of each specimen was therefore expressed as the mean of the three values. Measurements of the length of the initially wet sections were made before placing them in the chamber. They were laid on wet filter paper and covered with a glass plate to prevent drying during measurement.

In some cases, measurement of transverse dimensions was also made. For this purpose, five additional pairs of marks were placed close to the opposite edges of the specimen at equal intervals along its length. The transverse dimensions were measured simultaneously using the same microscope by moving the stage at right angles with a drum micrometer capable of being read to 0.0025 mm.

Humidity and Moisture Content Control

All measurements were made in a constant temperature room at 25 $\pm$ 0.2 °C. The humidity within the chamber was controlled by means of saturated salt solutions. Recirculated air was passed first through a bubbler containing an appropriate solution (or over a drying agent to produce dry air) and a trap to remove any spray, and then through the measuring chamber. Direct measurement of the moisture content of the measured specimens was not practicable. Instead, a matched specimen was suspended from a quartz helix in a glass tube through which the air leaving the measuring chamber was passed. The moisture content of this specimen, as indicated by the length of the helix, was followed with a cathetometer. Shrinkage measurements were made when moisture content equilibrium had been reached. The relative humidity was not measured.

In all cases, experiments commenced with the specimens in the green state. The moisture content was reduced in one or more steps, generally to less than 1%. The moisture contents were then raised until finally the specimens were removed and immersed in water in order to determine their fully re-swollen dimensions. For some purposes, the cycle of drying and re-wetting was repeated several times.

Initial Experiments with Microtomed Sections

Irreversible Changes Associated with Thin Sections

The first shrinkage measurements were made on three 50 μm sections cut with the inclination angle of the microtome knife at approximately 45°. These shrinkages were compared with those of an adjacent sawn specimen 1 mm thick from the same block. The sections were subjected to three cycles of drying and rewetting

and the 1 mm specimen to one such cycle. The results are shown in Figs. 2 and 3 respectively. It may be seen from Fig. 2 that the shrinkage during the initial desorption of the thin sections was at first negative (elongation), but that it increased in a normal manner as dryness was approached. At the end of the first adsorption which culminated in re-immersion of the specimen in water, the length was 0.2% greater than in the original green state. After the next desorption, the dry length of the sections had also increased, but over the final one and half cycles neither the wet nor dry dimensions changed appreciably.

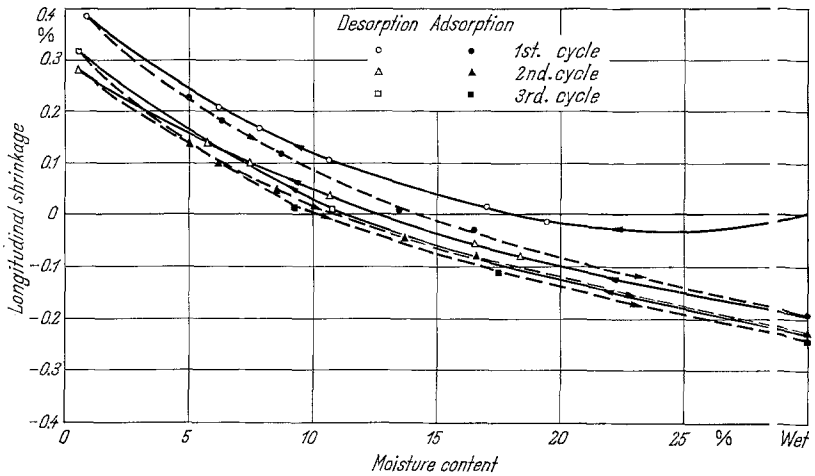

Fig. 2. Longitudinal shrinkage of 50 μm sections of hoop pine microtomed with the knife at an inclination angle of 45°.

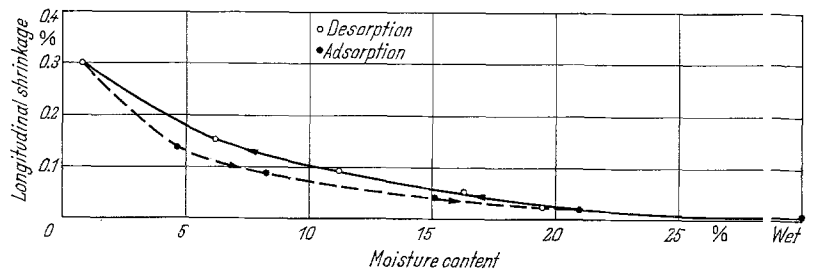

Fig. 3. Longitudinal shrinkage of a 1 mm thick sawn specimen matched with the sections in Fig. 2.

With the 1 mm specimen, on the other hand, the shrinkage was positive at all moisture contents (Fig. 3). Only one cycle of moisture content change was used in this case and at the end, after immersion of the sample, a small residual shrinkage persisted. The total shrinkage of this specimen (0.3%) was less than the total shrinkage of the 50 μm section (0.5%) during the latter's second and third cycles. The 1 mm sawn specimen took approximately three times as long to reach equilibrium as the microtomed sections, but the relative rates of sorption differed with the humidity to which the specimens were exposed.

The changes in wet and dry dimensions of the thin sections over the first two cycles suggest that the longitudinal shrinkage was affected by stresses or deforma-

tions, either present in the wood initially or introduced during cutting. The fact that freshly cut sections tended to curl suggests that the stress pattern was affected by the sectioning. The final excess length of the sections in the re-swollen state may thus have represented either partial or complete recovery from these stress changes.

Deformations introduced during cutting might be expected to be more severe the thinner the sections. In the next experiment, therefore, the behaviour of sections of different thicknesses was determined. At the same time, a test was made to see whether the increase in length produced by a cycle of moisture content change could also be achieved by soaking the freshly cut sections in hot water. The changes in swollen dimensions of two groups of matched specimens, 50μm, 100μm, 200μm and 1 mm thick were compared (Fig. 4), one group after a cycle of shrinking and re-swelling and the other after soaking for 24 hrs in water at 50 °C.

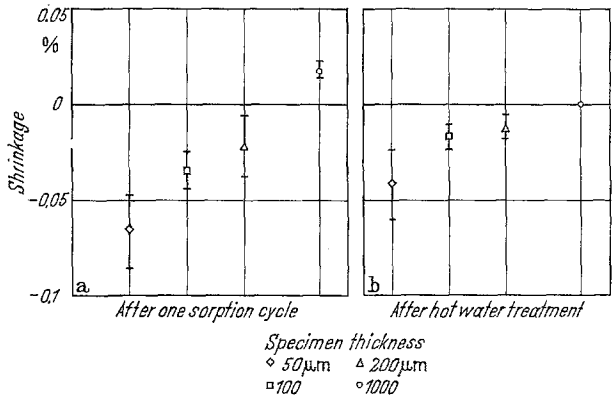

Fig. 4. Final change in longitudinal dimension of specimens of different thickness, (a) after one sorption cycle, (b) after hot water treatment. Standard deviation is indicated.

With the thin sections subjected to shrinking and swelling (Fig. 4a), there was a residual elongation which was greater the thinner the section, as expected. The 1 mm sample, on the other hand, showed a slight residual shrinkage as observed previously in Fig. 3. For the matched specimens given the hot water treatment (Fig. 4b), the elongation of the thin sections was less, and no change occurred in the 1 mm sample. It thus appeared that the hot water treatment was not as effective in producing recovery as a cycle of shrinking and re-swelling.

Changes in Dimensions on Cutting Thin Sections

The presence of distortions produced during cutting might well be shown by comparing the dimensions of the freshly cut sections with those of the surface of the parent block from which they were cut. This comparison was made for 7.5 cm × 1.3 cm microtomed and sawn specimens using an inclination angle of 45° initially, but subsequently increasing this angle to 82°.

The outcome of these measurements is shown in the first column of results in Table 1. Positive values indicate shortening of the specimen, in conformity with the previous graphs depicting shrinkage.

Considering firstly the inclination angle of 45°, it can be seen that in both the longitudinal and transverse directions, the microtomed sections shortened by an

amount which was greater the thinner the section. No dimension change was observed with the 1 mm sawn specimen. The extent to which this contraction was reduced by subsequent boiling of the sections is apparent from the second column of the table. Similar experiments were also made using blocks which were boiled before cutting of specimens. The changes in dimensions of the block itself on boiling were negligible, amounting to 0.01% expansion in both the longitudinal and tangential directions. Sections cut from the boiled block also showed contractions (third column) which were only slightly less than those of the specimens cut from the green blocks prior to boiling. Further boiling of these sections again led to partial recovery (fourth column).

From these results it is clear that the original stresses in the wood contributed very little to the non-reproducibility of the first shrinking and swelling cycle. The most important factor was obviously the distortion associated with the cutting action of the microtome knife.

Clearly, if any use was to be made of microtomed sections for shrinkage studies, reduction of this distortion was essential. In a further attempt to do this, the inclination angle was increased to the maximum possible, namely 82°. In this way, the "slicing" action of the knife was increased and direct pressure in the longitudinal direction of the wood decreased. Sections thus prepared were measured and treated in exactly the same way as described above for the 45° specimens and these results also are shown in Table 1.

Table 1. *Change in Dimensions During Cutting of Specimens*

Inclination Angle	Thickness of Specimens (μm)	Direction	Change in Dimensions Relative to Green Blocks %[1]			
			Sections Cut from Green Block		Sections Cut from Block Boiled before Cutting	
			After Cutting	After Boiling Sections	After Cutting	After Boiling Sections
45°	50	Longitudinal	0.68	0.55	0.54	0.39
	100		0.35	0.29	0.35	0.25
	200		0.28	0.26	0.26	0.17
82°	50		−0.01	0.01	0.02	0.02
	100		−0.02	0.00	0.01	0.01
	200		−0.02	−0.01	−0.01	0.00
—	1 mm (sawn)		0.00	0.00	0.01	−0.01
45°	50	Transverse	1.92	0.92	1.73	0.96
	100		1.38	0.61	1.38	0.67
	200		1.14	0.61	1.04	0.53
82°	50		2.02	0.82	1.97	1.00
	100		1.56	0.80	1.57	0.69
	200		1.15	0.46	1.06	0.51
—	1 mm (sawn)		0.00	0.01	0.00	0.01

[1] Positive values indicate shortening of specimen.

The effect of this method of cutting in reducing distortion in the longitudinal direction is immediately apparent in that negligible longitudinal changes occurred

either with or without boiling. However, this was achieved only at the expense of a possible increase in the tangential distortion.

Shrinkage of Sections Free from Longitudinal Distortions

It remained to determine whether, despite the tangential distortion, the longitudinal shrinkage of sections cut with an inclination angle of 82° was comparable with that of the undistorted 1 mm sawn specimens. Measurements were made using three sections cut at each thickness, 50, 100, 200 μm and 1 mm. Both the total shrinkage to the dry state and the residual shrinkage after re-wetting were determined, and the results, which were statistically analysed, are shown in Table 2.

Table 2. *Total Longitudinal Shrinkage to Dryness and Residual Longitudinal Shrinkage after Re-wetting, of Microtomed Sections Cut at an 82° Angle Compared with Sawn Specimens*[1]

Specimen	Longitudinal Shrinkage to Dryness (%)				Recovery in Longitudinal Dimension after a Sorption Cycle (Wet State) (%)			
	Mean	Standard Deviation	Standard Error	[2]	Mean	Standard Deviation	Standard Error	[3]
Micromoted								
50 μm	0.387	0.0437	0.0146	+	0.0070	0.0061	0.0020	+
100 μm	0.314	0.0208	0.0069	—	−0.0016	0.0040	0.0013	—
200 μm	0.310	0.0244	0.0081	—	0.0046	0.0087	0.0029	—
Sawn								
1 mm	0.290	0.0049	0.0017		0.0129	0.0029	0.0010	+

[1] Measurement was made on three specimens for each thickness and three positions for each specimen.

[2] +, Plus sign indicates that mean value is significantly different from that of the 1 mm thick specimen.

[3] +, Plus sign indicates that mean value is significantly different from zero.

Considering firstly the total shrinkage to dryness (less than 1% moisture content) values were greater for the sections than for the 1 mm specimens, although the difference was significant for the 50 μm section only. It is believed that this difference is associated with the actual thickness of the sample rather than with secondary effects associated with the cutting process and will be studied further below. Also, the standard deviation and standard error were considerably greater for the thin sections. Possibly the 1 mm specimen tends to average out the effects of minor structural variations which can affect thin sections and, in this respect, 1 mm samples may be considered to be preferable to microtomed sections. The statistical analysis showed also that there were no significant differences between any three specimens cut at one thickness, nor any significant, effects between the three positions (middle and two outer) across the width of each specimen at which longitudinal shrinkage was measured.

When the specimens were re-wetted, it was found that while the sawn wood had a residual longitudinal shrinkage of approximately 0.013%, the sections finished much closer to their original green dimensions. This experiment adds to the evidence in Table 1 that sections cut at 82° had little distortion in the longitudinal direction, since their swollen length was unchanged not only during cutting and subsequent boiling, but also after a sorption cycle.

Finally, to confirm that the shrinkage behaviour was reproducible, three 50 μm sections cut at an inclination angle of 82° were submitted to two complete cycles of desorption and adsorption (Fig. 5). The graphs of shrinking and swelling (mean of the three specimens) show that the first cycle was reproduced almost exactly by the second. The corresponding cycle for the 1 mm specimen is also shown in Fig. 5. These results show that, even though the irreversible effects in the 50 μm sections originally apparent in Fig. 2. were eliminated, the total longitudinal shrinkage remained higher than that of the 1 mm specimen.

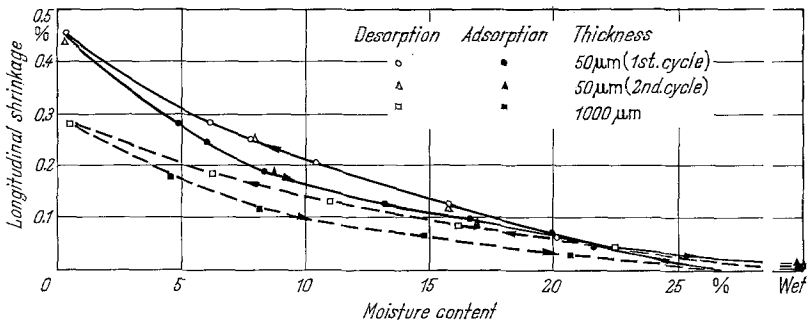

Fig. 5. Comparison of longitudinal shrinkage of 50 μm specimens microtomed at 82° inclination angle and a sawn 1 mm specimen.

Summarizing the results so far obtained, it has been established that the longitudinal shrinkage of thin sections of hoop pine cut at an inclination angle of 82° differs only slightly from that of sawn specimens 1 mm thick. The total shrinkage to dryness of the thinnest of these sections appears to be greater than that of the sawn wood for reasons yet to be discussed. On the other hand, the first cycle of drying and re-wetting of the thin sections is more nearly reproducible than that of the 1 mm specimens which show a small residual shrinkage after the first cycle. It would appear that either type of specimen can be used for some aspects of the study of longitudinal shrinkage, provided the distortion introduced in the transverse direction during sectioning is not likely to affect the results.

Exploratory Studies of Longitudinal Shrinkage using Thin Sections

In the remainder of Part I of this study, thin sections are used for the further examination of some characteristics of the longitudinal shrinkage of wood. These are essentially exploratory tests which may be carried out readily using thin specimens, but might be more difficult with larger samples. The particular aspects covered include the change in longitudinal shrinkage with change in thickness of section, the effect of progressive delignification and the phenomenon of hysteresis in the longitudinal shrinkage-moisture content relationship.

Effect of Specimen Thickness

Attention has already been drawn to the fact that the longitudinal shrinkage of the wood specimens increased with decreasing thickness.

In attempts to explain the phenomenon of wood shrinking and swelling, it is commonly supposed that restraint of the free swelling of the cell wall material occurs. That is to say that if a small portion of a cell wall could be removed and

its swelling behaviour studied separately, it would be found to shrink and swell more than it apparently does in the intact fibre.

One of these restraints is thought to arise from the helical arrangement of the microfibrils within the cell wall. It is thus likely that if the cell wall were cut longitudinally through to the lumen, a change in the pattern of shrinkage could result. However, there are at least two other possible sources of restraint to the swelling and shrinking of whole wood. One of these is the bonding of adjacent fibres through the middle lamella, so that the shrinkage of one fibre is affected by the shrinkage of its neighbours. The other is that, as a result of the lamellar structure of the cell wall, there is a possibility that the cell wall shrinkage is itself anisotropic in the transverse plane. If this is so, shrinkage of the fibre as an entity must lead to the development of shear stresses and strains within the cell wall. Some of these stresses may have components in the longitudinal direction of the wood. The implications of these restraints to longitudinal shrinkage will be considered in more detail when hysteresis is discussed and also in Part II of this investigation.

Experimental separation of these various restraints may be very difficult, if not impossible, but attempts have been made, for example, by studying the shrinkage of separate fibres in order to eliminate effects of interfibre bonding. However, the process of separation is necessarily drastic and secondary changes may well be produced within the fibres themselves. The use of thin longitudinal sections provides an alternative approach to this problem of simplifying the structure of the wood specimen to be studied.

In preparing specimens of any thickness for longitudinal shrinkage measurement, the outer fibres forming the specimen surface will include a large number from which a significant portion of the cell wall has been removed. As the thickness of the specimen is reduced, the proportion of cut fibres in the sample will rise. If the thickness is reduced to that of the average fibre diameter or less, all but a fraction of small diameter fibres will have been cut. It seemed likely that the increased shrinkages already observed in the thin sections may have resulted from the reduction of restraints normally arising during the shrinkage of whole fibres and it was decided to study this effect a little further.

Matched sections of hoop pine (mean fibre diameter approximately 40 μm) at thicknesses of 40, 60, 80, 100, 120 and 150 μm were used. Sawn 1 mm specimens matched with the above sections were also used. The thinnest sections could be expected to contain only a few whole fibres. The results of shrinkage measurements over a single drying-rewetting cycle are shown in Fig. 6. To show more clearly the effect of specimen thickness on the shrinkage at any moisture content, the results are represented in a different form in Fig. 7.

These results show that the increased shrinkage was most marked for specimens less than 80 μm thick were the proportion of cut fibres increased rapidly with decrease in thickness. If the fibres were all of uniform size and randomly distributed in the wood (that is, there were no linear arrays), the proportion of fibres which would be cut may be shown to equal D/T where D is the fibre diameter and T the specimen thickness. If non-randomness and fibre diameter variation are ignored in the present tests, it is possible to plot the longitudinal shrinkage of the sections against the fraction D/T of cut fibres. This has been done in Fig. 8, which includes also four points obtained later from sections of earlywood of *Pinus radiata*

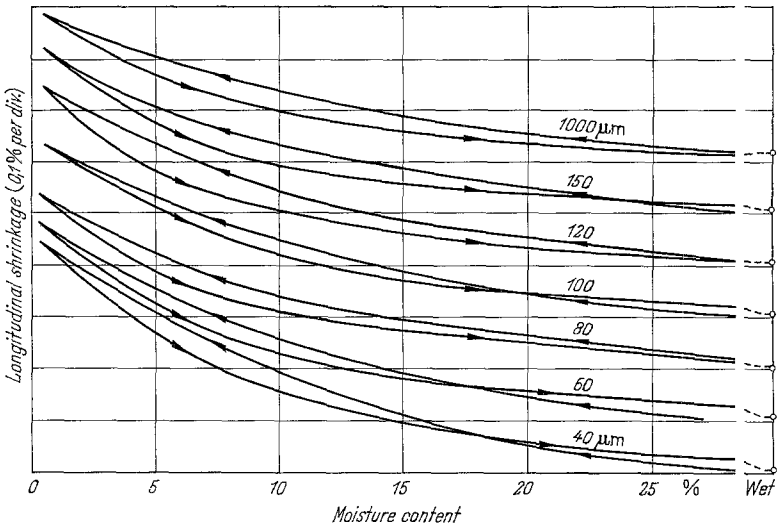

Fig. 6. Effect of specimen thickness on longitudinal shrinkage over one sorption cycle.

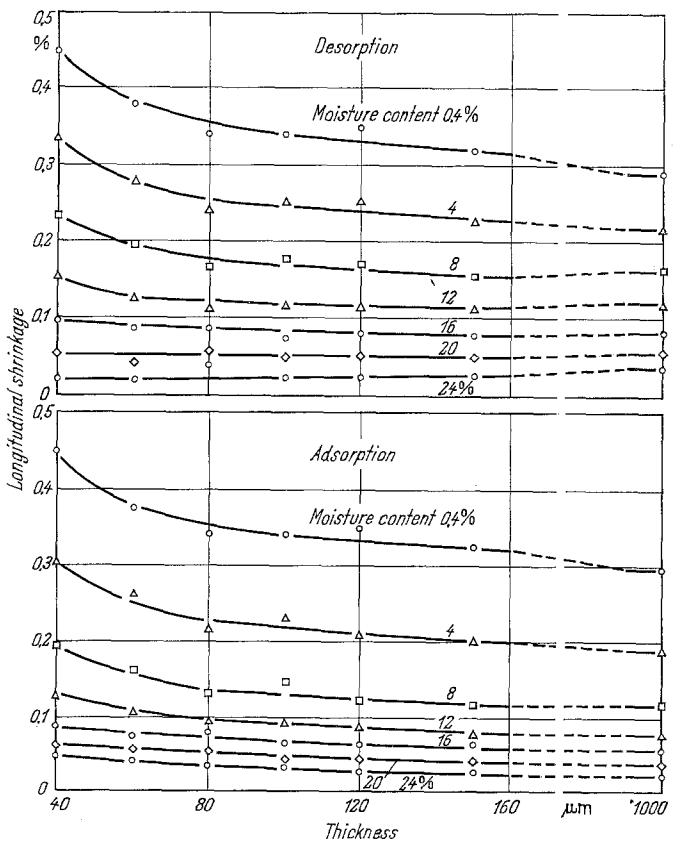

Fig. 7. Longitudinal shrinkage as a function of specimen thickness at various moisture contents.

(D. Don) wood (see Part II). A close approximation to a linear relationship was obtained suggesting that the change in longitudinal shrinkage was proportional to the fraction of cut fibres. An exactly linear relationship between shrinkage and D/T would imply that other restraints were either non-existent or were also reduced at the same rate with change of thickness. No definite conclusion can be drawn at this stage, however, since the slight scatter of the present results may mask some departure from linearity.

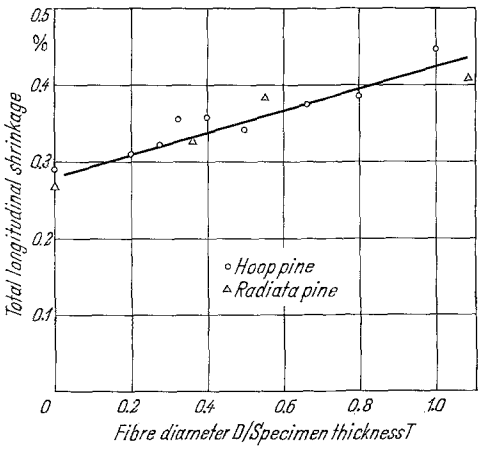

Fig. 8. Dependence of total longitudinal shrinkage on the ratio fibre diameter/section thickness.

Further information which would be valuable would be to determine the effect on longitudinal shrinkage of further reduction in specimen thickness, and also the effect on the corresponding transverse shrinkage. This latter would perhaps be meaningless if measured on the thinnest sections since the possibilities of introducing distortion due to broken cells could produce overall transverse dimension changes quite unrelated to the transverse shrinkage of the whole wood. Knowledge of accompanying changes in the volumetric shrinkage of the cell wall would also be valuable and could be deduced from changes in the sorption isotherms.

On this last point some information is available from published [CHRISTENSEN and KELSEY 1959, CHRISTENSEN 1960] and unpublished data. Of four species which have been examined, two having cell walls which were thick in relation to the cell diameter showed increases in equilibrium moisture content when they were cut into 20μm thick longitudinal sections. The other two were species of lower density having thin walls, and their sections showed no change in equilibrium

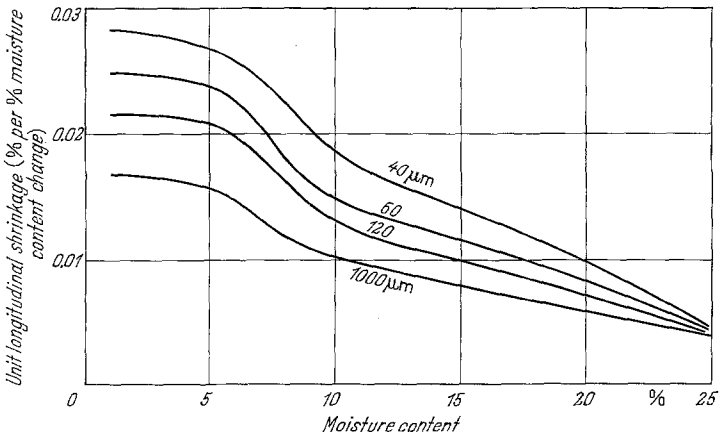

Fig. 9. Change in unit longitudinal shrinkage with moisture content.

moisture content below a relative vapour pressure of 0.75. One of these lower density species was *Araucaria hunsteinii* K. SCHUM., a species similar to the hoop pine of the present tests. The results of these sorption studies suggest that, in uncut thick-walled fibres, the restraints to swelling are sufficiently large to produce, not only shear stresses and strains, but reductions in the volumetric swelling of the wood substance as well.

Some further observations relating to the results presented in Fig. 6 may be mentioned. Reduction in specimen thickness, although increasing the shrinkage at each moisture content, did not markedly change the shape of the shrinkage curves. A clearer indication of this may be obtained by plotting the unit longitudinal shrinkage (shrinkage per 1% change in moisture content) against the moisture content. This has been done in Fig. 9 for the drying process for four of the thicknesses. The similarity in shape of the curves is apparent. This figure also shows that, although the unit longitudinal shrinkage increased throughout the drying process, the rate of increase was noticeably greater in the range of moisture content from 5 to 10%.

Swelling hysteresis also persisted at all specimen thicknesses but this will be discussed separately.

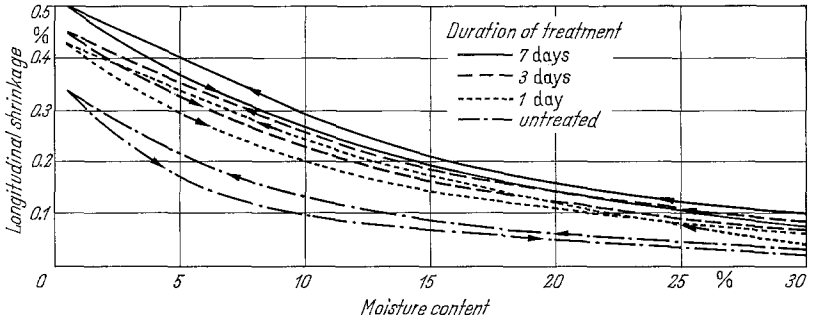

Fig. 10. Effect of partial delignification on longitudinal shrinkage.

Effect of Partial Delignification

The possibility that inter-fibre bonding may restrain shrinkage has also been mentioned. Although this source of restraint can be eliminated by studying single fibres, the use of thin samples permits of an alternative approach.

This approach is based on the fact that the distribution of lignin in wood is not uniform, being more highly concentrated in the region of the middle lamella and the outer layers of the cell wall. Partial delignification of wood can readily be achieved by treating small wood samples with acidified sodium chlorite. The resulting sample remains sufficiently coherent to be handled and measured as easily as the original wood. The removal of lignin from the inter-fibre layer, even though fibre separation is not complete, might well permit greater relative movement of adjacent cell walls during shrinkage and swelling, thereby reducing restraints from this cause. The likelihood of other changes in the fibres, due to the chemical treatment, still persists, however.

The effect of partial delignification on longitudinal shrinkage was studied on matched 100 μm sections of hoop pine. A drying and re-wetting cycle was first performed on six of the freshly cut sections and the mean shrinkage of these is shown

as the reference (untreated) curve in Fig. 10. These specimens, and others for lignin analysis and sorption measurements, were then immersed in acidified sodium chlorite (10% solution) at room temperature and removed in groups after 1, 3 and 7 days respectively. After thoroughly washing the sections in distilled water, the change in dimension resulting from the sodium chlorite treatment was determined. Then the longitudinal shrinkage was again determined during a drying and re-wetting cycle.

It was necessary to determine the sorption behaviour of the differently treated specimens since their equilibrium moisture contents differed at any humidity. The humidity of the conditioning air was not measured directly, but the moisture contents of the treated specimens relative to that of wood were determined at several humidities and found to be constant for each treatment [see also CHRISTENSEN 1959, Table 8]. The mean sorptive capacities relative to the untreated wood in the present tests were 1.13, 1.23 and 1.27 in decreasing order of lignin content. During the swelling cycle, the equilibrium moisture content of the untreated wood only was measured and the equilibrium moisture contents of the delignified specimens were calculated using the above ratios.

The lignin content was determined by the method of D. M. HALSE [1926] using additional matched specimens delignified at the same time as the shrinkage specimens. The lignin contents were 35, 19, 11 and 8% of the dry specimens for the original wood and the progressively delignified samples respectively.

Following the delignification treatment, the measured length of the specimens was found to have increased by 0.358, 0.432 and 0.514% for the three treatments. The reason for this elongation is not known, but one possibility is that the partial removal of the lignin permitted closer alignment of the microfibrils perhaps with a decrease in the average fibril angle. It is also possible that removal of lignin permitted greater swelling of the remaining tissue in the wet state, an effect which is also reflected in the higher equilibrium moisture contents of the delignified wood.

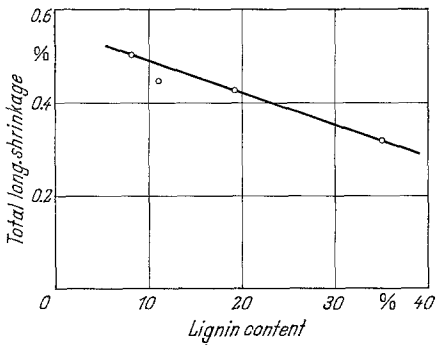

Fig. 11. Variation in total longitudinal shrinkage with lignin content.

The subsequent cycle of shrinking and swelling measured from the new wet length is shown in Fig. 10 for comparison with the mean behaviour of the same samples before delignification. It is at once apparent that as the delignification increased so too did the longitudinal shrinkage. Some increase in the total shrinkage to dryness could be expected to result from any excess swelling produced during delignification. When total shrinkage was expressed relative to the dimensions of the original wood, however, the final shrinkages in the dry state were 0.071, 0.017 and −0.014% respectively for the treated specimens compared with the original 0.33% for the untreated wood.

This suggests that despite the possible swelling during delignification, some structural re-orientations had occurred as well, at least by the time the sample was dry. This may also be reflected in changes in profile of the shrinkage curves in

which shrinkage increased rather more rapidly at high moisture contents and showed somewhat less curvature than was present in the control graph. Nevertheless, any re-orientation occurring during drying must have been largely reversible since, on re-wetting, the specimen dimensions were little changed from those in the freshly delignified condition.

Considering the distribution of the original lignin through the cell wall, it is possible that the changes in shrinkage behaviour and possibly microfibril re-orientations are the result of increased opportunity for relative displacement of the walls of adjacent fibres. A further observation from this study is shown in Fig. 11. This suggests that the change in the total longitudinal shrinkage of these specimens was a linear function of their lignin content. This point requires confirmation and further study.

Shrinkage-Moisture Content Hysteresis

Reference has already been made to the occurrence of an unusual form of hysteresis in the relationship between longitudinal shrinkage and moisture content. On the other hand, there is little evidence that transverse shrinkage is not reversible. If these observations are correct, it follows that there must be a hysteresis in the relationship between moisture content and either the cell wall volume or the cell wall thickness, without however producing a corresponding hysteresis in the external cell diameter. Neither of these possibilities appears likely, and a more probable explanation lies in the relative magnitudes of transverse and longitudinal shrinkage. If the hysteresis associated with longitudinal shrinkage was compensated by a corresponding but opposite hysteresis in shrinkage in both the transverse directions, the latter hysteresis would be so small as to be barely detectable using common methods of measurement.

Thus, if the maximum hysteresis in longitudinal shrinkage is 0.05% of the original length, the corresponding transverse shrinkage hysteresis would have a maximum value of 0.025% of the original dimensions. If this occurred in a total transverse shrinkage of say 10%, an accuracy in the measurement of transverse shrinkage of 1 part in 400 would be necessary to just detect the hysteresis at its maximum. Careful measurements by K. E. KELSEY (unpublished) indicated that hysteresis of this order of magnitude probably does occur in transverse shrinkage.

The fact that the form of hysteresis in transverse shrinkage is "normal", that is, the change in dimension lags behind the moisture content change producing it, suggests that the origin of the hysteresis really lies in the transverse shrinkage behaviour. The longitudinal shrinkage hysteresis would then be an inverse effect involving changes in shape of cell wall segments, consequent upon the volume of the cell wall being uniquely determined by the moisture content, whether adsorbing or desorbing.

If this interpretation is correct, the occurrence of strains giving rise to the hysteresis supports the idea that significant stresses are generated within the wood during swelling and shrinking. It has been shown [CHRISTENSEN 1962] that even in small wood samples, a stress applied externally to wood undergoing moisture content change can lead to changes in shape in a manner superficially resembling plastic deformation, and that these changes in shape can be reversed only by removing the stress and again changing the moisture content. In the present experiments, no external forces are applied and the stresses are developed internally

as the wood shrinks. The resultant change in shape ensures that during re-swelling the pattern of stresses will be different, thus giving rise to swelling hysteresis.

The magnitude of the hysteresis measured in experiments such as these may give some indication of the relative magnitudes of the stresses present. However, the accuracy of hysteresis measurement is limited by the errors in plotting the shrinkage graphs.

Reference to earlier results of this study, e.g. Fig. 6, suggests that the origins of the hysteresis are rather complex. The graphs depicting swelling and shrinking in a typical cycle tend to cross at high moisture contents (above 18%), this "cross-over" happening too frequently to be attributed to errors of measurement. There is even some evidence that the "cross-over" occurs at progressively lower moisture contents as the specimen is made thinner. However, there was little evidence of crossing of the curves with sawn specimens. No reason for these effects can be offered at present.

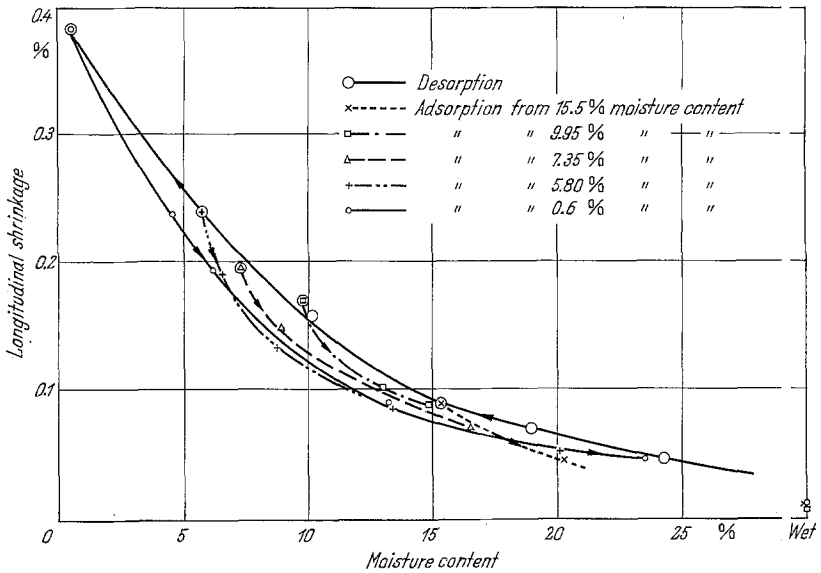

Fig. 12. Hysteresis in longitudinal shrinkage showing the effect of reversal of moisture content change at different moisture content values.

One additional experiment that was possible due to the comparatively fast sorption rates of thin specimens, was performed in a further study of hysteresis. In this, a single group of three specimens was subjected to a series of shrinking and swelling cycles having different lower limits of moisture content. The fresh green specimens were first dried in steps to 76% relative humidity (15.5% moisture content), then allowed to re-adsorb water vapour, again in steps, to finish in the wet state. The next cycle extended the drying to 52% relative humidity (9.95% moisture content), before re-wetting. The lower limits of the other cycles were 33, 18 and 0% relative humidity. The results (mean for three specimens) are shown in Fig. 12. For clarity, only the first part of the adsorption curves are drawn.

Despite the scatter evident in the plotted points, it is apparent that on reversing the direction of the moisture content change from desorption to adsorption,

the swelling curves rapidly crossed the hysteresis loop to the curve representing swelling from dryness. In terms of the previous discussion, this suggests that the change in shape of the cell wall produced by the internal stresses reached a limit after only a small moisture content increment and that thereafter further change in moisture content in the same direction had little effect on shape. Elaboration of this type of experiment can be readily suggested and the fuller interpretation of these hysteresis effects must await such further study.

Conclusions

To conclude Part I of this investigation, the main experimental observations and their implications are summarized. These apply only to the one medium density softwood (hoop pine) investigated and no indication is yet available as to the generality of their application.

It has been shown that it is possible to cut thin sections by microtome without materially changing the length in the grain direction, although this is achieved only at the expense of transverse distortion. The longitudinal shrinkage of sections greater than 80 μm thick differs little from that of sawn specimens 1 mm thick and the first cycle of shrinking and swelling is reproducible.

Either microtomed sections or sawn specimens may be preferred under given circumstances for the measurement of longitudinal shrinkages. Advantages arising from the use of thin sections are:

1. Equilibrium is reached more rapidly.
2. Moisture content gradients should be lessened.
3. Variations in longitudinal shrinkage over short distances, e.g. through a narrow growth ring, can be studied.
4. Matched samples may be obtained readily for comparing the effects of controlled treatments (e.g. chemical treatments).
5. Such treatments can be readily applied.

There are also disadvantages attached to the use of thin sections.

1. The transverse distortions preclude simultaneous measurement of transverse shrinkage.
2. Moisture content cannot readily be measured on the shrinkage test specimen.
3. Specimens must be conditioned in the same container in which they are measured.
4. Results may be more variable than those of thicker samples due to local variations in structure.

It seems likely that thin sections would be of maximum value in comparative studies of experimental factors affecting longitudinal shrinkage. Where large numbers of measurements are necessary, e.g. in sampling trees of many species, sawn specimens are likely to be preferred.

Some preliminary results from investigations of the type made possible by the use of thin sections have been obtained.

These include the study of

1. the influence of fine structure (revealed in the present instance by varying the section thickness),
2. the effect of chemical treatment, e.g. delignification, and
3. shrinkage-moisture content hysteresis.

There is a strong suggestion from each of these experiments that longitudinal shrinkage is markedly affected by stresses developed within the cell wall. The possible implications of these stresses for wood shrinkage will be considered in a hypothetical model of a shrinking wood fibre in Part II.

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