NOTES AND CORRESPONDENCE

This section of the journal is available for wood scientists and technologists to publish technical notes and accounts of work in progress, as well as to discuss matters of education and professional development. In general, such material should be limited to no more than six double-spaced typewritten pages. Where applicable, contributions should appear in the approved style for technical articles in *Wood and Fiber*.

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No discussion will be published unless the author of the original article has been provided with a copy of the discussion and has had an opportunity to submit a reply to the Editor, to be published with the discussion. Discussions and replies should not exceed the length suggested above. Where pertinent, references to the literature should be included.

It is anticipated that discussion and replies in *Wood and Fiber* can add, for both authors and readers, a dimension not normally found in technical journals.

Scanning Electron Microscopy of Mature Douglas-Fir Earlywood Intertracheid Pitting

ABSTRACT

Scanning electron micrographs of mature earlywood intertracheid pitting of Douglas-fir, *Pseudotsuga menzeisii* (Mirb.) Franco, demonstrate the advantages and limitations of the scanning electron microscope. The micrographs portray the variation encountered in pit membrane structure. The micrographs are interpreted to support Harada and Côté's (1967) proposed organization of the pit border and, at least in some instances, the aggregation of primary wall fibrils to form large margo strands.

INTRODUCTION

The scanning electron microscope is a comparatively new instrument, quite different in principle and application from the conventional transmission electron microscope, and has been commercially available for only the past few years. It is now finding a rapidly expanding use in the diverse fields of biology, geology, metallurgy, plastics, paper, and the general area of surface studies.[1]

Scanning electron microscopy has been well received in the area of wood technology. The ease of specimen preparation and attendant minimizing of artifacts makes scanning microscopy especially suitable for studying wood ultrastructure in those instances where the equipment's resolution capability is adequate.

Resolution of an image with the scanning electron microscope is limited to the size of the area emitting electrons at any moment. When the electron probe hits the specimen, scattering causes the probe to spread so that

[1] For a detailed discussion of the scanning electron microscope and its application, the reader is referred to a text by P. R. Thornton, "Scanning Electron Microscopy," Chapman and Hall Ltd., London, 1968, and to "Scanning Electron Microscopy/1968" and "Scanning Electron Microscopy/1969," respectively, the proceedings of the first and second annual symposia on the scanning electron microscope held in Chicago under the auspices of IIT Research Institute.

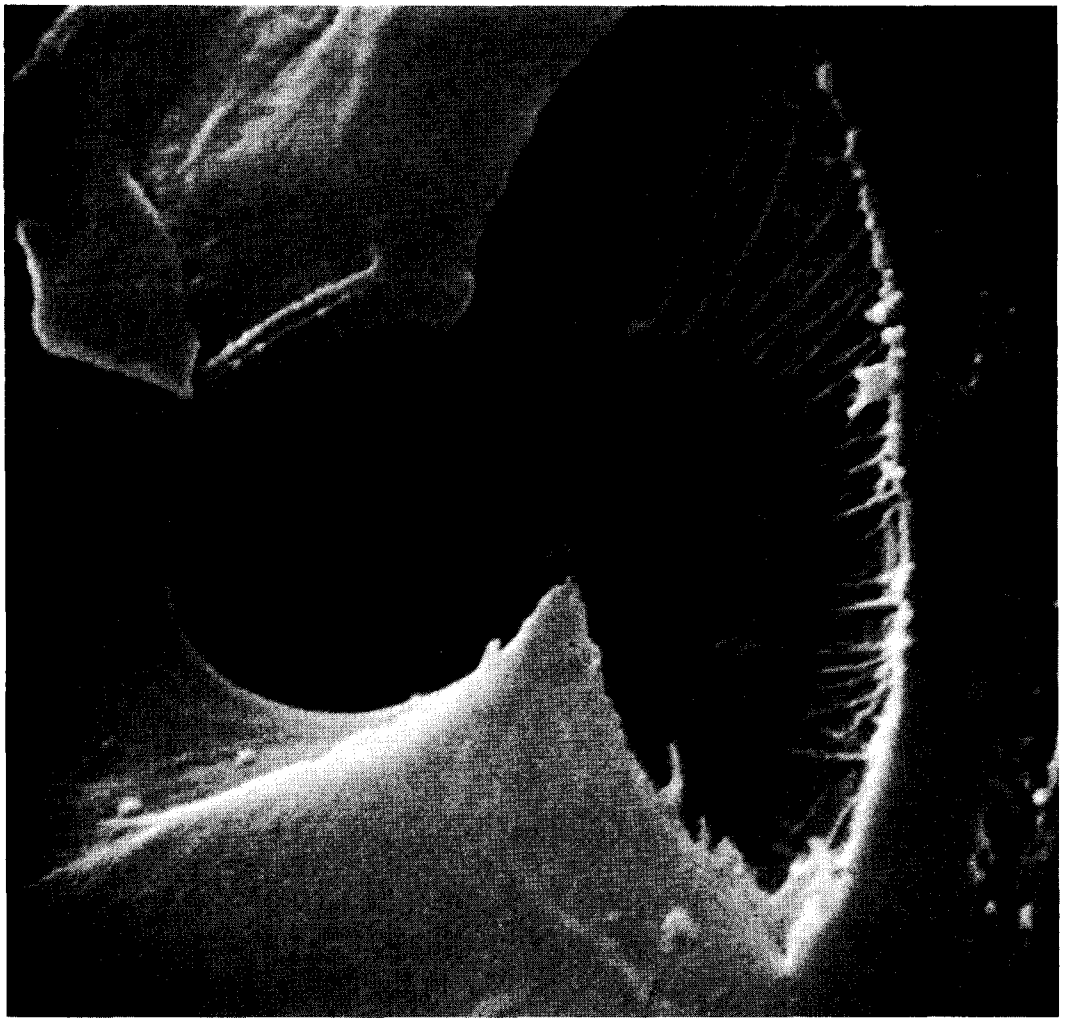

FIG. 1. A tangential oblique view of a bordered pit aspirated away from the viewer. 13,400×

the final volume of electron capture is roughly teardrop-shaped. The estimated escape depth of secondary electrons lies between 100 and 500 Å and depends on the acceleration of the electrons of the probe. Therefore the practical resolution of the instrument lies between 300 and 500 Å.

Depth of field is another advantage that is especially applicable to studies of wood. The long working distance (usually 10–25 mm) and small aperture of the final lens produce a highly parallel, uniform, and "in focus" probe over a considerable distance, and any intersection of the probe with a surface within that distance produces an image that is in focus. Depth of field varies inversely with magnification, varying from greater than 1000μ to approximately 10μ at magnifications of 100 to 10,000 times, respectively.

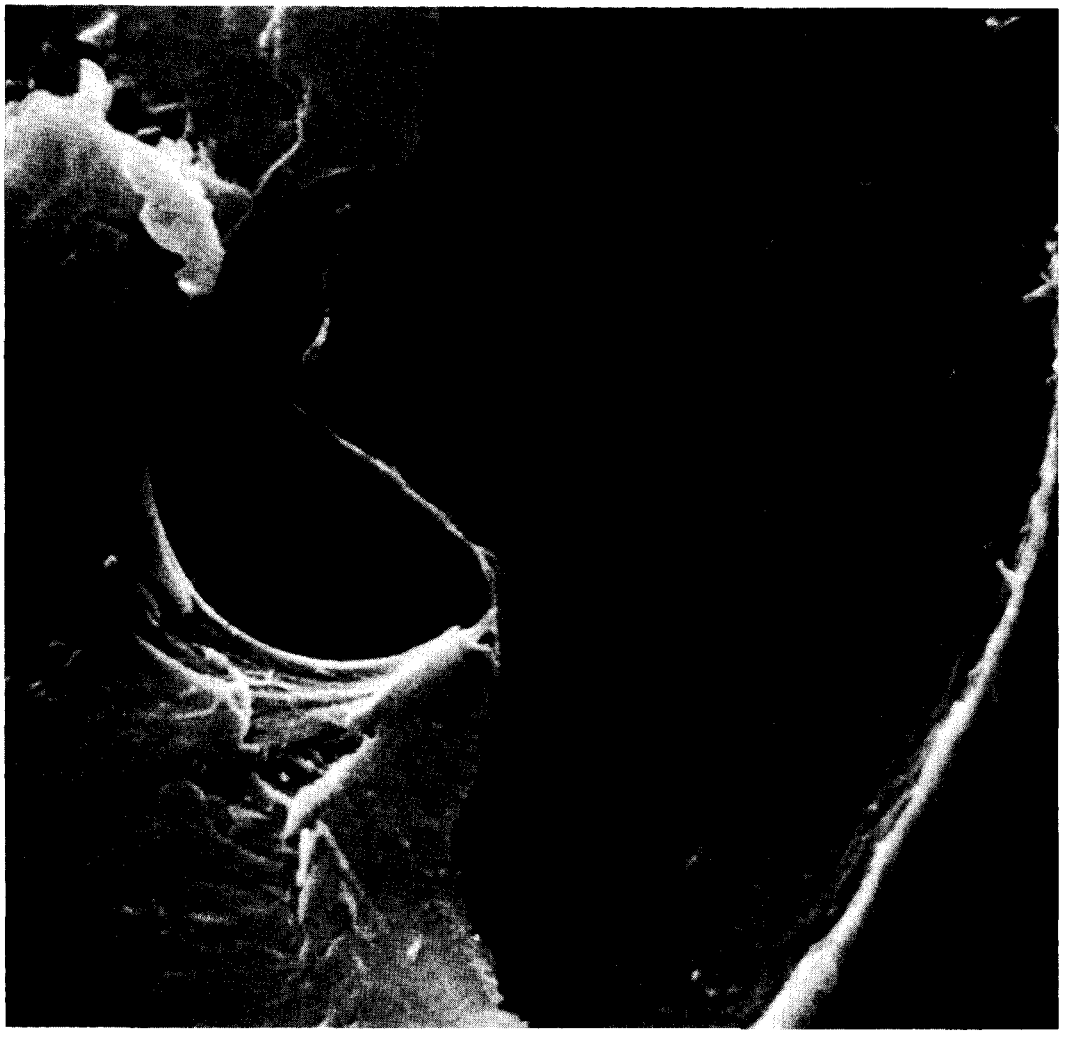

FIG. 2. A tangential oblique view of a bordered pit aspirated towards the viewer. The pit chamber shows a light warty deposit and fibrillar orientation in the border at the aperture is shown. 13,600×

Incidental to our use of the equipment in studies of chemical applications to wood, we have obtained many interesting photographs of wood surfaces. Included here are micrographs that collectively provide an understanding of spatial configuration of intertracheid pitting in earlywood of mature Douglas-fir, *Pseudotsuga menzeisii* (Mirb.) Franco. All micrographs were made from commercial, rotary cut veneer. Surfaces were freehand razor blade sections subsequently coated with a layer of gold or gold-palladium approximately 400 Å thick.

RESULTS

Figures 1 to 5 provide oblique tangential views of earlywood intertracheid pitting. Figures 1 and 2 demonstrate a change in

FIG. 3. A tangential view of a bordered pit aspirated to the right. 12,399×

direction of aspiration as found in contiguous pits. Variation of thickness of tori is shown in Figs. 2, 4, and 5.

Figure 6 is a slightly oblique radial view with a segment of one border removed. The torus is partially suspended above the level of the lower border. Some aggregation of margo strands is indicated with termination of the strands at the pit rim.

Figures 7, 8, and 9 demonstrate membrane structure in the second tracheid produced in the year's growth. All three figures clearly indicate suspension of tori below the plane of their respective pit rims. Here we see a very open margo structure with individual margo strands formed from a multiplicity of finer strands. Point of contact of margo strands with the pit border coincides

FIG. 4. A tangential view of a bordered pit aspirated to the left. The "rolled" characteristic of the aperture lip is shown. The torus was buckled by cutting action. 12,800×

with the point of aggregation of the finer strands, and these finer strands terminate at the pit rim or annulus. Integrity of the contact between the fine strands and the pit border cannot be elucidated because of limits of resolution.

Figure 10 further portrays border and membrane structure. The left side of Fig. 10 shows the S_1 layer of the secondary wall as it curved away from the viewer and covered the lumen side of the "lower" initial pit border, while the right side of the micrograph shows the S_3 layer of the companion tracheid. The torus is aspirated, and margo strands are lying against the pit border. The margo is very open with little or no aggre-

FIG. 5. A tangential view of a bordered pit aspirated to the left. The torus is very thin as compared to previous views. 12,100×

gation. The concentric orientation of fibrils on the chamber side of the pit border is obvious wherever the surface is not covered by the torus. Again margo strands terminate at the annulus. Figure 11 shows the lamellar structure of the secondary wall forming the border. Variation in fibrillar orientation is very evident.

Figure 12 shows variation in margo structure, and is especially interesting in that it shows margo strands attached at two different levels in the region of the pit rim.

DISCUSSION

These micrographs demonstrate some of the advantages and limitations of the scan-

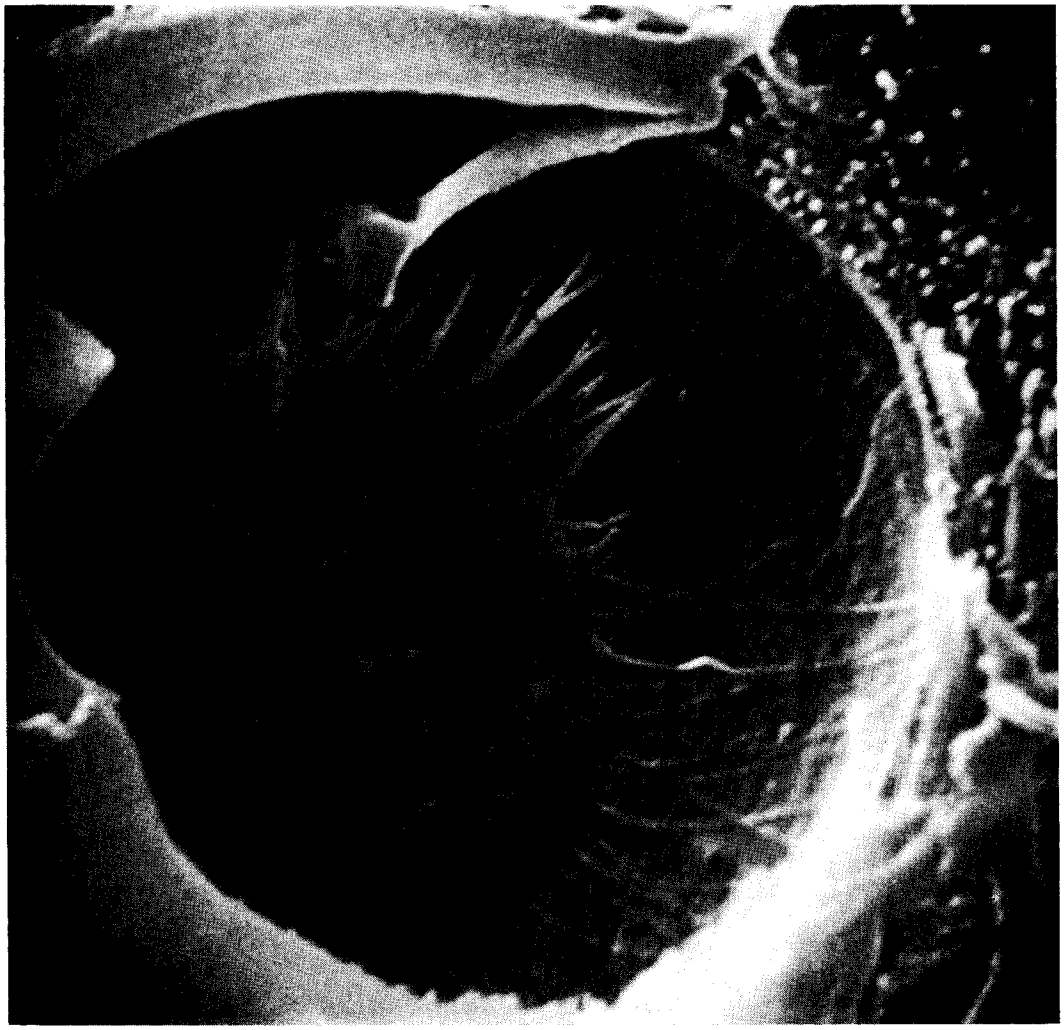

FIG. 6. An oblique radial view showing aggregation of margo strands and at least partial suspension of the torus above the pit border. Fine fibrils of the margo terminate at the pit annulus. 13,200×

ning electron microscope. The depth of focus attainable is an outstanding advantage and is clearly demonstrated. The very clear view of the membrane structure and suspension in Figs. 7, 8, and 9 is largely dependent on the depth of focus provided. On the other hand, the technique as used here did not provide definition of microfibrillar orientation through the thickness of the pit border. Better surface preparation should provide improved definition since resolution of fibrillar orientation, as demonstrated in Figs. 7, 10, and 11 is within the instrument's capability.

Various proposals on organization of the coniferous pit border have been recently reviewed by Murmanis and Sachs (1969) and Harada and Côté (1967). A point of

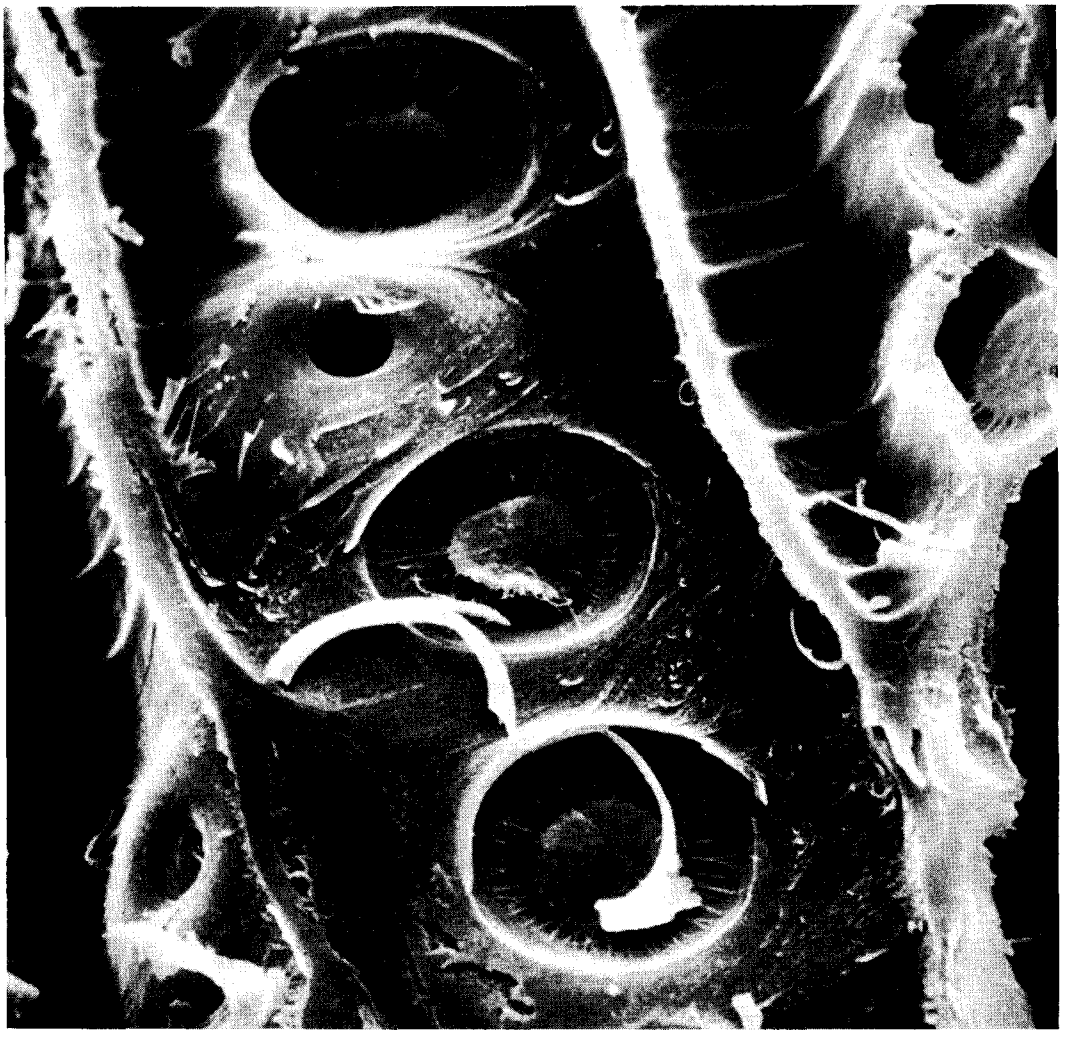

FIG. 7. A radial section near the end of the second earlywood tracheid. 2330×

difference involves the origin of the tissue lining the chamber side of the border. Figure 10 reaffirms the concentric fibrillar alignment of the chamber side of the border. Figure 4 suggests a rolled lip of tissue terminating at the edge of the aperture. Figure 11 shows a flap of tissue peeling away from the aperture, and the cleanness of separation suggests that these layers of tissue terminate at or near the aperture on the lumen side of the border. Figure 2 provides a similar suggestion where a bit of secondary wall has been torn from the lumen side of the border. These micrographs suggest that the exposed surface on the chamber side of the border is continuous from the annulus to aperture and that the layers of tissue on the lumen side of the

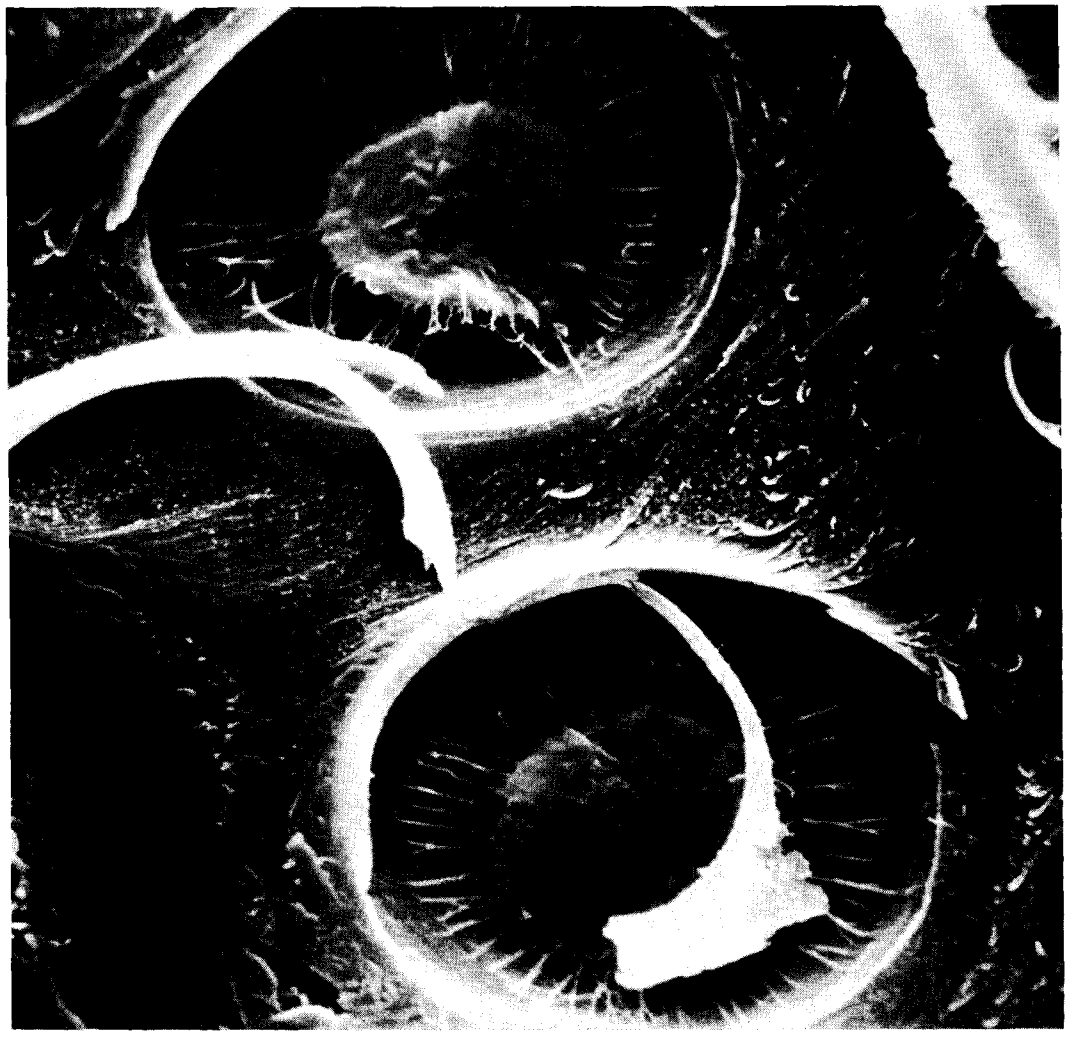

FIG. 8. An enlargement of Fig. 10. 4620×

border terminate at or near the aperture. With reference to initial pit border, this supports the organization proposed by Harada and Côté (1967) and Wardrop and Davies (1961).

A further point of difference among various proposals of border organization is the extent to which secondary S_1 covers the initial pit border on the lumen side. Figure 11 shows a layer of tissue with fibrillar alignment nearly at right angles to the tracheid axis and shows this layer to be essentially continuous to the pit aperture. The S_3 and S_2 layers in this micrograph are peeled partially from the border, suggesting the intact layer to be S_1. This supports the proposal of Harada and Côté (1967) as regards the extent of S_1 coverage of the initial pit border.

Several of the micrographs provide a

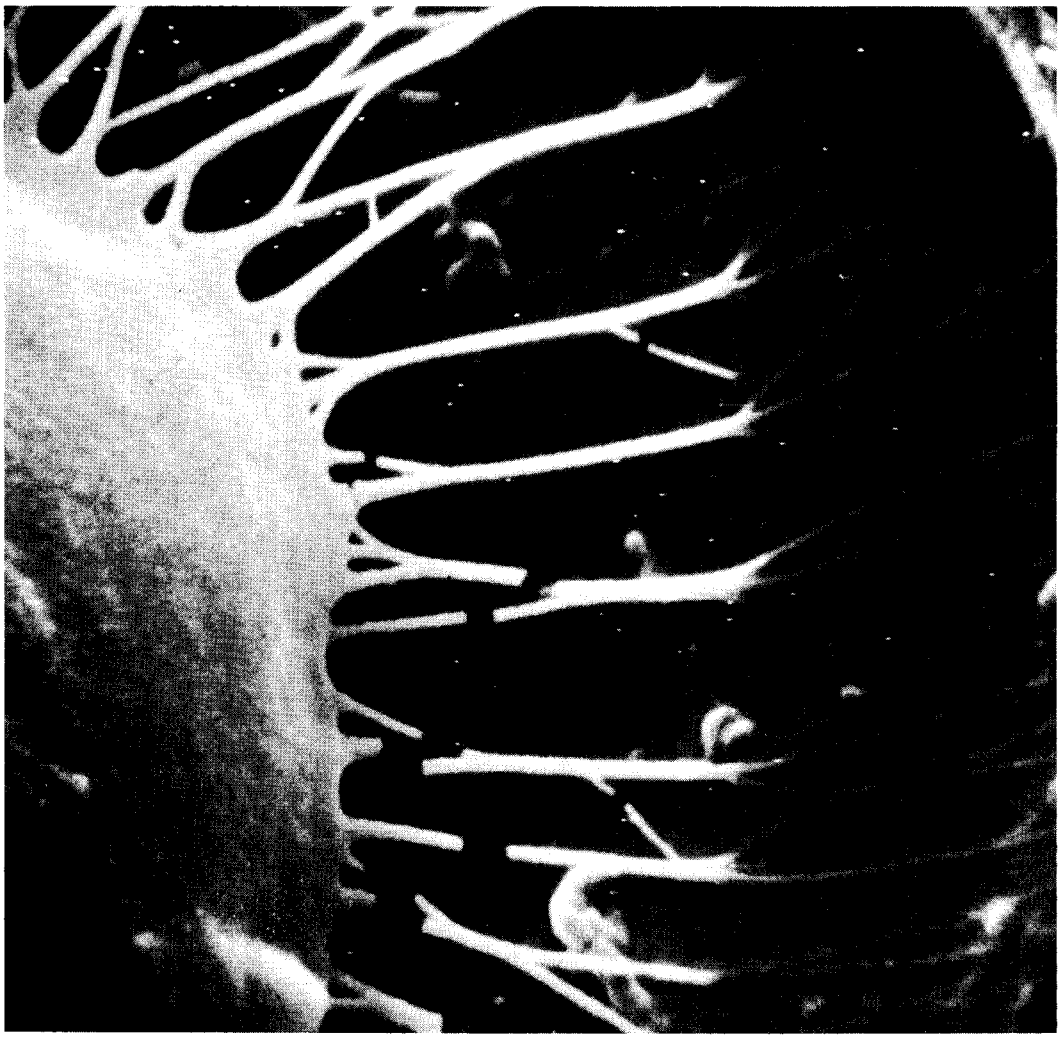

FIG. 9. A further enlargement of Fig. 10 showing the manner of aggregation of margo strands and termination of the fine strands in the pit rim or annulus. 23,300×

clear view of margo structure. Figures 7, 8, and 9 leave no doubt that, in this very early springwood tracheid, margo strands are aggregates of smaller strands. In this series of micrographs, tori are not aspirated although the tori are obviously displaced from the plane of the pit rim. The same stress that caused the displacement may have effected the aggregation evident in the margo strands. Figure 6 also shows aggregation in a later earlywood tracheid from a different specimen.

Figures 6, 9, 10, and 12 show margo strands terminating at the pit rim or annulus. Figure 12 shows at least two, and possibly three, levels of margo attachment,

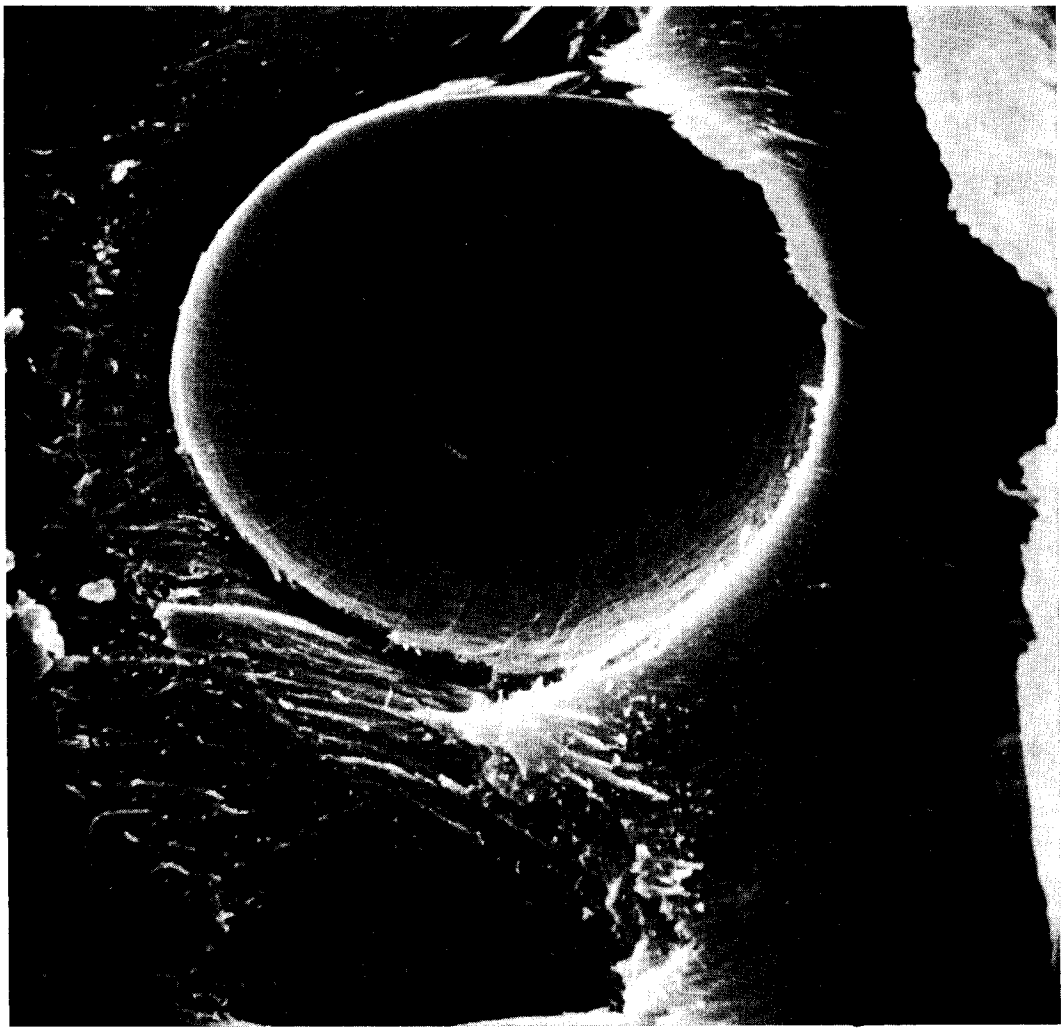

FIG. 10. A radial view of the pit chamber with aspiration away from the viewer. The section is slightly oblique, right to left, showing S_3 on the right and S_1 of the companion tracheid on the left. Margo strands are evident with all strands terminating at the annulus. 5500×

or alternatively—margo projection, in a low-angle view of the rim area. Study of Figure 12 suggests that the viewer is looking into the space between two levels of margo strands, that the margo strands have been encased by layers of border substance at the periphery of the original pit field, and that the large margo strands may have originated from the primary wall.

Figures 9, 10, and 12 show the same sort of margo variation that has been reported previously by Thomas (1968, 1969) for southern yellow pines. Until further study of differentiating pits is completed, no positive conclusions concerning mechanisms or correlations of margo formation are possible. However, the above interpretation of Fig. 12 and the very clear indication in

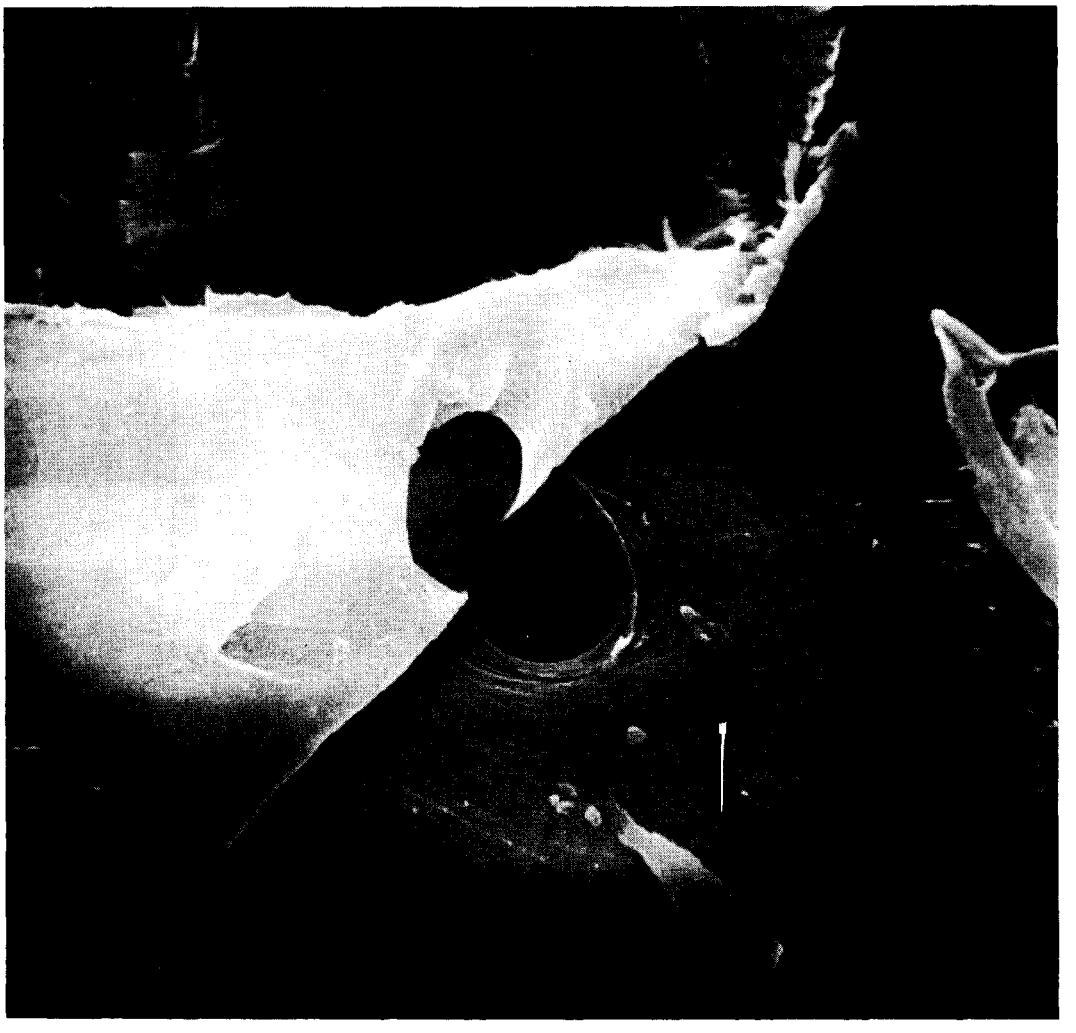

FIG. 11. A radial view showing varying layers of the secondary wall. S_3 and S_2 are partially peeled away showing the S_1 below. 5500×

Fig. 9 of aggregation of individual strands terminating at the annulus suggest that in some cases the large radiating strands of the margo of Douglas-fir may be of primary wall origin.

The scanning electron microscope provides a very fast route to the study of surfaces at magnifications and limits of resolution intermediate between the light and transmitting electron instruments. These factors, with the attendant depth of focus, provide a unique opportunity for further elucidation of the anatomical features of wood.

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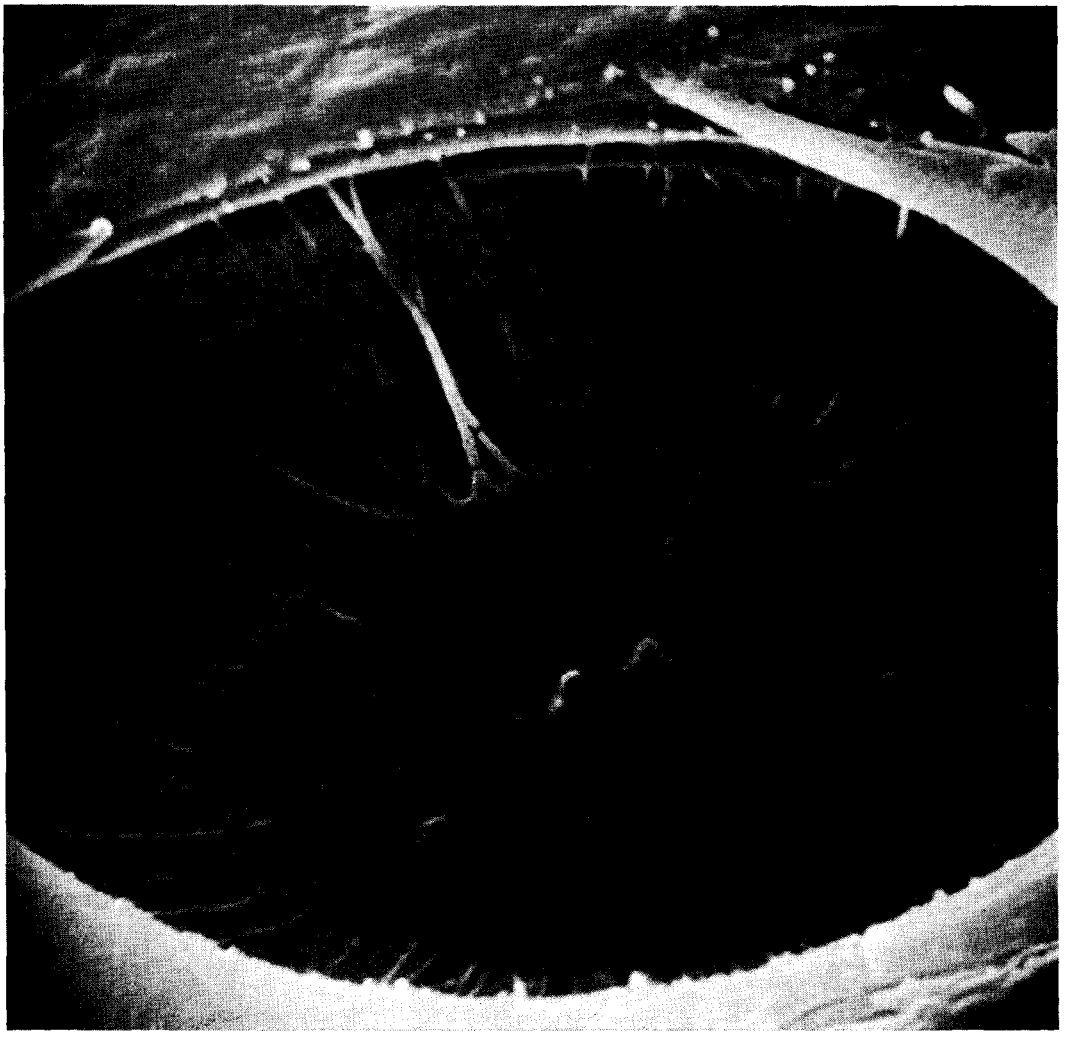

FIG. 12. A radial section viewed at a low angle and showing margo strands attached at two different rim levels. 13,400×

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Surface Tension of the Sap of Several Species of Wood

INTRODUCTION

When water is evaporated from swollen hygroscopic solids, such as wood or various wood products, its removal causes a drawing together of the structure because of surface tension forces and shrinkage results (Campbell 1933). When water in wood is replaced with alcohol, the latter by hexane or benzene, and the final liquid is flash evaporated, the resulting shrinkage is greatly reduced (Stamm 1964). This is because those liquids have surface tensions of one-quarter to one-third that of water.

When dry or partly dry wood is impregnated with liquids under pressure, trapped air has to be displaced or compressed. It is necessary to apply a pressure great enough to overcome the force with which films of the condensed liquid are held in the finest communicating void structure (the pit membrane pores) to allow liquid to displace the air. This too is a function of the surface tension of the liquid.

If these surface tension forces were not operative, the free water in wood could be readily removed by applying air pressure to one end or face. Recent measurements (Stamm et al. 1968) indicate that air pressures as high as 400 psi would have to be applied to the green heartwood of resistant species in the fiber direction to displace the free water and allow air to pass through the easiest path when the length of the specimen is only 3 inches. Similar pressures are required to displace water from the easiest path through 1/16-inch-thick veneer. For thicker material, the required pressure would be prohibitively high. Although blowing liquids from wood with a gas has no conceivable commercial value as a means of drying wood, it serves as a simple means of determining opening sizes in wood, when the surface tension of the liquid is known (Stamm 1966; Stamm et al. 1968). When measurements are made on naturally green wood, it is necessary to know the surface tension of the original sap or equilibrium solution. This may vary considerably from the high values of 70 to 72 dynes/cm for pure water at room temperature. No data for the surface tension of the sap of various species of wood were located in the literature. It thus appeared desirable to obtain and record a few surface-tension values for sap largely to show the appreciable variations from the values for pure water.

SURFACE TENSION MEASUREMENTS

The surface tension measurements were made at room temperature (22–24 C), using the ring tensiometer method, and correcting for the weight of the liquid lifted, according to the method of Zuidemn and Waters (1941).

The sap was obtained by pressing 1.5-inch cubes of green, never-dried wood specimens in a small hydraulic press. The specimens were placed in a 5-inch-square by 0.5-inch-deep galvanized iron tray within the press. The applied pressure was slowly raised over a period of about 10 min to a pressure of approximately 15,000 psi. Enough cubes were pressed to exude about 50 ml of liquid into the tray from which the liquid was pipetted while the specimens were still under pressure.

Three sets of surface-tension measurements were made on the original sap at different times and averaged. In no case did the standard deviation vary by more than one dyne/cm.

Twenty-five ml of each sap sample was evaporated to dryness and constant weight at 45 C by blowing dry air at that temperature over the surface of the samples in 50-mm weighing bottles to obtain the dry weight.

Table 1 gives the data for different zones in redwood and for the sapwood of ponderosa pine, Douglas-fir, and tanoak. Solid contents of the sap ranged from the small values of 0.08 g/100 ml for the Douglas-fir sapwood to 4.33 g/100 ml for the top heartwood of redwood. Surface tensions of the undiluted sap ranged from 57.3 dynes/cm for the Douglas-fir sapwood to 45.5 dynes/

TABLE 1. *Concentration of nonvolatile water-soluble extractives in the sap of several different never-dried green woods isolated by compression of the wood and the surface tension of the sap before and after dilution to one-quarter of the original concentration*

Species	Part of wood	Concentration	Surface tension	
			full conc.	¼ conc.
		g/100 ml	dynes/cm	dynes/cm
Redwood	Sapwood butt sinker log	0.11	56.9	61.8
	Sapwood butt sinker log	0.17	50.0	56.8
	Heartwood butt sinker log	3.99	45.6	51.3
	Heartwood butt sinker log	3.92	45.5	51.2
	Heartwood top log (80 ft up)	4.33	49.8	52.8
Ponderosa Pine	Sapwood	0.27	50.8	53.3
Douglas-fir	Sapwood	0.083	57.3	60.5
Tanoak	Sapwood	1.34	47.5	52.5

cm for the heartwood from the butt log of the redwood. Values for the sap diluted to one-fourth concentration were increased only slightly, compared to what they would be increased if the surface tensions were additive. For example, the surface tension of the distilled water used for dilution was 70.1 dynes/cm. Three times this value plus an undiluted sap value divided by four gives additive values of 66.9 dynes/cm for the sapwood of Douglas-fir and 64.0 dynes/cm for heartwood of the butt log of the redwood. These values are significantly greater than the measured values as would be expected if the sap constituents act as wetting agents.

The limited data given for the few species in no way indicate the surface tensions of the sap of these species. They merely show that the sap of different species may have considerably lower surface-tension values than that of water. The surface-tension values for sapwood appear to be higher than those for the heartwood, as a result of the lower extractive content.

CONCLUSIONS

The sap of several species of wood was shown to have surface tensions considerably less than that of distilled water as a result of the wetting agent action of the extractives. The sap from heartwood, as a result of its higher solute content, tends to reduce the surface tension of water more than the sap from the sapwood.

Calculations should never be made in which the surface tension of the sap of wood is assumed to be that of distilled water. Measured equilibrium surface tensions of the sap should always be used in calculations of pore radii or shrinkage effects in wood (Stamm et al. 1968).

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The Preparation of Microsections by Sawing

The technique used to prepare thin specimens for experimental investigations markedly affects the results obtained. The usual technique for the preparation of specimens up to 200 mμ is by microtoming; but even so, when the specimen thickness exceeds 100 mμ, particularly with some species, only a highly skilled technician can produce

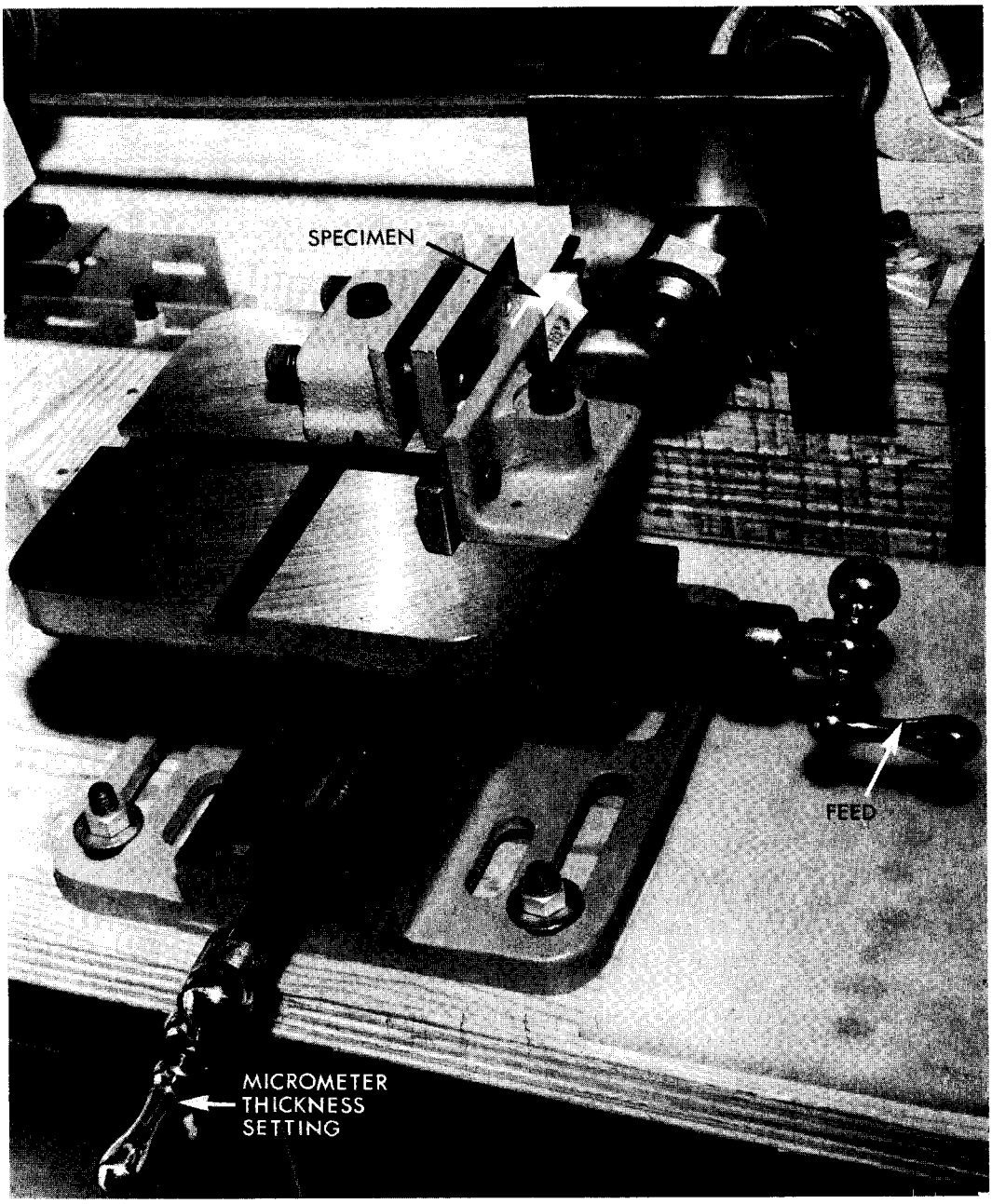

Fig. 1. Authors' table saw, showing arrangements of components for precision sawing.

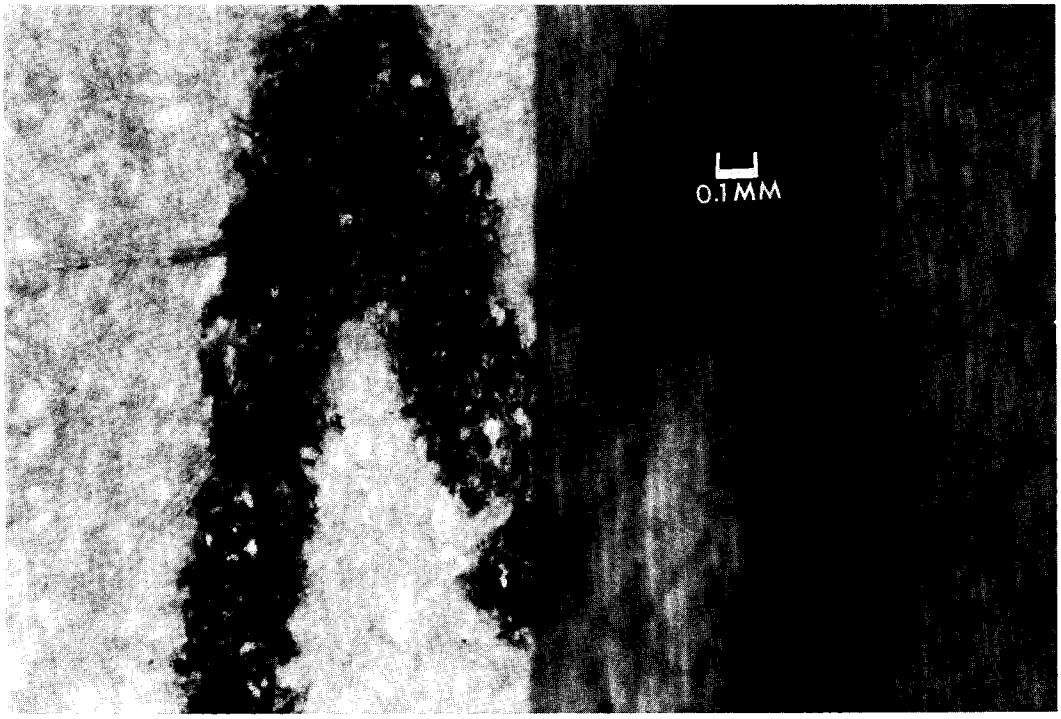

FIG. 2. Microsection (right) over typewritten *M* as viewed by transmitted light.

usable specimens. The experimental results obtained are even then subject to doubt. For example, Biblis (1969) in tensile stress determinations of loblolly pine found that microtomed sections approximately 100 mμ thick gave half the values obtained from similar wood specimens 3/16 inch thick. The specific reason for the different values was not determined, since the microtomed sections were cut while wet and were subject to some flexure and possible damage in cutting. The sawn specimens were cut dry and were not subject to flexing. Recognized techniques do not permit the microtoming of specimens significantly thicker than 100 mμ, nor the sawing of specimens significantly thinner than 2 mm with any degree of precision. This means, in effect, that no recognized method exists for preparing precision specimens in the range from 100 mμ to about 2 mm.

Similar problems of microsample preparation are experienced in investigations of wood permeability, because damage to the specimen has a profound effect on the results. This led the authors to design and build a precision table saw for the preparation of specimens in this thickness range.

Figure 1 shows the arrangement of the components for precision sawing. The essential features are a 4.5-inch-diameter saw, 0.050 inch thick, mounted by means of a 1.5-inch precision collar on a 1-inch-diameter shaft. The shaft is mounted in antislop ball bearings. The saw is driven at a rim speed of 6000 ft/min by means of a ¾-hp, 3450-rpm motor. The saw is a standard 34-tooth metal-slitting saw. It was reground so that the teeth now have a 15° hook and are alternately bevelled at 15°. There is no swage or set, but the teeth and plate taper from 0.05-inch thickness at the rim to 0.04-inch thickness at a distance 1 inch inward from the rim. The cost of the saw and grinding was approximately $15. A specimen to be sectioned is clamped in a vise

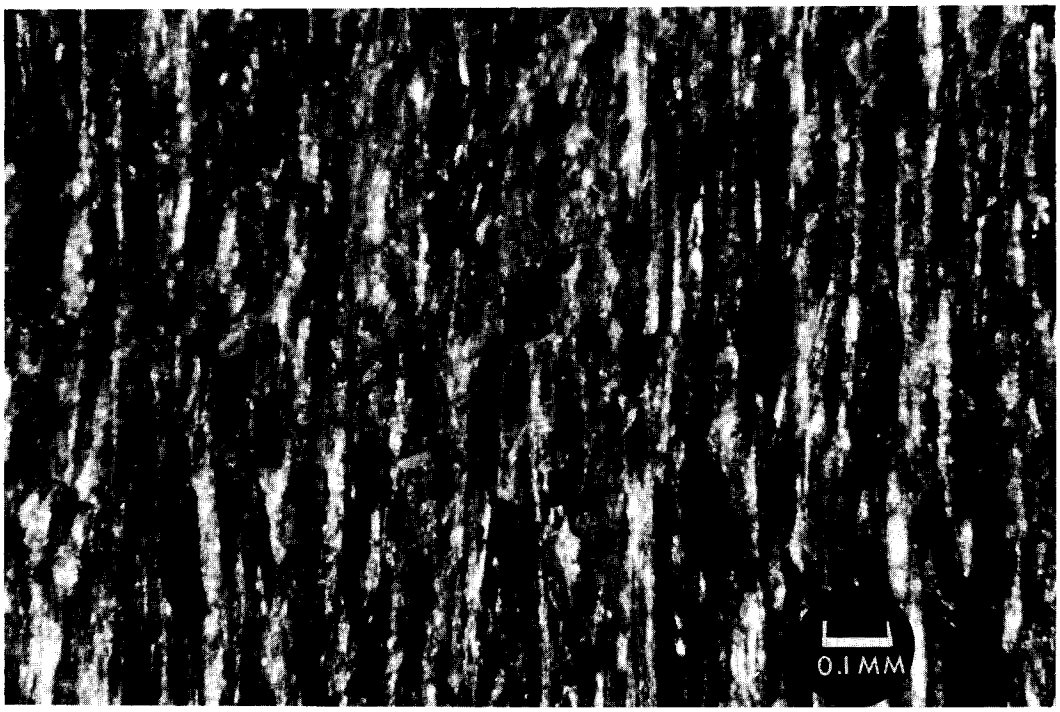

FIG. 3. Woolly nature of surface, still permitting observation of some wood elements.

mounted on a plate that is operated in feed and cross-feed by micrometer drives similar to those used on precision lathes.

With normal care, it is possible to cut smooth sections of uniform thickness as thin as 100 mμ (0.004 inch) in the radial and tangential directions, and as thin as 900 mμ (0.035 inch) in the transverse direction. The thicknesses were exceptionally uniform, the variations across the full width of the 12.5-mm (0.5 inch) square specimens being less than 10 mμ (0.0004 inch).

The disadvantages of this preparation technique are the large loss in saw kerf (i.e., specimen), which measures about 1.25 mm (0.05 inch) per cut, and the slightly woolly but very uniform appearance of the specimens as viewed under a microscope. The advantages are the possible range and uniformity of specimen thickness, and the assurance that the specimens have not been damaged or altered by compression, bending, wetting, or other undesirable treatment.[1]

Figure 2, which is a photomicrograph of a typewritten letter *M* partly overlaid by a sawn microsection, gives an indication of the thinness of the sections; whereas Figure 3 is a photomicrograph of the wood surface, demonstrating the roughness of cut obtained by this method.

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[1] The authors wish to acknowledge the work of D. Kusec in the design and construction of the unit.