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The Effects of Elevated Temperature on Certain Wood Cells

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Summary

From the microscopic evidence, the modifications observed certainly indicate that the structures viewed are temperature-sensitive, although the critical temperature is still to be determined. This sensitivity may be linked only to a change from a solid to a liquid state, and visual indications support such a tenet. This does not perclude, however, the possibility of thermo-chemical changes occurring at the elevated temperatures used.

It is interesting to note that lignin has been found to soften under elevated temperature in the presence of moisture and pressure [GORING 1963], and it is very tempting to speculate along this avenue. The results thus far, however, indicate that much further work is needed before their significance can be fully established.

Zusammenfassung

Bei der mikroskopischen Untersuchung von Teilen des Holz-Feingefüges zeigten sich Veränderungen, die darauf hinweisen, daß die beobachteten Gefügeteile temperaturempfindlich sind. Die kritische Temperatur muß jedoch noch genauer festgestellt werden. Die Temperaturempfindlichkeit hängt wohl hauptsächlich mit dem Übergang vom festen zum flüssigen Aggregatzustand zusammen, eine Annahme, die durch visuell beobachtbare Vorgänge gestützt wird. Dies schließt jedoch nicht die Möglichkeit thermisch-chemischer Veränderungen aus, die bei den angewendeten höheren Temperaturen eintreten.

Aufschlußreich ist die Feststellung, daß Lignin bei erhöhter Temperatur, unter Feuchtigkeit und Druck erweicht. Überlegungen in dieser Richtung erscheinen erfolgversprechend. Die bisherigen Ergebnisse zeigen aber, daß weitere umfangreiche Untersuchungen erforderlich sind, bevor die Bedeutsamkeit der bislang beobachteten Vorgänge als erwiesen betrachtet werden kann.

Introduction

Despite the many advances in our knowledge of the results of thermal degradation and pyrolysis of wood, many questions relating to the effect of elevated temperature on wood remain unanswered.

The decomposition of wood by heat under ordinary conditions over a reasonable period of time does not become evident until the temperature reaches the neighbourhood of 275 °C. Long before this temperature is reached the water is driven off and as F. F. P. KOLLMANN and D. FENGEL [1965] and D. FENGEL [1966a,b] showed changes in the composition and structure of wood polysaccharides take place already at 150 °C and below. Information on possible effects of the chemical changes on the physical properties of wood is naturally important. A considerable

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amount of information has been recorded concerning the breakdown of wood substance as a result of temperature. Little has been done, however, in recording physical changes that occur at the submicroscopic level.

Electron microscopy has helped greatly in clarifying our knowledge of the cellular structure of the wood cells and the location of some of its chemical constituents. With improvement and new developments in the techniques of specimen preparation, better knowledge of cell structure should ensue.

Making use of such electron microscopy techniques, a preliminary investigation was undertaken to detect and record any fine -structure changes in the elements of xylem tissue subjected to high temperature treatment.

Materials and Methods

Samples of green *Fagus sylvatica* L. (European beech) were exposed to controlled heat in a thermogravimetric balance in the absence of air and heated for $2^1/_2$ hours at temperatures of 190°, 200°, 210°, 220°, or 240° C. Samples of *Picea abies* (L.) Karst. (European spruce) and *Fagus grandifolia* Ehrh. (American beech) were scorched with a bunsen burner flame and the cells just below the carbonized layer were studied. Internal temperature within the scorched specimens rose to approximately 280 °C within the block.

All heat-treated samples and non-heated controls were split, usually in the radial plane, and the exposed surface was then replicated with acrylic resin. The negative replica, after being stripped from the wood, was shadowed with carbon and then coated with platinum; then the resin was dissolved in acetone. All preparations were observed in Zeiss and RCA E.M.U. 3D electron microscopes.

Discussion

Like other plant materials, wood consists predominantely of tubular fiber units or cells bonded together in a matrix of lignose cemeting material. The walls of such structures are composed in the main of cellulose, lignin, and hemicellulose. The structure, shape, arrangement, and chemical makeup of the cells differ greatly and these differences are responsible for the various properties attributed to the different woods.

Variations in arrangement, size, and number of cellulose fibrils provide different tubate structural patterns within which the pits are formed (Figs. 1 and 2). The pits provide a common avenue from fiber to fiber for the passage of liquids and gases. Stretched within the pit chamber is a membrane built up in the main by the primary wall. Pits of the hardwoods lack the thickened central torus common to the bordered pits of the softwoods.

In many species of wood, the lumen side of vessels and fibers is covered by a warty layer which is adpressed to the tertiary wall. This membrane is frequently found lining the pit chamber and in half-bordered pit pairs. It is present, however, only on the side from the tracheidal elements [LIESE 1963].

After heat treatment at elevated temperatures, the intricate morphological detail of wood structure was scrutinized for any alteration of structure.

Cell Wall and Middle Lamella

Cracks and fissures were evident in the cell walls and middle lamella of both the heat-treated specimens (Fig. 3) and the controls (Fig. 4). Their presence must,

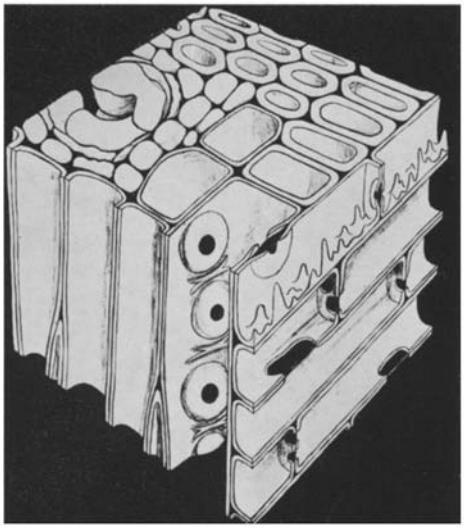

Fig. 1.

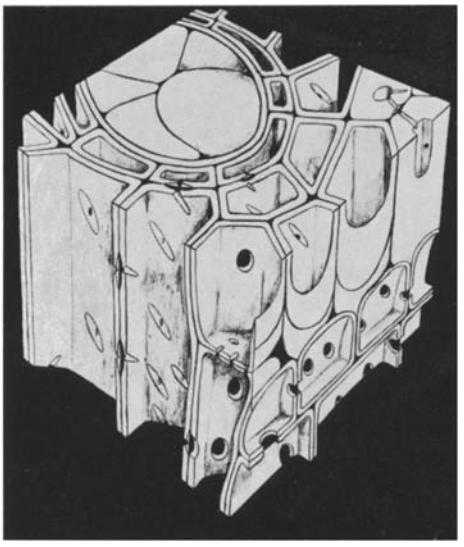

Fig. 2.

Fig. 1. Diagrammatic three-plane drawing illustrating the general arrangement of cell wall structure and pits peculiar to conifers. (Not drawn to scale).

Fig. 2. Diagrammatic three-plane drawing illustrating the general arrangement of cell wall structure and pits peculiar to angiosperms. (Not drawn to scale).

therefore, be attributed entirely to the preparation technique, since they show no relation to the topographic features of the original material. In a later figure (Fig. 6) definite evidence of heat effects are not accompanied by cracks.

Pits and Pit Membrane

During the last few years, evidence has accumulated indicating that no torus or torus-like structure is present on the membrane of fiber pits in angiosperms. Our study of pit structure in *Fagus sylvatica* and *Fagus grandifolia* confirmed the absence of such torus-like features in these species.

No change of the pits and membrane microfibrils could be detected in the fiber pits of the heat-treated hardwood used (Fig. 5). It was clearly obvious, however, that surface distortion of some of the vessel pits had occurred (Fig. 6). This effect could be produced by heated volatiles or gases of decomposition, but this premise requires future verification.

In gymnosperms, where tori occur, the margo or membrane coming off the torus fibers of normal untreated spruce (*Picea abies*) appear smooth and tenous (Fig. 7). We believe [SACHS 1963] that the torus is a cellulosic sac containing the incrusting constituents of the original primary pit field wall. When the cellulose was removed by hydrofluoric acid, a flap of lignin was left stretched across the pit

Fig. 3. Sample of fiber wall of *Fagus sylvatica* after heat treatment at 210 °C. Approximately 4400:1.

Fig. 4. Sample of fiber wall of *Fagus sylvatica* not heat treated. Presence of cracks here and in Figure 3 indicate that the cracks are due to preparatory techniques and are not induced by heat treatment. Approximately 2500:1.

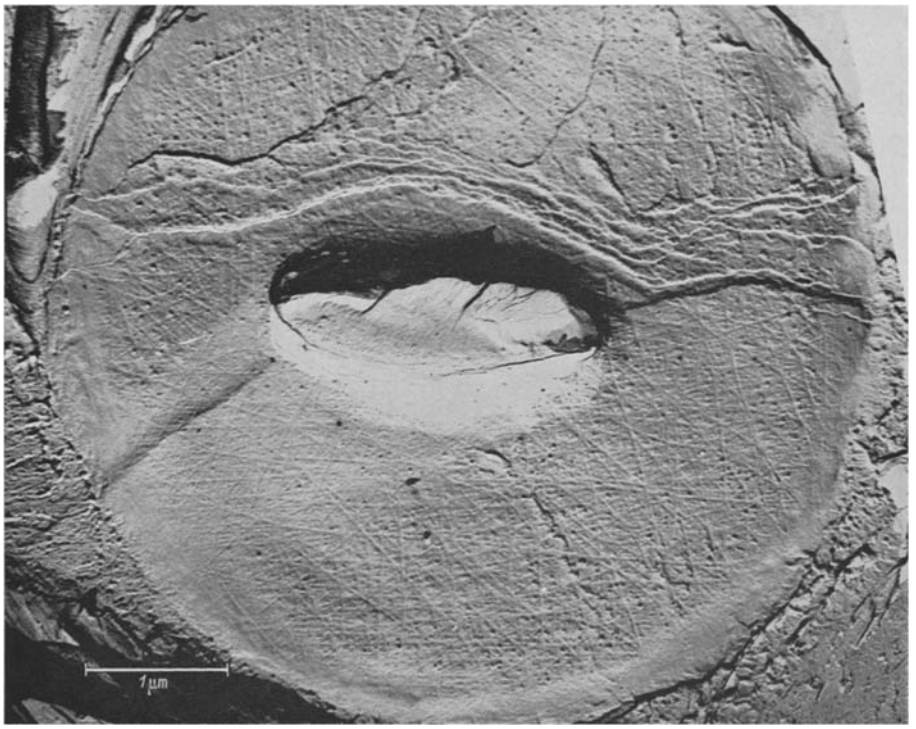

Fig. 5. Fiber pit microfibrils of *Fagus sylvatica* retained their normal appearance after heat treatment at 240 °C. Approximately 19 000:1.

aperture (Fig. 8). Subsequent heating at elevated temperatures appeared to cause some material to soften and flow along the fibrils of the margo. The membrane fibrils lost their smooth appearance and it is obvious that some of the material from the torus congealed on them (Figs. 9 and 10).

Warty Layer

The warts of the warty layer of *Fagus grandifolia*, as seen in the light microscope, appear as granulations (Fig. 11). In general they appear spherical in shape and randomly spaced. In the electron microscope they appear as elevations varying in size from 0.05 to 0.1 μm and irregularly spaced (Fig. 12). W. LIESE [1963] is of the opinion that the warts may be spherical particles—remnants of the dying protoplast—trapped between the tonoplast of DE VRIES and the plasma membrane. The tonoplast can be clearly seen in a profile view of the warts (Fig. 13). Masses of myelin-forming material have been reported [SCARTH 1942] which under certain conditions are associated with lining of plant vacuoles. Evidence of their composition is confirmed by staining with lipoid soluble dyes such as chrysoiden and by blackening with osmic acid. These phenomena indicate that the film-forming substance is rich in lipoids as compared with the protoplasm as a whole. In a study of the warts of *Actinostrobus pyramidalis* it was reported [WARDROP, LIESE, DAVIS 1959] that the warts gave staining reactions for lignin when stained with phloroglucin-hydrochloric acid and for protein when stained with potassium

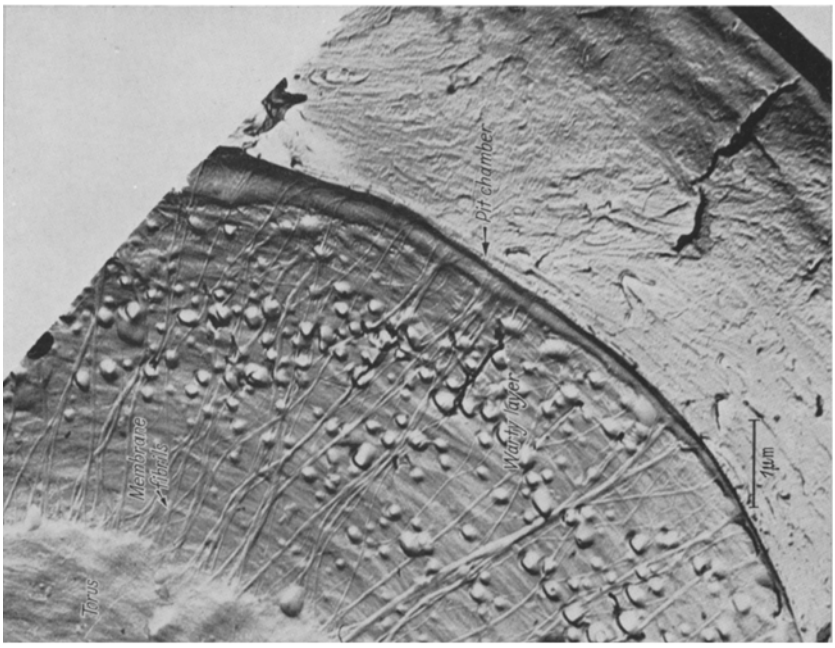

Fig. 7. Pit membrane fibrils of *Picea abies* of smooth and normal appearance. Approximately 11 500 : 1.

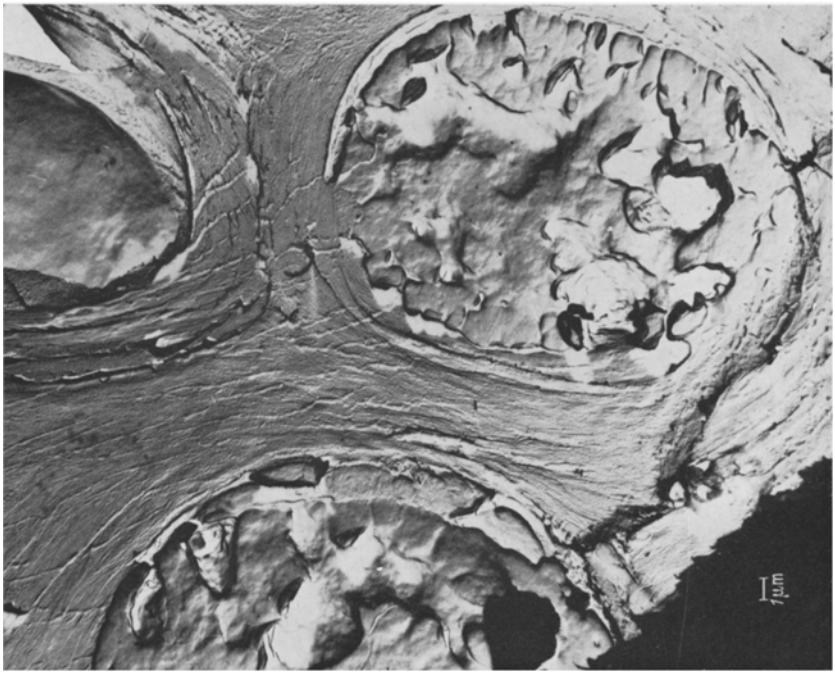

Fig. 6. Surface distortion of vessel pitting in *Fagus sylvatica* after treatment at 240 °C. Approximately 3 000 : 1.

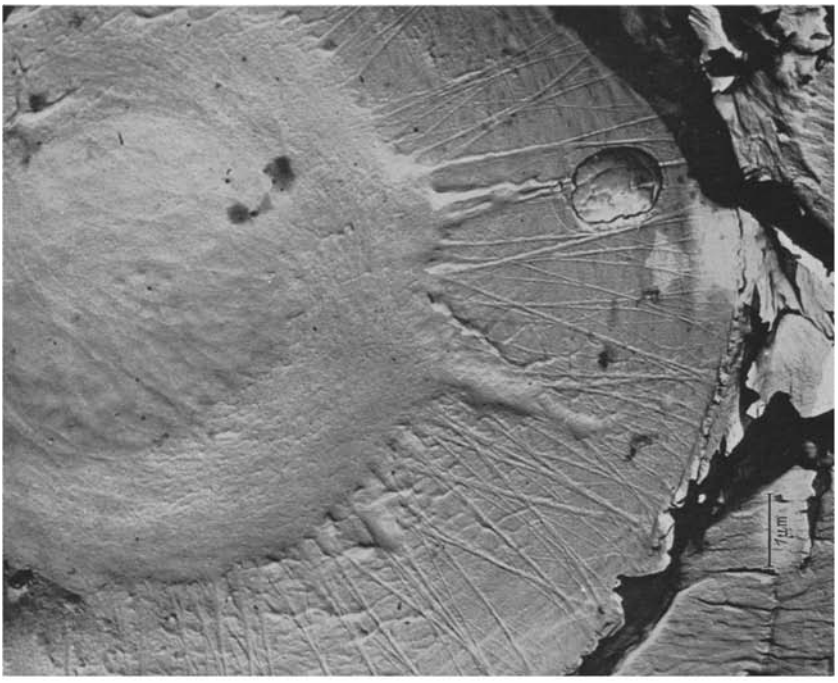

Fig. 9. Softening and flowing of material along the membrane of the bordered pit of *Picea abies* after heat treatment of 280 °C. Approximately 10000:1.

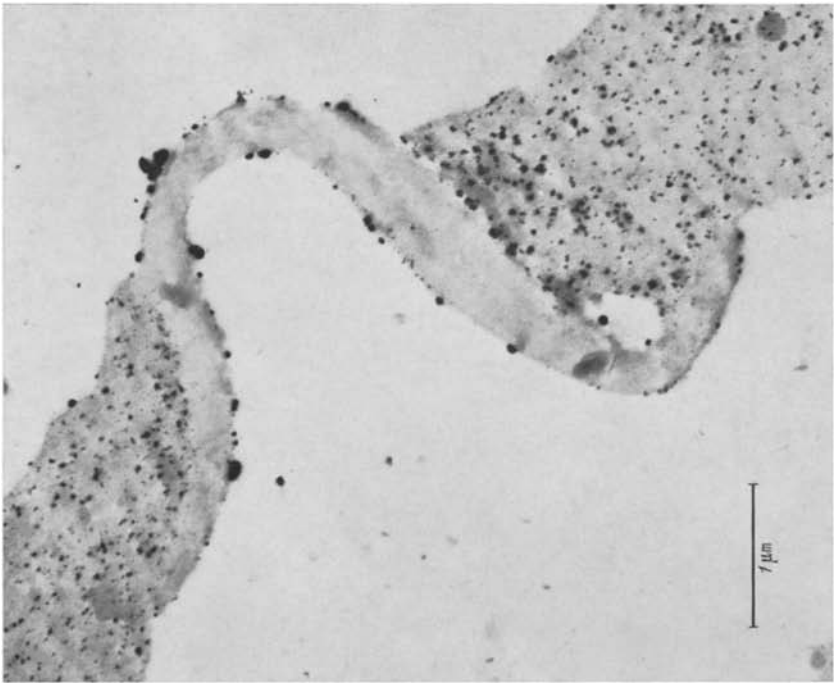

Fig. 8. Bordered pit torus of *Pinus taeda* after treatment with hydrofluoric acid leaving flap of lignin stretched across pit aperture. Approximately 19000:1.

Fig. 11. Warty layer as seen with the light microscope. *Fagus grandifolia*. Approximately 1200:1.

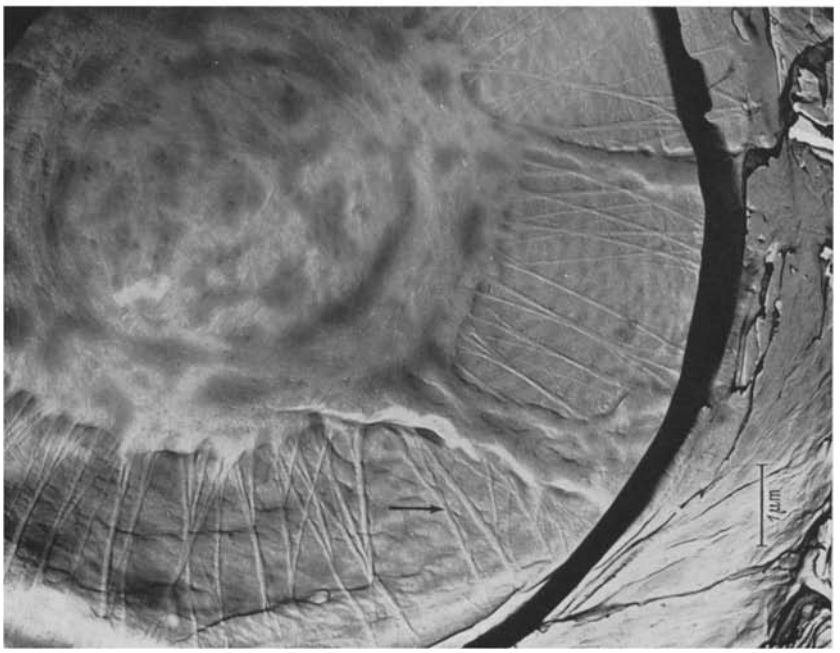

Fig. 10. Profuse flow of material from the torus of *Picea abies*. Congealing on the membrane fibrils shown at arrow. Approximately 11 000:1.

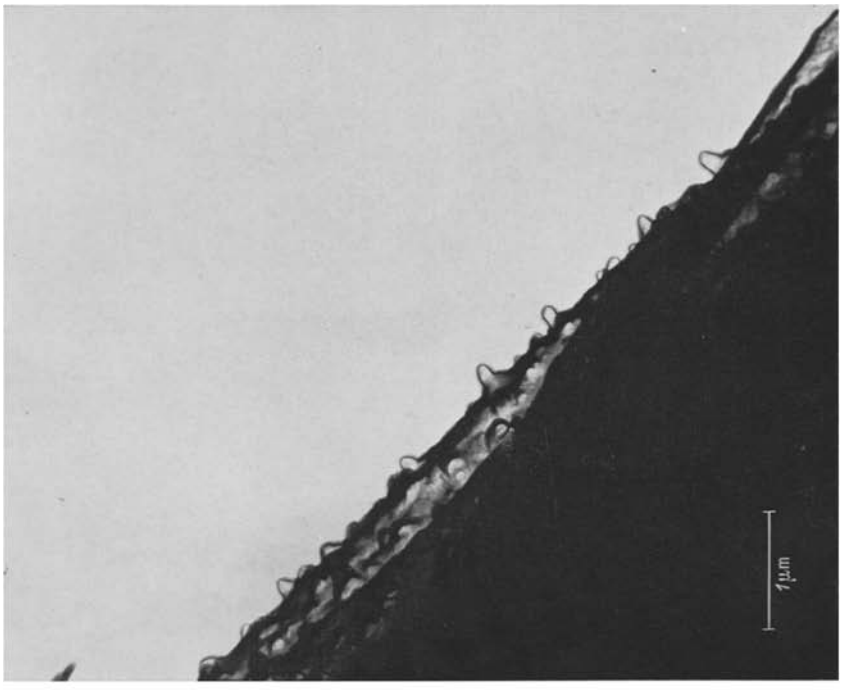

Fig. 13. Profile view of the warty layer of *Fagus grandifolia* showing the tonoplast overlying the warts. Approximately 16 000:1.

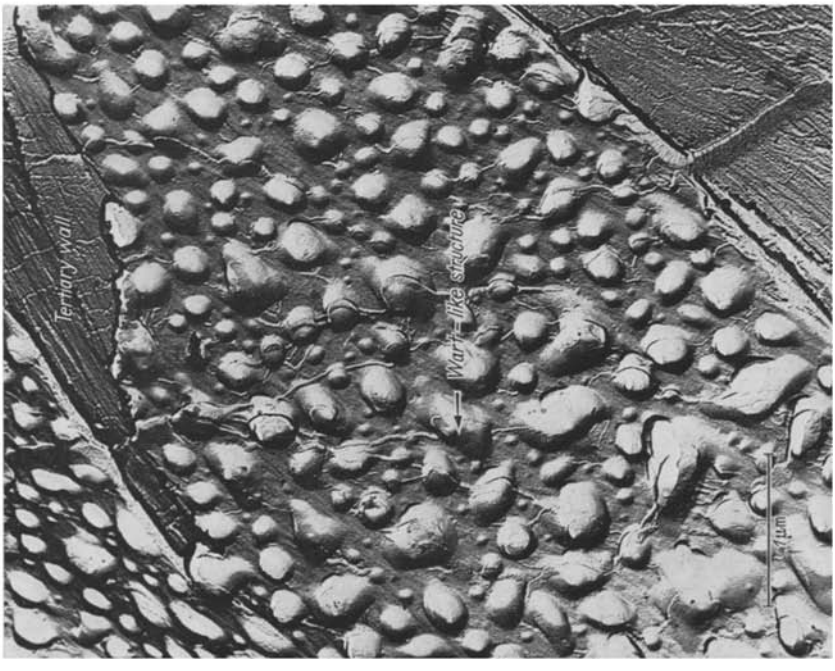

Fig. 12. Warty layer as seen with the electron microscope. *Fagus grandifolia*. Approximately 20 000:1.

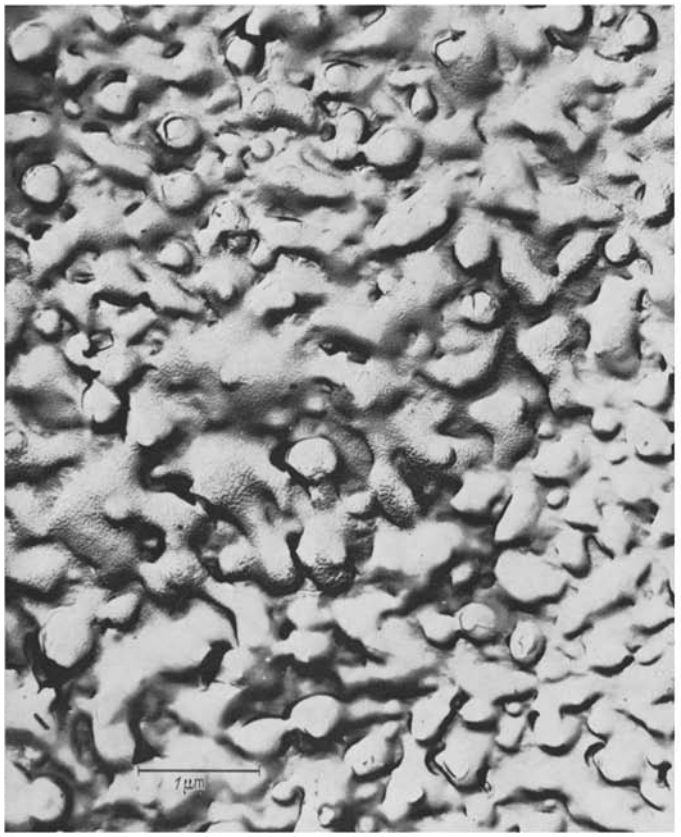

Fig. 14. Warty layer after heat treatment of 210 °C shows evidence of flow and drying. *Fagus sylvatica*. Approximately 16 000 : 1.

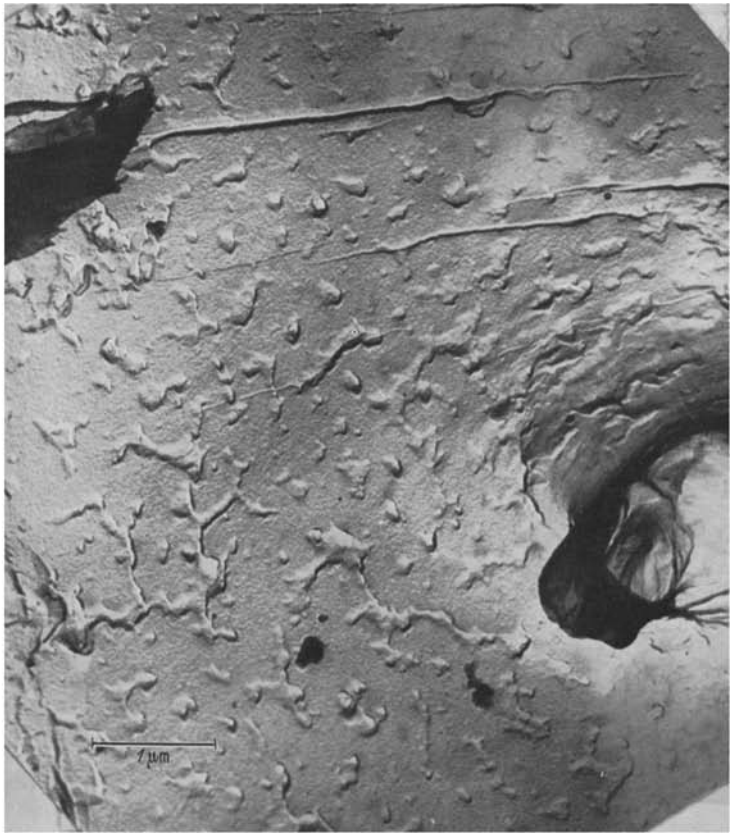

Fig. 15. Warty layer after heat treatment of 220 °C shows evidence of collapse and loss of mounded appearance. *Fagus sylvatica*. Approximately 16 500 : 1.

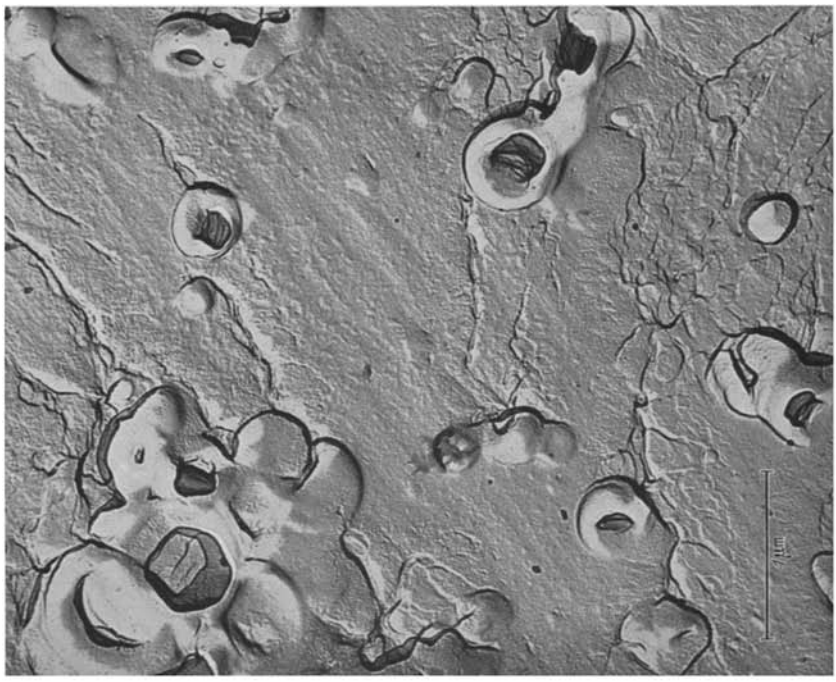

Fig. 17. Rupture of the tonoplast after heat treatment of 240 °C. The bursting is presumably due to the development of high internal pressures. *Fagus sylvatica*. Approximately 22 500 : 1.

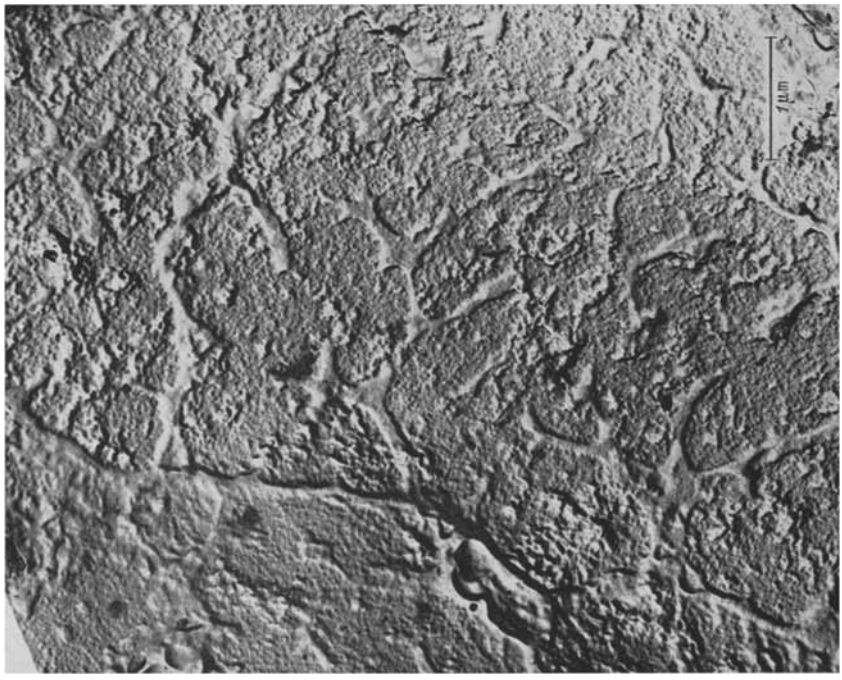

Fig. 16. Warty layer after heat treatment of 240 °C shows evidence of collapse. *Fagus sylvatica*. Approximately 16 200 : 1.

ferrocyaniferric chloride. Also, the vesicle reported in the wart-like structure was found to be osmiophilic. In the present study, the presence of a phenol ring compound in the warts of *Fagus grandifolia* was indicated by the light red stain obtained with an aqueous solution of potassium iodide plus 10% NH_3.

Warts have been dissolved rapidly with sodium hydroxide (5 minute boiling) followed by treatment with chlorine water and by boiling 4 hours in hydrogen peroxide and glacial acetic acid [SCARTH 1942]. In the present study, these treatments were effective also on the warts of *Fagus grandifolia*.

Samples of *Fagus sylvatica* treated at temperatures of 190 °C to 240 °C exhibit changes in the warty structure. In the treatment at 200 °C the warts changed shape and apparently flowed as well as showed evidence of drying (Fig. 14). In the treatment at 240 °C they lost their mounded appearance and seem somewhat collapsed (Figs. 15 and 16).

In some areas where presumable high internal pressures developed, bursting of the tonoplast layer resulted in the cavities (Fig. 17). There is no evidence of wart remnants in these cavities.

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