

STEAM OR HEAT FIXATION OF COMPRESSED WOOD

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ABSTRACT

Dimensional stability can be improved by either steaming or heating wood while the wood is in a compressed state. This study investigated the effect of steam or heat on fixation of compression set and the effect of these treatments on hardness, mechanical properties, and color of compressed and uncompressed wood specimens. To determine the effect of steaming before and after compression set, one group of wood specimens was steamed and then compressed, and another group was compressed and then steamed. Simple boiling and cyclic swelling tests were used to evaluate recovery of compression set. Hardness of compressed specimens was measured by the Brinell test. A two-point bending test on noncompressed specimens was used to calculate moduli of elasticity and rupture. A L-a-b color system was used to determine color changes. Compressed wood steamed for 1 min at 200 C or 8 min at 180 C showed no recovery of set, large increases in hardness, minimum decreases in mechanical properties, and slight darkening. We conclude that almost complete fixation of compression set in wood can be achieved by steaming compressed wood.

Keywords: Compressed wood, steaming, heating, permanent fixation, physical properties.

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INTRODUCTION

Researchers are searching for an effective method to stabilize the dimensions of wood during changes in moisture content. The work reported in this paper is a joint effort between

Kyoto University in Japan and the USDA Forest Service, Forest Products Laboratory, in the United States. Many methods have been studied, ranging from bulking the cell wall with simple aqueous sugar solutions to chemical reactions with the cell-wall polymers. Most methods use chemicals that bulk, react, cross link, or coexist within the wood cell wall.

Stamm et al. (1946) reported a method of dimensional stabilization of wood that was based not on the use of chemicals but on the use of heat alone. Their approach was based on work of Tiemann (1920), who showed that high-temperature kiln drying decreased the hygroscopicity and subsequent swelling and shrinking of lumber. Stamm and Hansen (1937) found that if dry wood was heated, hygroscopicity decreased substantially. However, if moist wood was heated, hygroscopicity did not decrease. They also found that heating wood in the presence of oxygen caused more degradation of strength properties than if wood was heated in the absence of oxygen.

Stamm et al. (1946) heated wood under molten metal and found that improvements in dimensional stability were accompanied by decreases in mechanical properties. Modulus of rupture (MOR) decreased 20% with an antishrink efficiency of 40%. When wood was heated in air, MOR decreased 25% with an antishrink efficiency of only 30%. These experiments were done between 160 C and 280 C. Stamm et al. called this product Staybwood.

Other researchers also worked to stabilize wood by a heating process. Hillis (1984) reviewed the literature in this area. Hillis and Rozsa (1978) found that a minimum heating of 2 h at 100 C plasticized the hemicellulose-lignin matrix to effect additional stability. To minimize thermal degradation of the wood, they suggested that the wood be presteamed or preheated for as short a period as possible. Skaar (1976) reported that heating wet wood degraded it 10 times faster than heating it dry.

In theory, wood is stabilized by decreasing its hygroscopicity by thermally degrading the hemicelluloses (the most hygroscopic poly-

mers in the cell wall). Seborg et al. (1953) speculated that crosslinking reactions were also taking place within and between cell-wall polymers during the heating process.

Seborg et al. (1945) compressed wood while heating it to produce a densified, stabilized product known as Staypak. At 160 C and 12% moisture content, they found that a specific gravity of 1.3 could be achieved at 10 to 17 MPa.

Burmester (1973) found that under optimum moisture content, heat, and pressure, deformation caused by moisture swelling was reduced 75% for oak heartwood, 60% for beech sapwood, 55% for pine sapwood and heartwood, and 52% for spruce. Giebelier (1983) found swelling decreases of 50% to 80% when beech, birch, poplar, pine, and spruce were heated at 180 C to 200 C in an inert gas atmosphere of 0.8 to 1.0 MPa.

Hsu (1988) increased dimensional stability by presteaming fibers before compressing them into fiberboards. In a 24-h swelling test, Hsu found thickness swelling of the fiberboard decreased about 33% compared to control specimens after presteaming 4 min at 1.55 MPa.

In preliminary tests, Inoue et al. (1991) found that dimensional stability could be improved by either steaming or heating while the wood was in a compressed state. Dimensional stability was achieved by heat plasticization of the thermoplastic matrix (lignin and hemicelluloses) which is reset in its compressed state and not by degradation of the hemicelluloses. Results showed that minimizing the degradation of hemicelluloses also minimized the decrease in mechanical properties of the compressed wood. In addition, we found that compressing the wood increased its surface hardness.

The purpose of the study reported here was to (a) investigate permanently compressing wood using steam or heat while the wood is in a compressed state, (b) evaluate changes in wood hardness and mechanical properties using heat or steam, and (c) determine color changes in the wood caused by the steam or heat treatments.

EXPERIMENTAL

Wood specimens

Specimens of Sugi (*Cryptomeria japonica* D. Don) were cut from one large board. Specimens for the compression and hardness tests were 20 by 20 by 30 mm (longitudinal by radial by tangential); specimens for bending and color difference tests were 120 by 10 by 5 mm (longitudinal by radial by tangential). All specimens were extracted with benzene/ethanol (1/1, v/v). Specific gravity of the wood was 0.36; average annual ring width was 1.7 mm.

Compression tests

Steam and heat treatments.—To determine the effect of steaming on dimensional stabilization of wood before and after compression set, one group of specimens was steamed at 180 C for 2, 3, 4, or 8 min and then compressed. Another group was compressed and then steamed at 180 C for 2, 3, 4, or 8 min. All specimens were then placed in boiling water (98 C) for 30 min, then measured for recovery of compression set.

To determine the effect of heat or steam on compression set, specimens were saturated with water and irradiated by microwaves at 2.4 kW. Specimens were then compressed to about 50% of their original thickness in the radial direction and then oven-dried at 105 C. Half the specimens were then heated in a dry oven, while compressed, at 160 C, 180 C, 200 C, or 220 C for various lengths of time. The remaining half were fitted between stainless steel plates that were placed inside stainless side restraints. Specimens in the metal restraints were steamed in an autoclave at 140 C, 160 C, 180 C, or 200 C for various lengths of time.

Percentage of compression set (C) was calculated by

$$C = \frac{T_0 - T_c}{T_0} \times 100\% \quad (1)$$

where T_0 is the oven-dry thickness before compression, and T_c is the oven-dry thickness after compression.

Recovery of compression set.—Recovery of

compression set was determined on specimens that were compressed and then steamed or heated by two tests. The first was a simple boiling test in which specimens were soaked in water until saturated (30 min reduced pressure, then 210 min at atmospheric pressure), placed in boiling water (98 C) for 30 min, then measured for final thickness after oven-drying. The second test was a cyclic swelling test in which specimens were soaked in water until saturated (30 min decreased pressure, then 210 min at atmospheric pressure), then measured for thickness. Specimens were dried at 40 C for 20 h, then 105 C for 4 h, then again measured for thickness. This procedure was repeated five times. After the fifth soaking cycle, the specimens were placed in boiling water (98 C) for 2 h, then measured for thickness. Finally, specimens were measured for thickness after oven-drying.

Percentage of recovery of set (R) was calculated by

$$R = \frac{T_R - T_c}{T_0 - T_c} \times 100\% \quad (2)$$

where T_R is the oven-dry thickness after recovery test.

Hardness test

A Brinell hardness test was conducted on specimens that were compressed and then steamed or heated according to Japanese Industrial Standard JIS Z2117 (1977). A 10-mm-diameter steel ball was imbedded into the surface of the wood at a head speed of 0.5 mm/min to a depth of 0.32 mm. Hardness was measured at 16 locations on each specimen, and the results were averaged. The hardness test was conducted at 20 C and 50% relative humidity.

Bending test

A single-point bending test was conducted on noncompressed specimens that were either steamed or heated for the same times as the specimens that were compressed and then steamed or heated. The span for the bending

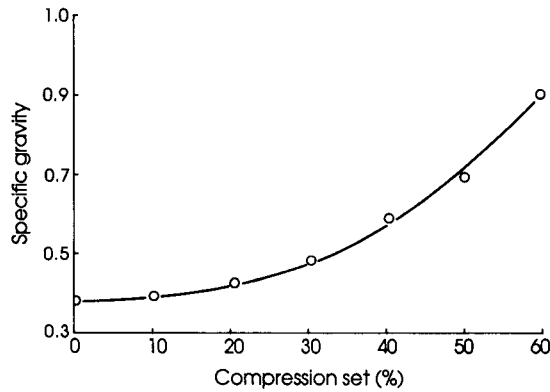


FIG. 1. Effect of compression set on specific gravity.

specimen was 100 mm with a loading speed of 10 mm/min at 60% relative humidity and 20 C. Modulus of elasticity (MOE) and MOR were calculated. Five specimens were tested at each test condition.

Color test

Color difference before and after steam or heat tests was determined using the L-a-b color system on a color difference meter. Diameter of the measuring circle was 6 mm. Sixteen locations were measured on each specimen and then averaged. Color difference and shade (lightness or darkness) were determined.

RESULTS AND DISCUSSION

Compression tests

Figure 1 shows the increase in specific gravity with increasing compression set (Inoue et al. 1991). Specific gravity increased from 0.36 to 0.50 with a compression set of 30%. At a compression set of 60%, specific gravity increased to 0.9, which was more than twice the initial specific gravity.

Figure 2 shows the results of the tests to determine the effect of steaming before and after compression set. Specimens compressed after steaming showed about an 85% regain of thickness. Therefore, steam pretreatment of wood was not effective in preventing recovery of set. Specimens steamed while compressed showed decreased recovery of set as the time of steaming increased. Almost no recovery of

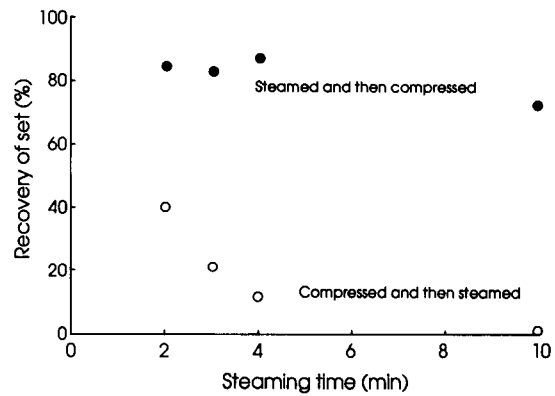


FIG. 2. Recovery of compression set after 30 min boiling for specimens steamed and then compressed and compressed and then steamed.

set was observed in specimens that were steamed for 10 min while compressed. Therefore, steaming after compression was necessary to effect permanent fixation.

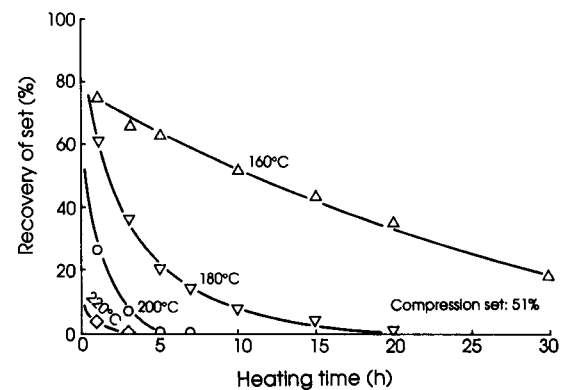
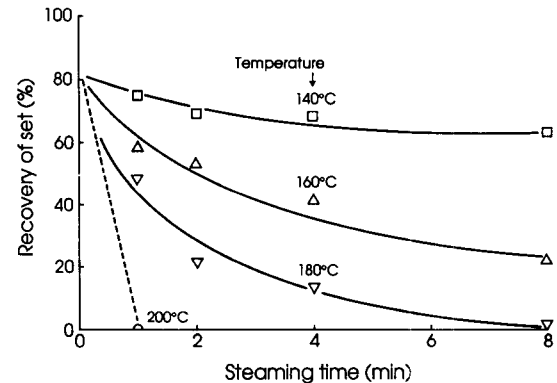


FIG. 3. Recovery of compression set of specimens steamed while compressed (top) and heated while compressed (bottom).

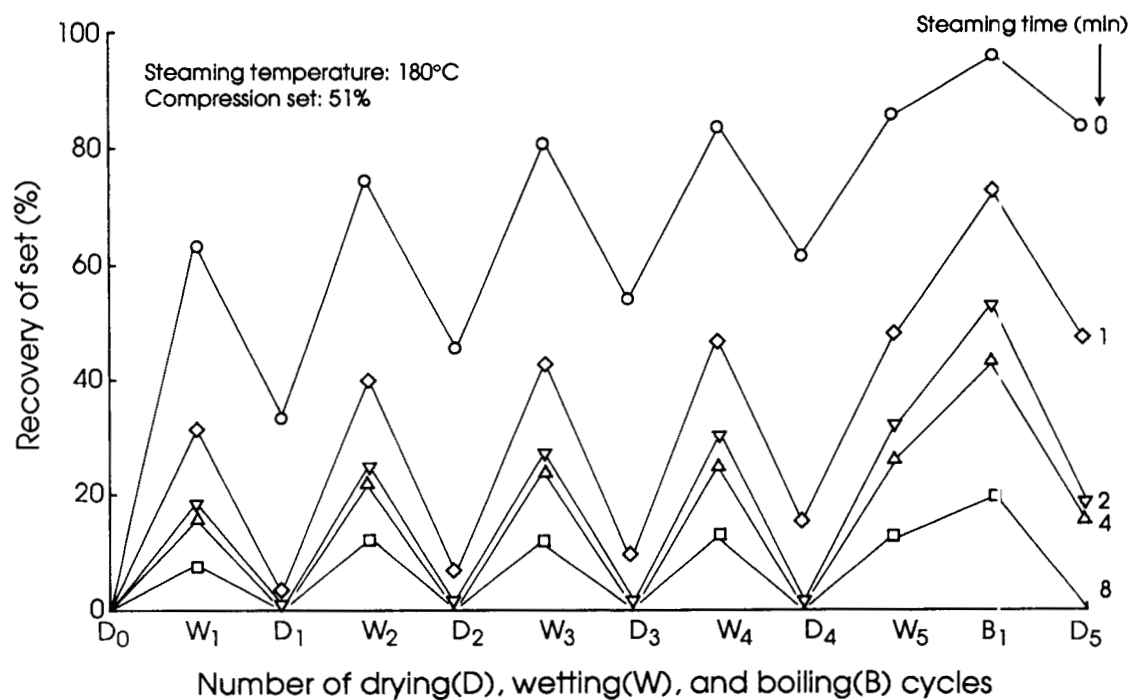


FIG. 4. Recovery of set for steam-heated compressed specimens after wetting, drying, and boiling cyclic test.

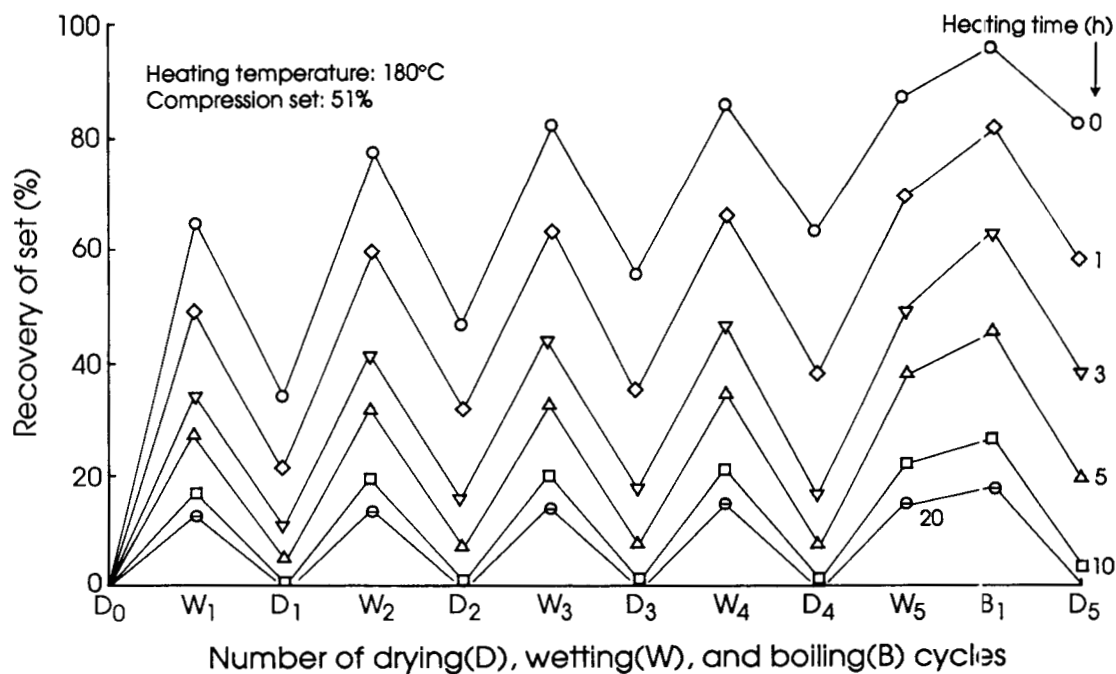


FIG. 5. Recovery of set for dry-heated compressed specimens after wet, oven-drying, and final boiling cycle test.

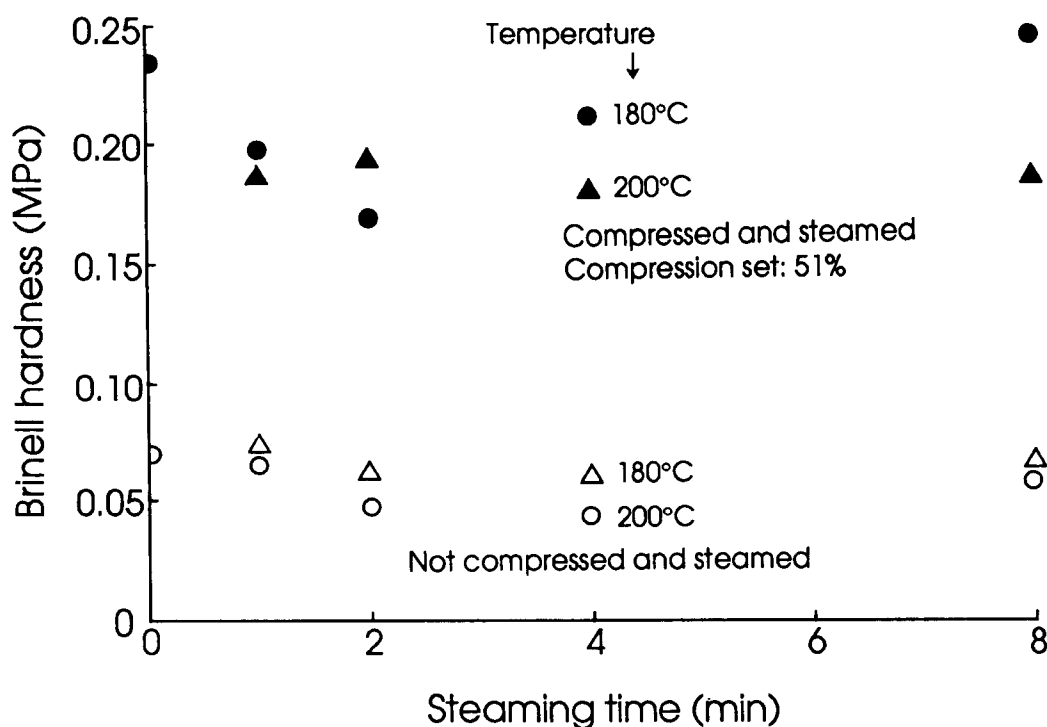


FIG. 6. Hardness of steamed specimens.

Figure 3 shows the effect of heat or steam on recovery of compression set. As the steam temperature increased, the recovery of compression set decreased. Almost no recovery of set was observed after only 1 min of steaming at 200°C and 8 min at 180°C. Dry heating required 20 h at 180°C or 5 h at 200°C to achieve less than 2% recovery.

Figure 4 shows the effects of cyclic wetting and drying on recovery of set of compressed specimens steam heated at 180°C. The unsteamed specimens (control) recovered about 60% on the first wetting cycle; after five wetting cycles, specimens recovered more than 80% of initial thickness. Specimens steamed at 180°C for 8 min while compressed recovered less than 5% on the first wetting; after five wettings, they recovered a little more than 10%. The amount of swelling that occurred in the 8-min steamed specimens was about equal to the normal, reversible, volumetric swelling of noncompressed wood.

Boiling steamed specimens after five wetting

cycles increased the recovery of set in all cases. Specimens steamed 1, 2, and 4 min had the largest increase.

Figure 5 shows the effect of dry-heated compressed specimens. Heating for 5 h at 180°C resulted in almost 50% recovery of set after five wetting cycles. Heating for 10, 15, or 20 h reduced recovery of set to 20% to 25% after five wetting cycles.

The fixation of the compressed wood structure by steaming is probably a result of the steam softening of the lignin-hemicellulose matrix in which the crystalline cellulose microfibrils are embedded. Within the softened matrix, microfibrils move. Tanahashi et al. (1989) showed that steaming of wood results in an increase in cellulose crystallinity, microfibril width, and micelle width. As water is removed from the matrix, hydrogen bonds reform between the polymers in the matrix. Together with the temperature decrease, this process leads the amorphous fraction to return to the glassy state where the elastic deformation

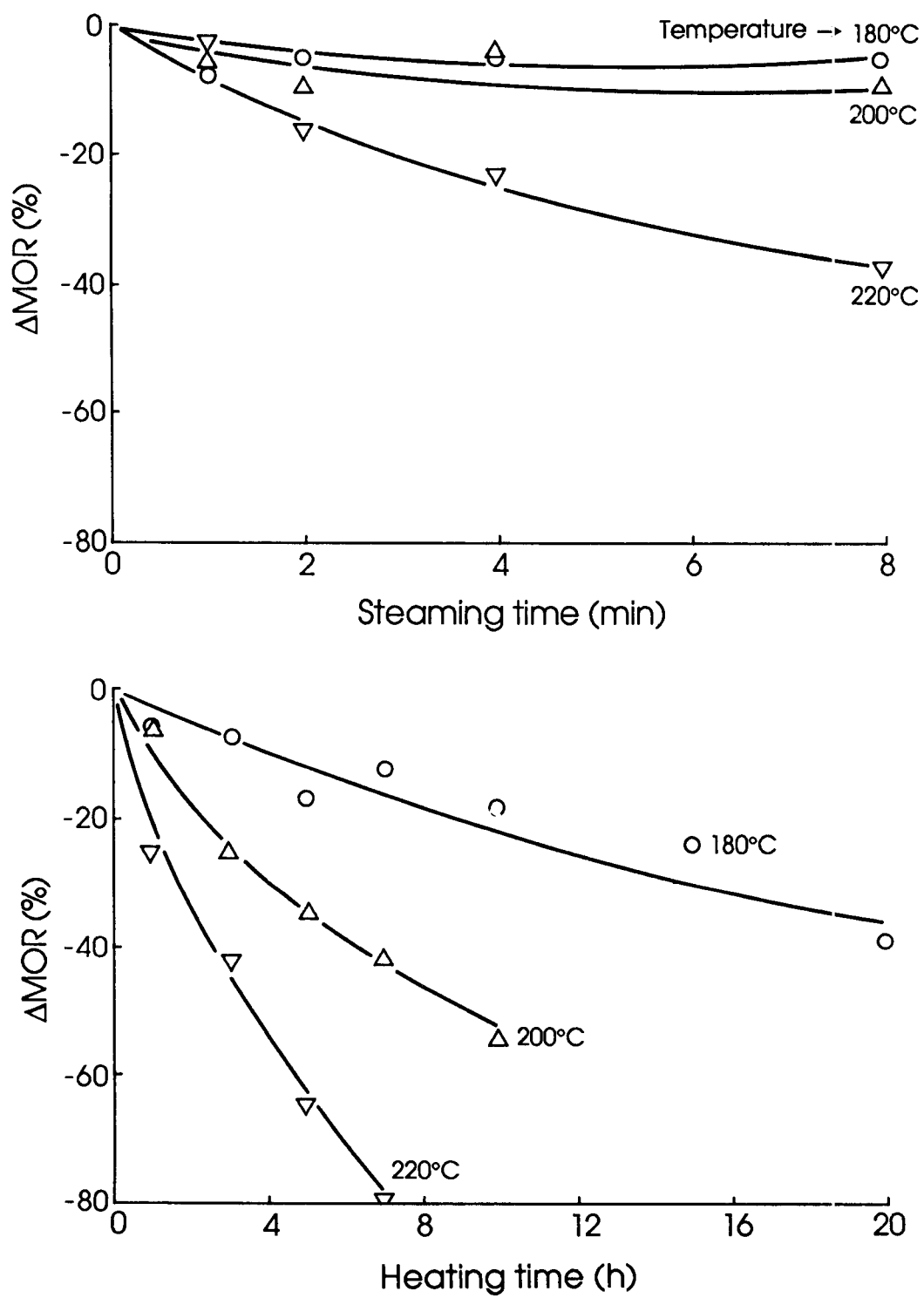


FIG. 7. Changes in modulus of elasticity (MOE) of noncompressed specimens steamed or dry heated.

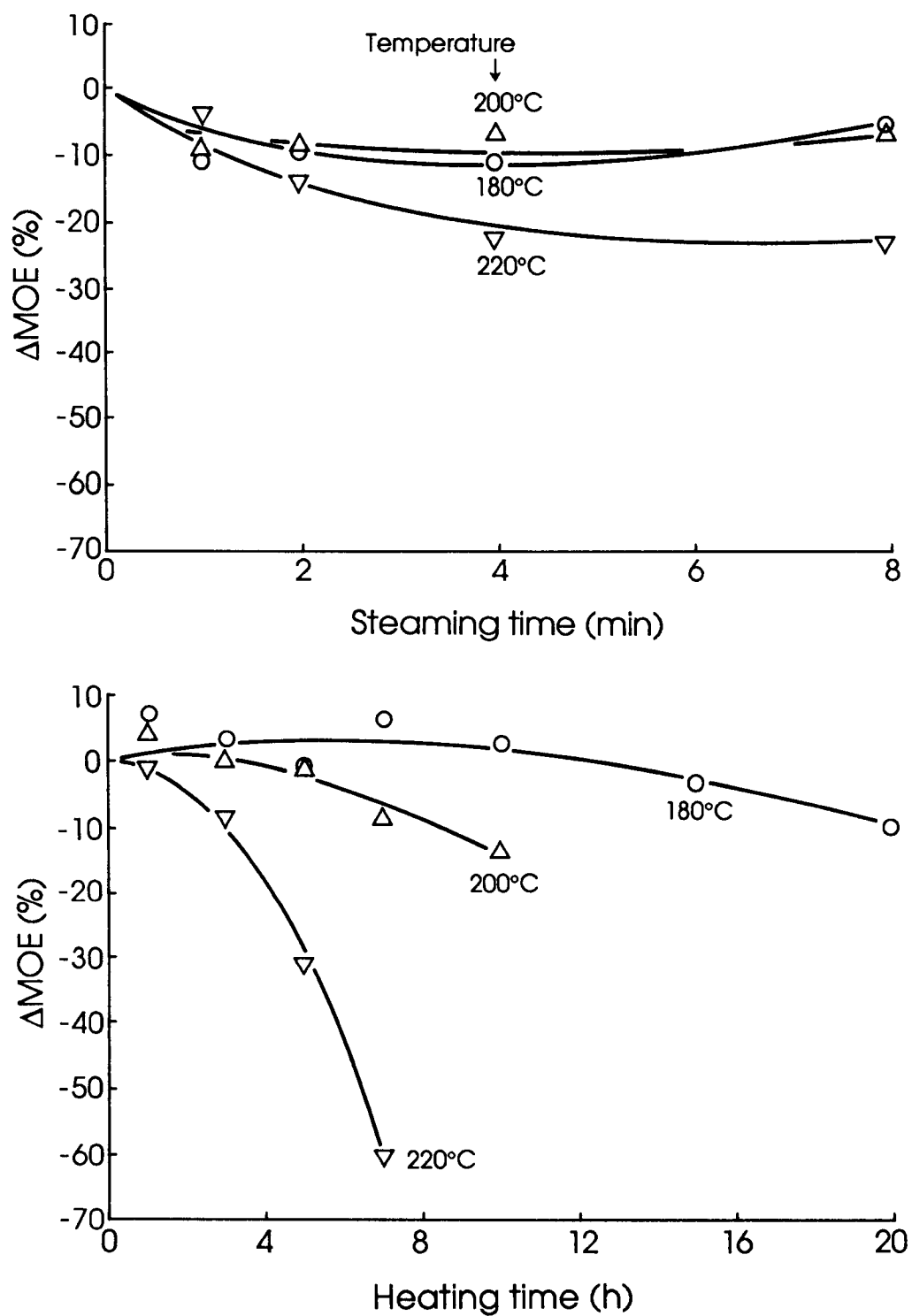


FIG. 8. Changes in modulus of rupture (MOR) of noncompressed specimens steamed or dry heated.

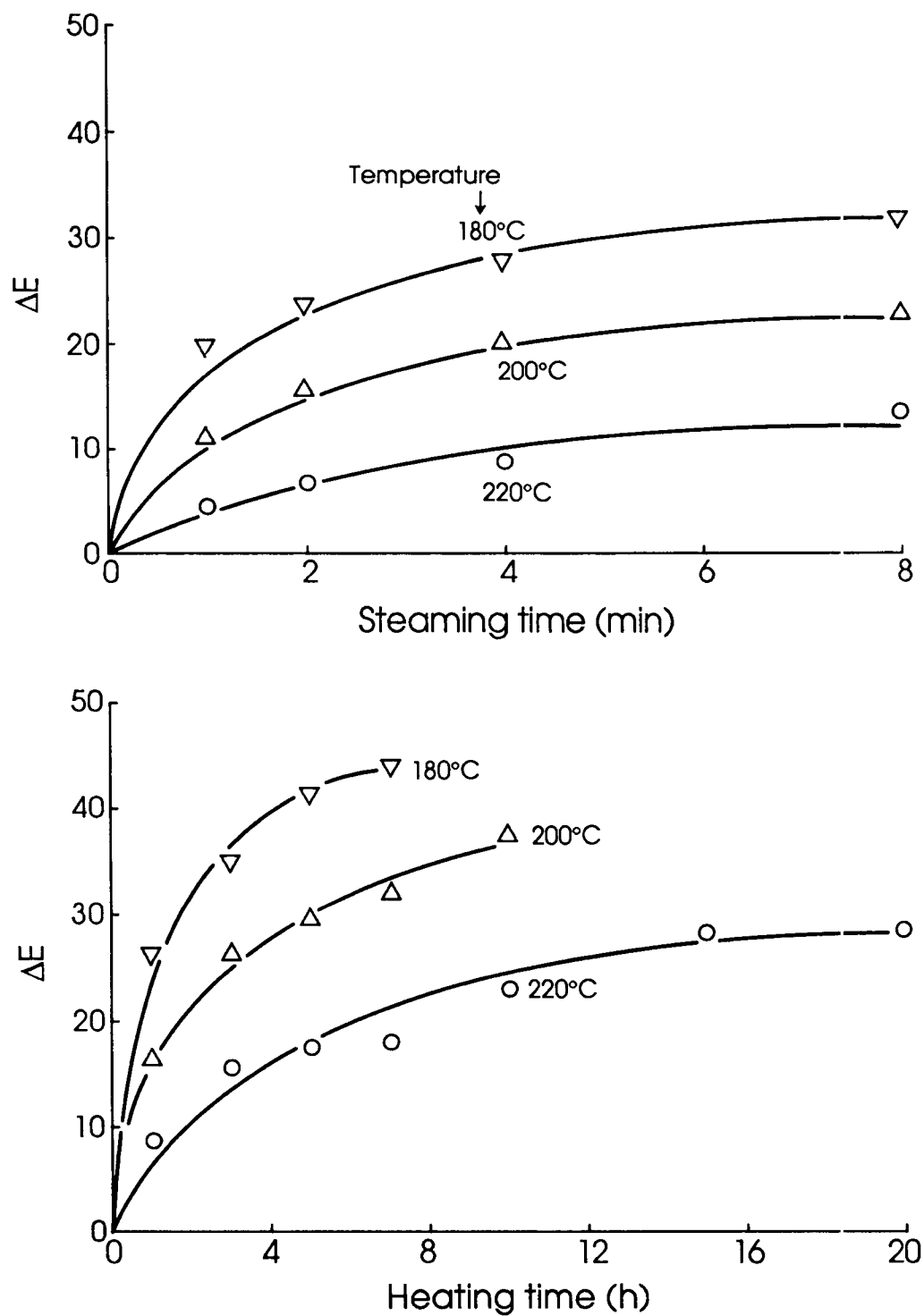


FIG. 9. Overall color change (ΔE) in noncompressed specimens steamed or dry heated.

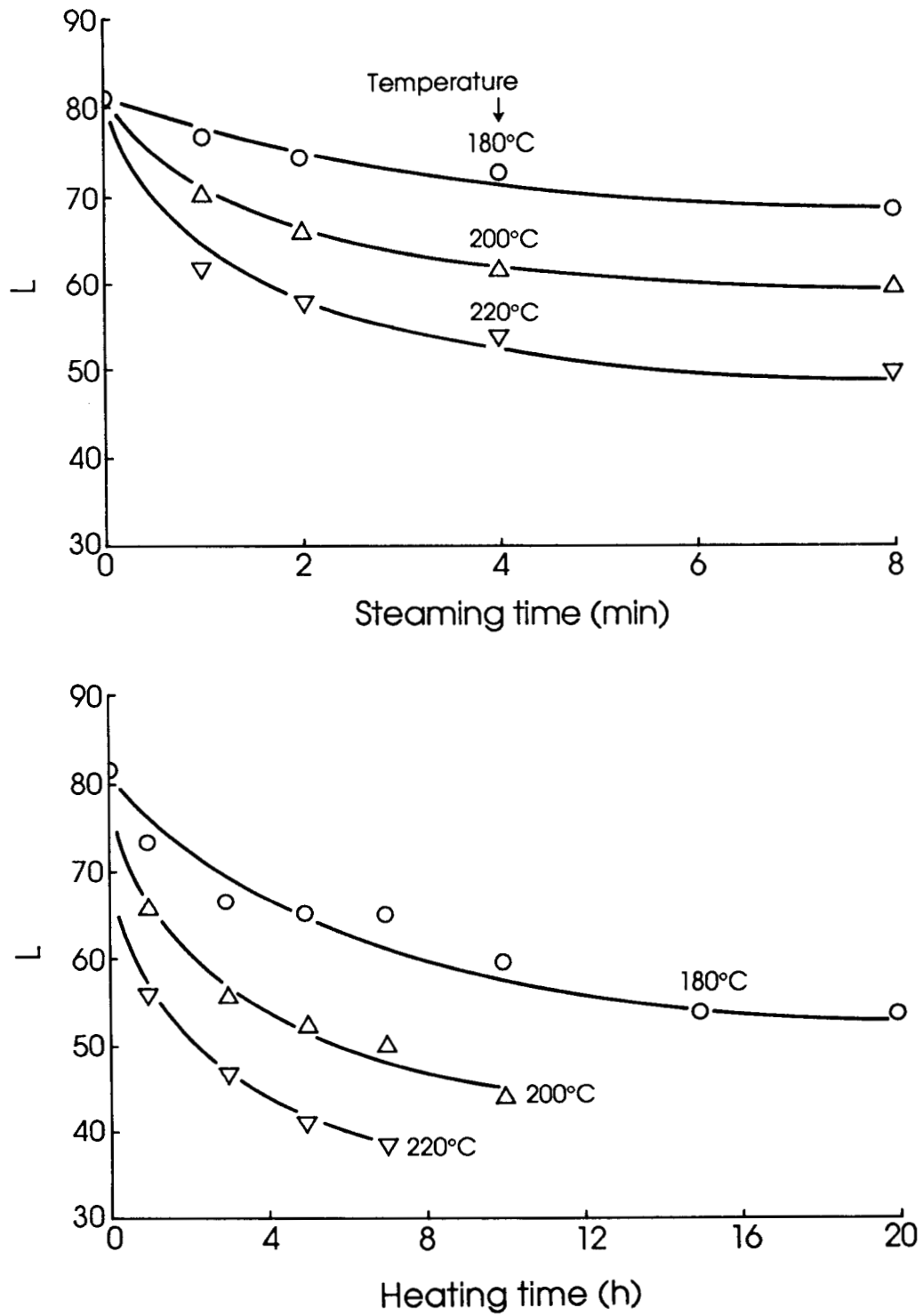


FIG. 10. Lightness or darkness (L) of noncompressed specimens as a result of steaming or dry heating.

of the microfibrils and the matrix is frozen. Part or all deformation is recovered when the matrix is resoftened with water alone or with water and heat. The part of deformation not recovered is due to the stability of the new microstructure imparted during the steam treatment.

Hardness test

Figure 6 shows the effect of steaming on hardness of specimens as measured by the Brinell test. Surface hardness remained unchanged by steaming noncompressed specimens. The hardness resulting from compression increased from 0.07 to 0.25 MPa, an increase of about three times. A small reduction in surface hardness was observed when steaming compressed specimens. This may be due to a slight loss in hemicelluloses during steaming and rearrangement of the matrix polymers.

Bending test

Figures 7 and 8 show changes in MOE and MOR of noncompressed specimens that were heated or steamed. Note that the time in Figs. 7 to 10 for steamed specimens is in minutes, while that for dry heated specimens is in hours. Steaming at 180 C for 8 min or 200 C for 1 min caused a 3.3% and 8.6% decrease in MOE, respectively. The MOE increased in specimens dry heated for 5 h at 180 C but remained unchanged at 5 h at 200 C. Steaming at 220 C for 8 min caused more than a 20% decrease in MOE, and dry heating at 220 C for 5 h caused more than a 30% decrease in MOE.

Only a small change in MOR resulted from steaming specimens at either 180 C or 200 C for 8 min (Fig. 8). A significant decrease in MOR resulted from steaming at 220 C and from all dry heating conditions, especially at 200 C and 220 C.

Color test

All heat and steam tests in this study caused a color change in the wood (Fig. 9). Steaming at 180 C caused the least color change; only a

slight yellowing occurred. Heating at 200 C and 220 C, dark yellowing occurred. Untreated specimens maintained a lightness or darkness of 80%. The lightness was reduced to about 70% for 8 min at 180 C, to about 60% for 8 min at 200 C, and to about 50% or 8 min at 220 C. All specimens were significantly darker when dry heated for lengthy periods especially at high temperatures (Fig. 10).

As seen in Figs. 7 to 10, more degradation occurred during steaming than in dry heating.

CONCLUSIONS

We conclude that almost complete fixation of compression set in wood can be achieved by steaming wood while compressed. Compressed wood steamed at 180 C for 8 min or at 200 C for 1 min showed no recovery of set, large increases in hardness, minimum decreases in mechanical properties, and slight darkening. Dry heating of compressed wood for lengthy periods can also give complete fixation of compressive set but with significant decreases in mechanical properties and severe darkening.

We are continuing this research using a water-soluble, polymerizable monomer and high-temperature curing while the wood is in a compressed state. The polymer should increase hardness and, if it bulks the cell wall, should help in the fixation of compressive set. Mechanical properties should also increase.

REFERENCES

- BURMESTER, V. A. 1973. Effect of heat-pressure-treatments of semi-dry wood on its dimensional stability. *Holz Roh- Werkst.* 31:237-243.
- GIEBELER, E. 1983. Dimensional stabilization of wood by moisture-heat-pressure-treatment. *Holz Roh- Werkst.* 41:87-94.
- HILLIS, W. E. 1984. High temperature and chemical effects on wood stability. *Wood Sci. Technol.* 18:281-293.
- _____, AND A. N. ROZSA. 1978. The softening temperatures of wood. *Holzforschung* 32:68-73.
- HSU, W. E. 1988. Steam pretreatment of wood fibers. Pages 65-71 in O. Suchsland, ed. *Proceedings, Stabilization of the wood cell wall.* Michigan State Univ., East Lansing, MI.
- INOUE, M., M. NORIMOTO, Y. OTSUKA, AND T. YAMADA.

1991. Surface compression of coniferous wood lumber II: Permanent set of compression wood by low molecular weight phenolic resin and some physical properties of the products. *Mokuzai Gakkaishi* 35(3):227–233.
- JAPAN INDUSTRIAL STANDARD. 1977. Surface hardness of wood. Standard JIS Z2117.
- SEBORG, R. M., M. A. MILLETT, AND A. J. STAMM. 1945. Heat stabilized compressed wood (Staypak). *Mech. Eng.* 67(1):25–31.
- , H. TARKOW, AND A. J. STAMM. 1953. Effect of heat upon the dimensional stabilization of wood. *Japan. Forest Prod. Res. Soc.* 3:59–67.
- SKAAR, C. 1976. Effect of high temperatures on the rate of degradation and reduction in hygroscopicity of wood. Pages 113–127 in C. C. Gerhards, and J. M. McMillen, eds. *Proceedings, High temperature drying effects on mechanical properties of soft-wood lumber*. February 25–26, 1976. USDA Forest Service, Forest Products Laboratory, Madison, WI.
- STAMM, A. J., AND L. A. HANSEN. 1937. Minimizing wood shrinkage and swelling: Effect of heating in various gases. *Ind. Eng. Chem.* 29(7):831–833.
- , H. K. BURR, AND A. A. KLINE. 1946. Stayb-wood—A heat stabilized wood. *Ind. Eng. Chem.* 38(6): 630–634.
- TANAHASHI, M., T. GOTO, F. HORI, A. HIRAI, AND T. HIGUCHI. 1989. Characterization of steam-exploded wood III: Transformation of cellulose crystals and changes of crystallinity. *Mokuzai Gakkaishi* 35(7):654–662.
- TIEMANN, H. D. 1920. Effect of different methods of drying on the strength and hygroscopicity of wood, 3rd ed. *The kiln drying of lumber*. Chap. 11, J.P. Lippincott Co., Philadelphia, PA. Pp. 256–264.