

Radial compression of sugi wood (*Cryptomeria japonica* D. Don)

W. Dwianto, M. Norimoto, T. Morooka, F. Tanaka, M. Inoue, Y. Liu

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This paper deals with the heat fixation of compressive deformation of sugi wood (*Cryptomeria japonica* D. Don) which is a typical Japanese coniferous wood with a low density. After wet specimens were compressed in the radial direction to about 50% of their original thickness and dried under restraint, they were subjected to heat treatment by three methods: beneath the surface of molten metal, and in the presence or absence of air. The relationship between the recovery of deformation and the weight loss was expressed by a hyperbolic equation regardless of heating methods. The retentions of the MOE and the MOR of compressed specimens at the perfect fixation were 89% and 81%, respectively. The perfect fixation of deformation was speculated to have resulted from the release of stresses stored in the cell wall polymers by their degradation as well as the reduction of hygroscopicity of the cell wall polymers.

Radiale Kompression von Sugiholz

Bei dieser Untersuchung handelt es sich um die Hitze-fixierung von Druckverformung bei Sugiholz (*Cryptomeria japonica* D. Don), einer typischen japanischen Nadelholzart mit niedriger Rohdichte. Nachdem feuchte Proben in radialer Richtung auf ungefähr 50% ihrer ursprünglichen Dicke zusammengedrückt, und in diesem Zustand getrocknet worden waren, wurden sie nach drei verschiedenen Methoden einer Hitzebehandlung unterzogen: Unter der Oberfläche von geschmolzenem Metall, sowie mit und ohne Gegenwart von Luft. Die Beziehung zwischen der Rückbildung der Verformung und dem Gewichtsverlust wurde mittels einer hyperbolischen Gleichung, ungeachtet der Heizmethoden, ausgedrückt. Bei perfekter Fixierung der Verformung der gepreßten Proben beliefen sich MOE und MOR auf 89% und 81%. Es wurde angenommen, daß die perfekte Fixierung der Verformung sowohl von dem Nachlassen der Spannung in den Zellwandpolymeren und durch deren Abbau, als auch durch die Verminderung ihrer Hygroskopizität bewirkt wurde.

1 Introduction

Nowadays, in Japan the supply of commercial hardwoods with high strength properties is decreasing and in the future softwoods with low densities and strength properties will be more and more available. To use these softwoods

for flooring boards, interior wall materials, furniture, and engineered composite materials, it is necessary to improve their strength properties. For this, compressing the wood in the radial direction is effective. When a compressed wet specimen is dried under restraint, the stress gradually decreases until it has disappeared (drying set). Compressed solid wood was made in Germany under the trade name of Lignostone (Stamm, 1964). However, the fixation is only apparent because it is almost reversed by boiling. The product of permanent fixation of compressive deformation if possible, may become a substitute for commercial high density hardwoods.

Many attempts to permanently fix the compressive deformation of wood have been made. They include chemical modifications of compressed wood (Fujimoto, 1992, Inoue, 1994) and resin treatments in which impregnated resins are polymerized during the deformation stage of wood (Inoue, 1990, Itoh, 1997). Heating or steaming at high temperature while the wood is in a compressed state is another effective way of fixing (Inoue, 1993). Steaming is performed in an autoclave or using a hot press equipped with an airtight seal. Although the complete fixation is achieved in very short time, the apparatus is expensive and the operation is troublesome. On the other hand, heat treatment can be easily done, using a conventional hot press, and it is of practical use in small scale production. However, not only does it take a long time to achieve complete fixation, but it is also accompanied by a reduction in mechanical properties of compressed wood. Seborg et al. (1945) compressed wood while heating it to produce a densified product known as Staypak.

The purpose of this study was to (a) measure the stress-strain relationship with loading-unloading cycles in the radial compression of sugi wood (*Cryptomeria japonica* D. Don), a typical Japanese coniferous low-density wood, to elucidate structural changes of wood by load history, (b) discuss the mechanism of drying set and its recovery, (c) achieve the permanent fixation of deformation by heating, and (d) evaluate changes in the mechanical properties of compressed wood by heating.

2 Materials and methods

2.1 Measurements of stress-strain relationship

Sugi wood (*Cryptomeria japonica* D. Don) specimens with an average density of 0.48 g/cm³ were used. The size of specimens was 3 cm in the tangential direction (T) by 2 cm in the radial direction (R) by 2 cm in the longitudinal direction (L). After conditioning for more than one month at 20 °C and 60% RH or soaking in water until saturated, they were subjected to radial compression tests with

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loading and unloading cycles in three conditions: air-dried condition at 20 °C and 60% RH, wet condition at 20 °C, and wet condition at 100 °C. The number of specimens for each condition was four. The tests were carried out using a testing machine (Shinkoh Com. Ind., Co.Ltd, TOM 5000X) with loading and unloading rates of 10 mm/min. The stress (σ) is expressed by the nominal stress (load divided by the initial section area) and the strain (ϵ) by the nominal strain (relative decrease of the end-to-end length).

2.2

Measurements of stress relaxation

Oven-dried sugi wood specimens with an average density of 0.3 g/cm³ and a size of 10 mm (L) by 20 mm (T) by 20 mm (R) were used. A testing machine (Shinkoh Com. Ind., Co. Ltd., TOM 5000X) equipped with hot plates was used for the stress relaxation measurement. Specimens were left on the hot plates for 90 sec, then compressed in the radial direction to about 50% of original thickness. Measurements were performed for 24 h at room temperature (T) and 120 °C to 200 °C at intervals of 20 °C. The number of specimens was three for each heating temperature.

2.3

Preparation and heat treatment of compressed wood

Specimens 30 mm (L) by 30 mm (T) by 20 mm (R) with an average density of 0.27 g/cm³ were used. Wet specimens irradiated with microwaves at 2.4 kW for 30 sec were compressed in the radial direction to about 50% of their original thickness by means of a pressing machine controlled at 100 °C, left under restraint for 24 h at room temperature, and oven-dried at 105 °C. The compressed specimens were then heated in an oven at 160 °C, 180 °C or 200 °C for various lengths of time by three methods, beneath the surface of molten metal (MH), and in the presence (AH) and absence (VH) of air. The composition of the molten metal was Pb = 30.6% : Sn = 51.2% : Cd = 18.2% and its melting point was 142 °C. The weight loss (WL) of compressed specimens through heat treatment was determined. Then, the specimens were soaked in water for 30 min under reduced pressure, left for 210 min under atmospheric pressure, immersed in boiling water for 30 min, and finally dried at 105 °C. The strain recovery of a specimen (SR) was defined by

$$SR = \frac{T_R - T_C}{T_O - T_C} \times 100,$$

where T_O is the oven-dried thickness before compression, T_C is the oven-dried thickness after compression and T_R is the oven-dried thickness after recovery. The number of specimens was five for each condition. To evaluate the dimensional stability of specimens provided by heating, the anti swelling efficiency (ASE) was measured using uncompressed specimens of 5 mm (L) by 20 mm (T) by 30 mm (R). The ASE was calculated by

$$ASE = \frac{S_U - S_T}{S_U} \times 100,$$

where S_T is the sum of radial and tangential swellings of the treated specimen between the oven dried state and the water-saturated state, and S_U is the value measured for the untreated specimen under the same condition. The number of specimens was eight for each condition.

2.4

Static bending test of compressed wood

Specimens 140 mm (L) by 40 mm (T) by 24 mm (R) with an average density of 0.27 g/cm³ were compressed to about 50% of their original length in the radial direction according to the procedure described in 2.3. Both the compressed and uncompressed specimens were heated in an oven at 160 °C, 180 °C or 200 °C for various lengths of time by three methods. The final size of compressed and uncompressed specimens for static bending tests was 140 mm (L) by 4 mm (T) by 12 mm (R). The test was performed using a testing machine (Shinkoh Com. Ind., Co. Ltd., TOM 5000X) at 20 °C and 60% RH. The span was 100 mm and the loading speed was 10 mm/min. The modulus of elasticity (MOE) and the modulus of rupture (MOR) of compressed and uncompressed specimens in the longitudinal direction were calculated by load-deflection curves. The number of specimens was eight for each condition.

2.5

X-ray diffraction and IR absorption measurements

Sugi wood powder passed screens of 40 and 60 meshes were used for X-ray diffraction and IR absorption measurements, respectively. The heat treatment of powder was done in the presence of air at 120 °C–210 °C at intervals of 10 °C for 20 h. Disk samples of wood powder (0.3 g) were used for X-ray diffraction measurements. Disk samples (wood powder: 0.005 g, KBr: 0.5 g) were used for IR absorption measurements. The number of samples was three for each heating condition. X-ray diffraction patterns were obtained over the range of 5°–40° using an automatic diffractometer (Rigaku-denki Geigerflex RAD2.C). The degree of crystallinity (DC) was calculated by the ratio of the area in a diffractogram corresponding to the crystalline region to that of both crystalline and amorphous regions. IR spectra were obtained using a Fourier transform infrared spectrometer (JASCO FT-IR7000).

3

Results and discussion

3.1

Stress-strain relationship

Figures 1–3 show the stress-strain relationships with loading-unloading cycles for the radial compression of sugi specimens. Almost complete recovery was observed in the wet condition at 20 °C and 100 °C provided the strain level of the first loading 1 remained small within the linear range. The curve of the second loading was nearly superimposed on that of the first loading. When the linear limit (yield point) was exceeded, the strain increased with little or no further increase of the stress. After unloading, there remained only a slight residual deformation, so that most of the strain was not plastic (apparent plasticity). The subsequent loadings never followed the previous path. Instead, the curve had a lower initial slope, followed by a new plateau for a much lower yield stress, indicating a reduction in stiffness. As soon as point 2 before unloading was reached, the apparent plasticity started again, as if no unloading had taken place in-between. As an interpretation for this plateau, we suggested that the portions of wood with the lowest density (early springwood) be crushed first, followed by the next highest in terms of density, and so on, until the portions of wood with the highest

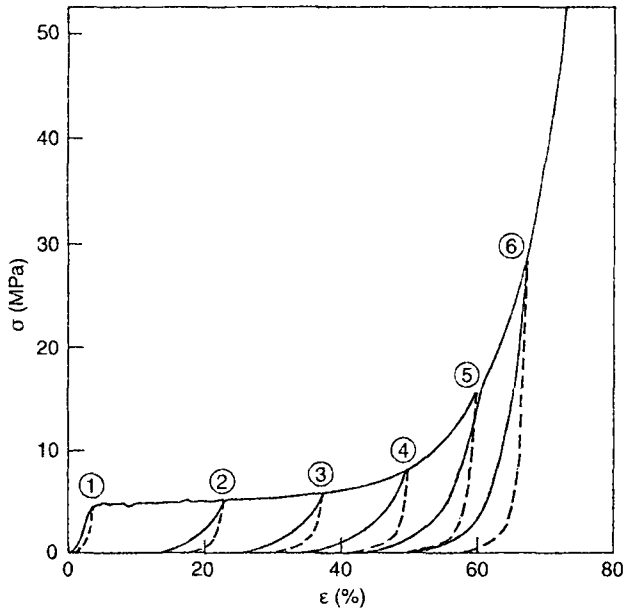


Fig. 1. The compressive stress (σ)-strain (ϵ) diagram with loading-unloading cycles in the radial direction at 20 °C for air-dried sugi wood (*Cryptomeria japonica* D. Don). Continuous lines: loading process, dotted lines: unloading process
Bild 1. Kompressionsbelastungs- (σ) und Verformungs- (ϵ) - Diagramm mit Be- und Entlastungszyklen in radialer Richtung für luftgetrocknetes Sugiholz (*Cryptomeria japonica* D. Don) bei 20 °C. Durchgehende Linien: Belastungsvorgang; gepunktete Linien: Entlastungsvorgang

density (summerwood) were crushed as well. When the cells collapsed to the extent that opposing cell walls touched at large strains, the curve showed a steep rise (densification) due to the deformation of the cell wall itself. Eventually, the highest previous stress and strain were

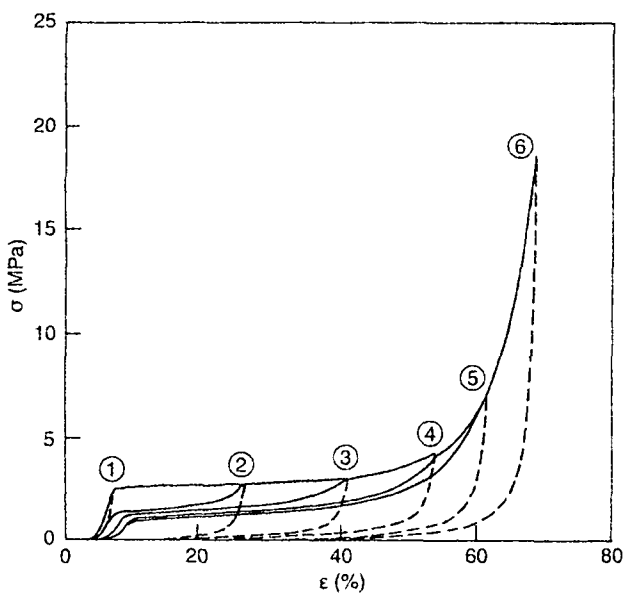


Fig. 2. The compressive stress (σ)-strain (ϵ) diagram with loading-unloading cycles in the radial direction at 20 °C for wet sugi wood. Continuous lines: loading process, dotted lines: unloading process
Bild 2. Kompressionsbelastungs- (σ) und Verformungs- (ϵ) - Diagramm mit Be- und Entlastungszyklen in radialer Richtung für feuchtes Sugiholz bei 20 °C. Durchgehende Linien: Belastungsvorgang; gepunktete Linien: Entlastungsvorgang

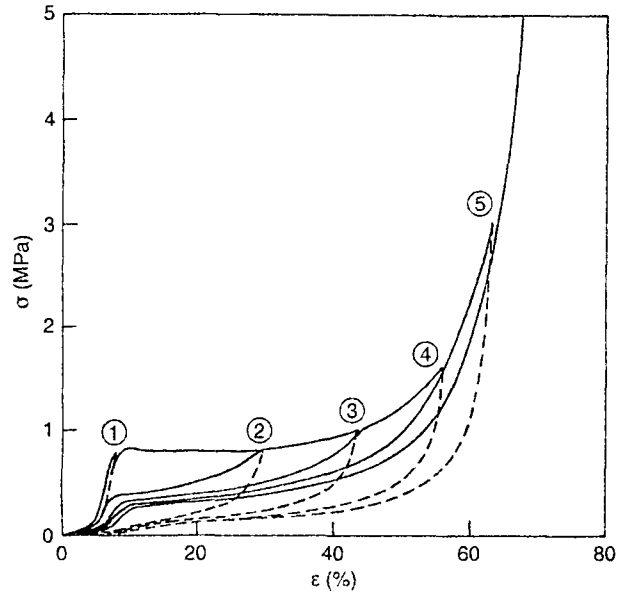


Fig. 3. The compressive stress (σ)-strain (ϵ) diagram with loading-unloading cycles in the radial direction at 100 °C for wet sugi wood. Continuous lines: loading process, dotted lines: unloading process
Bild 3. Kompressionsbelastungs- (σ) und Verformungs- (ϵ) - Diagramm mit Be- und Entlastungszyklen in radialer Richtung für feuchtes Sugiholz bei 100 °C. Durchgehende Linien: Belastungsvorgang; gepunktete Linien: Entlastungsvorgang

reached simultaneously, so that the original curve corresponding to uniform loading returned to exactly where it had been previously. Scanning electron microscopy revealed no indications of damage such as a split in the middle lamella or between layers of the cell wall. However, the irreversibility of the deformation process suggested the occurrence of some structural changes inside the cell wall. On the other hand, a large residual strain occurred on loading beyond the yield point in the air dried condition at 20 °C. Figure 4 shows the relationship between the strain (ϵ) at the unloading point and the residual strain (ϵ_R) after unloading. Although the ϵ_R of air-dried specimens increased largely with increasing ϵ , it was almost recovered by boiling. The ϵ_R of wet specimens was much smaller than that of the air-dried specimens.

Wood is composed of highly elongated cells whose walls have a complex multi-layered structure. In each layer, cellulose molecules are grouped together in long filaments called microfibrils, embedded in a matrix composed of amorphous hemicelluloses and lignin. Wood is a cellular solid and a fiber reinforced composite. Both, moisture and temperature act differently on the matrix and the microfibrils. Because of the crystalline nature of cellulose, water molecules cannot reach the microfibrils. Bound water is held in the matrix and at the interfaces between the matrix and the microfibrils, and acts as both a swelling agent and a plasticizer. The raising of temperature in the wet condition softens the matrix and its two main constituents, hemicelluloses and lignin, shifting it from the glassy state to something near the rubbery state. On the other hand, the microfibrils remain in the glassy state and are almost unaffected by moisture or heat. When a load is applied to the material, most of it is supported by the microfibrils at the local level. Softening of the matrix enables the relative displacement of the microfibrils so that their whole framework deforms elastically to adjust to the local load-

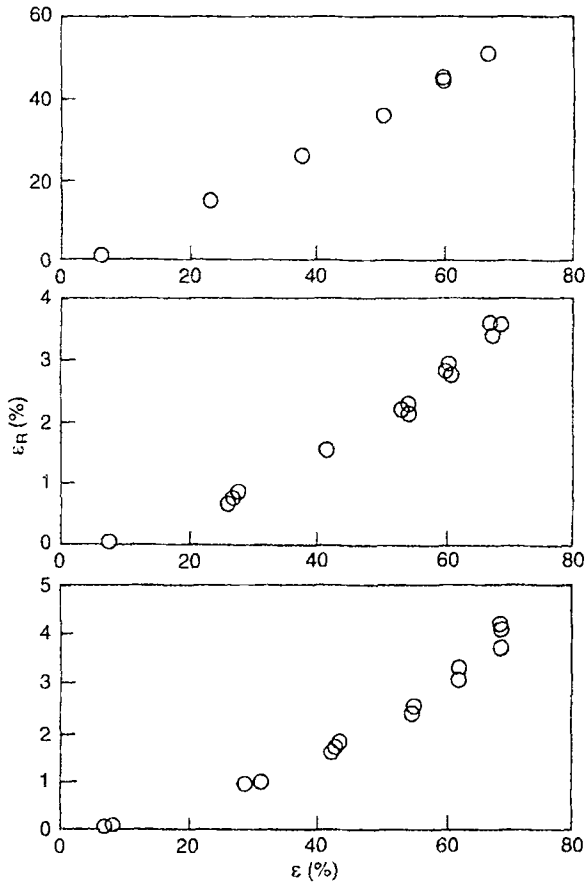


Fig. 4. The relationships between the residual strains (ϵ_R) and the strains (ϵ) at unloading points in stress-strain diagrams with loading-unloading cycles for sugi wood. Upper graph: air-dried condition at 20 °C, intermediate graph: wet condition at 20 °C, lower graph: wet condition at 100 °C

Bild 4. Beziehungen zwischen Restverformung (ϵ_R) und der Verformung (ϵ) an Entlastungspunkten in Belastungs- und Verformungsdiagrammen für Be- und Entlastungszyklen für Sugiholz. Obere Kurve: luftgetrockneter Zustand bei 20 °C; mittlere Kurve: feuchter Zustand bei 20 °C; untere Kurve: feuchter Zustand bei 100 °C

ing. As lignin is a slightly cross-linked high polymer, its deformation should be viewed as an increased viscoelastic strain rather than as plastic flow. Most of the deformation is recovered due to the liberation of energy-elastic strain stored in the microfibrils and entropy-elastic molecular movements in the matrix.

When a compressed wet specimen is dried under restraint, the stress gradually decreases until it has disappeared. The departure of water molecules due to drying induces the reformation of hydrogen bonds between the molecules of the matrix constituents. Together with the temperature decrease, this process leads to a return to the glassy state, where the elastic deformations of the microfibrils and the matrix are frozen. The deformation is fixed in the deformed state (drying set). However, this drying set is only apparent because it is almost recovered by boiling. The ratio of the recovered strain to the applied strain was 0.80–0.85 regardless of the extent of applied strain.

3.2

Permanent fixation of deformation

Figure 5 shows the stress relaxation curves of oven-dried specimens at indicated temperatures. The stress decreased rapidly with time and disappeared in 12 h at 180 °C and in

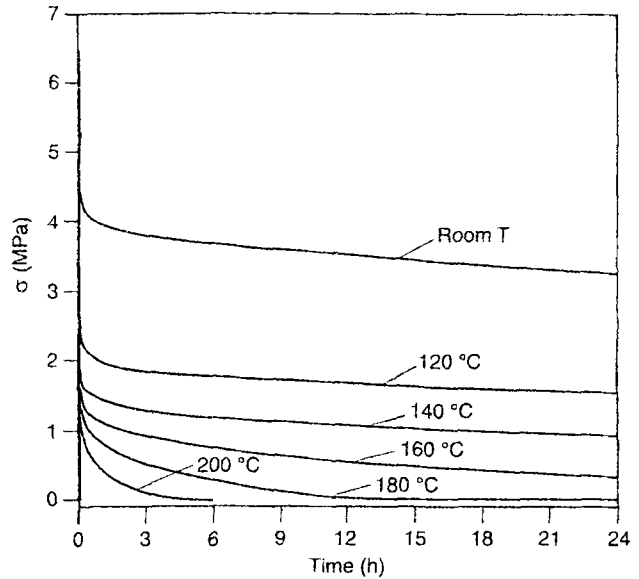


Fig. 5. The compressive stress (σ) relaxation curves in the radial direction at indicated temperatures for oven-dried sugi wood Bild 5. Relaxation der Verformungsspannung (σ) in radialer Richtung bei angegebenen Temperaturen für ofengetrocknetes Sugiholz

3 h at 200 °C. This result suggested that heating while under deformation was effective in fixing the deformation. Accordingly, the fixation of compressive deformation by heating was examined. Figure 6 shows the relationship between the SR and the heating time at indicated temperatures for three heating methods. Open circles, triangles and squares in the figure indicate the results of AH, MH and VH, respectively. Both, heating time and temperature had strong effects on the reduction of SR. The SR

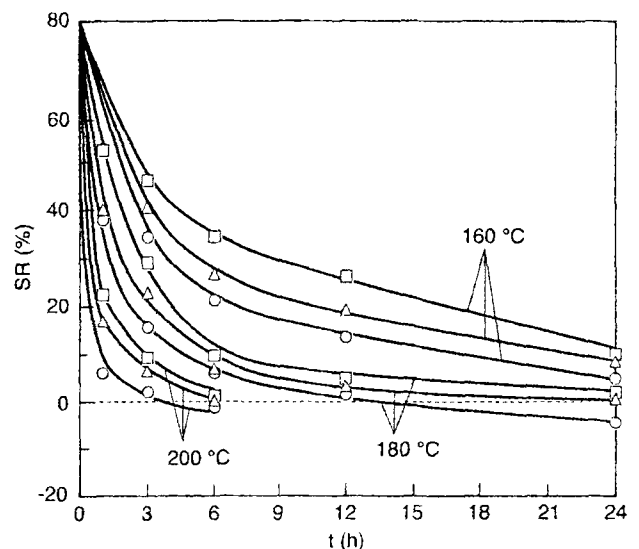


Fig. 6. The relationship between the strain recovery (SR) of compressed sugi wood and the heating time (t) at indicated temperatures and heating methods. \circ : heating in the presence of air, \triangle : Erhitzung unter der Oberfläche von geschmolzenem Metall, \square : Erhitzung ohne Gegenwart von Luft

Bild 6. Die Beziehung zwischen der Rückbildung der Verformung von Sugiholz und der Erhitzungsdauer (t) bei angegebenen Temperaturen und Erhitzungsmethoden \circ : Erhitzen in Gegenwart von Luft, \triangle : Erhitzung unter der Oberfläche von geschmolzenem Metall, \square : Erhitzung ohne Gegenwart von Luft

of unheated specimens was about 80%. The SR of heated specimens decreased with increasing heating time and temperature. The SR decreased in the order of VH, MH and AH when compared at the same heating time and temperature. A much longer time or higher temperature was necessary to fix the compressive deformation by either VH or MH. Figure 7 shows the relationship between the WL and the heating time at indicated temperatures for three heating methods. The WL increased with increasing heating time and temperature. The WLs of specimens for VH or MH were lower than those for AH when compared at the same heating time and temperature. Figure 8 shows the relationship between the SR and the WL for three heating methods. The WL increased with decreasing SR. The SR was independent of heating methods when compared at the same WL. These findings suggested that the specimens lost the same weight to reach the perfect fixation of compressive deformation. Almost perfect fixation was achieved at a WL of 4.0%. The relationship between the SR and the WL was expressed by the following hyperbolic equation.

$$SR = \frac{16.80}{WL + 0.20} - 4.00, 0 \leq WL \leq 4.0. \quad (1)$$

In heat treatment, it has been suggested that wood be stabilized by decreasing its hygroscopicity due to thermally degrading hemicelluloses as the most hygroscopic polymers in the cell wall (Tiemann, 1920, Stamm, 1964), cross-linking reactions within the cell wall polymers (Stamm, 1937) or the plasticization of the hemicellulose-lignin matrix (Hillis, 1978). Therefore, uncompressed and compressed specimens were treated with three heating methods and the SRs of compressed specimens were compared with the ASEs of uncompressed specimens. The result is shown in Fig. 9. The SR decreased linearly with

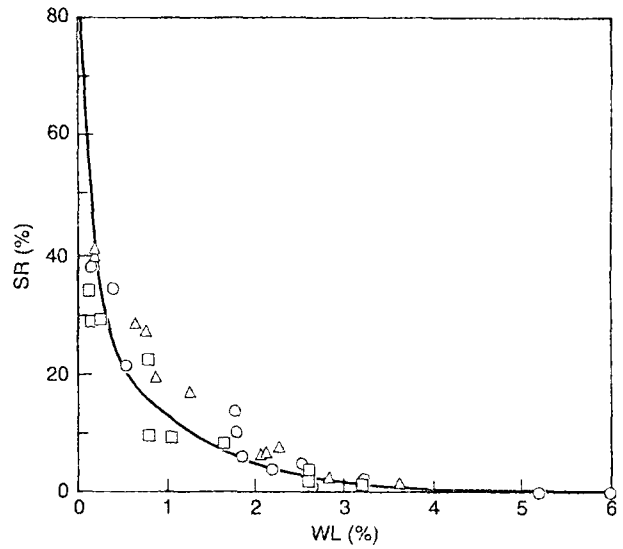


Fig. 8. The relationship between the strain recovery (SR) and the weight loss (WL) of compressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
Bild 8. Die Beziehung zwischen der Rückbildung und dem Gewichtsverlust von verformtem Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

increasing ASE. The relationship between the SR and the ASE was expressed by the following linear equation.

$$SR = -2.67ASE + 80. \quad (2)$$

The ASE at the complete fixation was about 30%.

Figures 10 and 11 show the changes of MOE and MOR of compressed specimens with heating time, respectively.

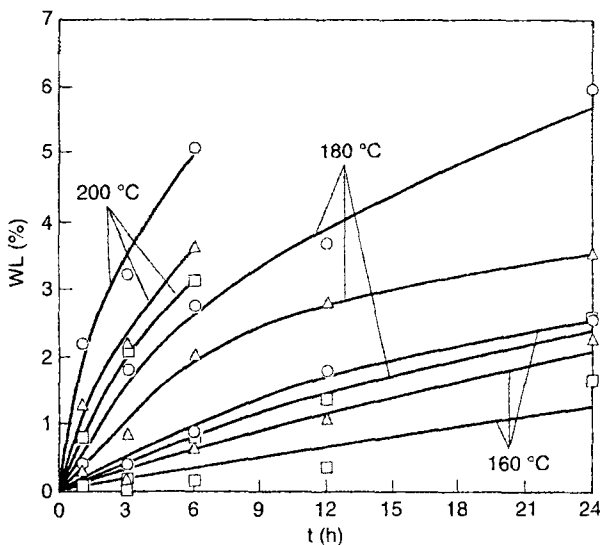


Fig. 7. The relationship between the weight loss (WL) of compressed sugi wood and the heating time (*t*) at indicated temperatures and heating methods. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
Bild 7. Die Beziehung zwischen dem Gewichtsverlust von verformtem Sugiholz und der Erhitzungsdauer (*t*) bei angegebenen Temperaturen und Erhitzungsmethoden. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

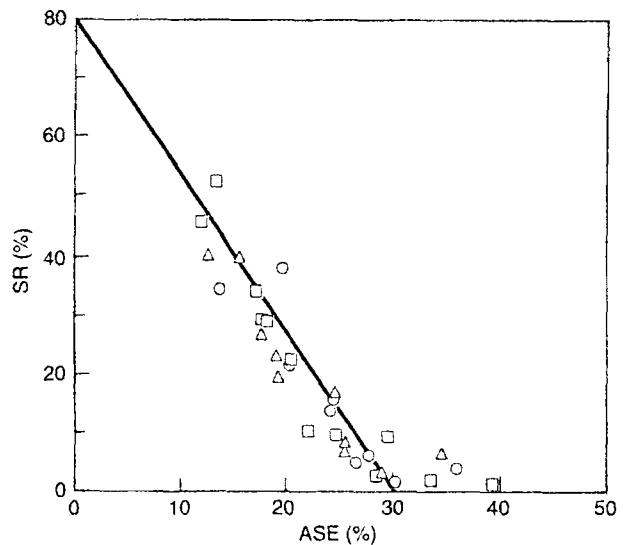


Fig. 9. The relationship between the strain recovery (SR) of compressed sugi wood and the anti-swelling efficiency (ASE) of uncompressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
Bild 9. Die Beziehung zwischen der Rückbildung von verformtem Sugiholz und der ASE von unverformtem Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

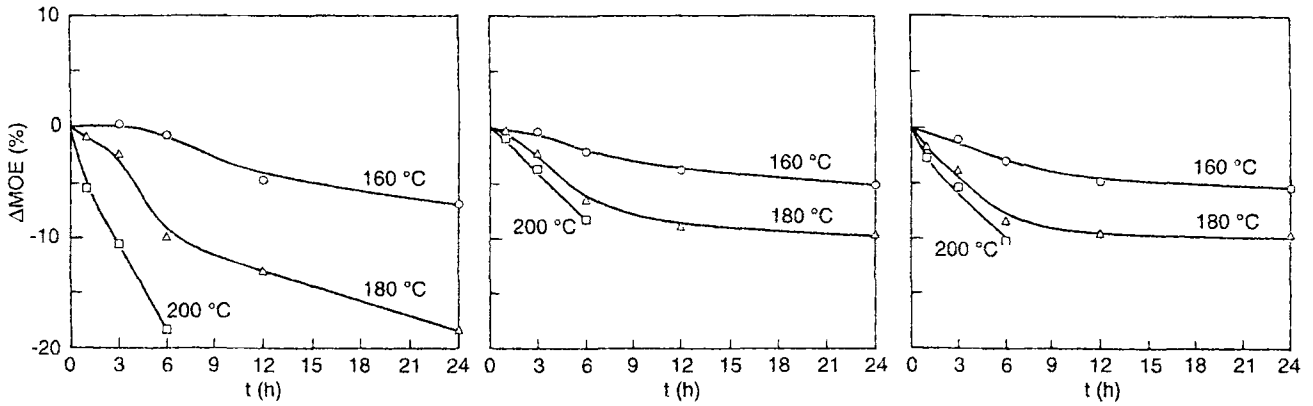


Fig. 10. The relationship between the change of MOE (Δ MOE) of compressed sugi wood and the heating time (t) at indicated temperatures. Left graph: heating in the presence of air, middle graph: heating beneath the surface of molten metal, right graph: heating in the absence of air.

Bild 10. Die Beziehung zwischen der Änderung des MOE (Δ MOE) von verformtem Sugiholz und der Erhitzungsdauer (t) bei angegebenen Temperaturen. Linke Kurve: Erhitzen in Gegenwart von Luft, mittlere Kurve: Erhitzung unter der Oberfläche von geschmolzenem Metall, rechte Kurve: Erhitzung ohne Gegenwart von Luft

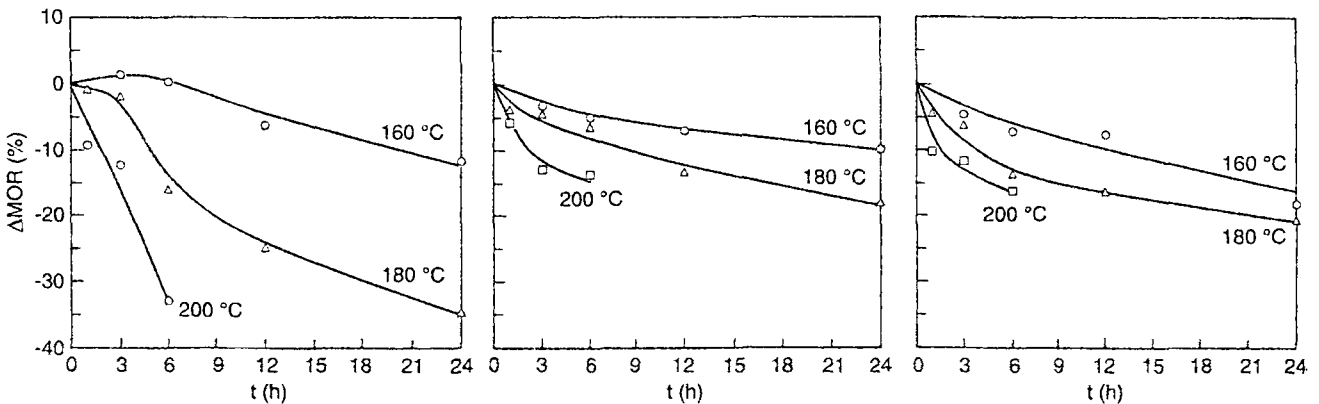


Fig. 11. The relationship between the change of MOR (Δ MOR) of compressed sugi wood and the heating time (t) at indicated temperatures. Left graph: heating in the presence of air, middle graph: heating beneath the surface of molten metal, right graph: heating in the absence of air

Bild 11. Die Beziehung zwischen der Änderung des MOR (Δ MOR) von verformtem Sugiholz und der Erhitzungsdauer (t) bei angegebenen Temperaturen. Linke Kurve: Erhitzen in Gegenwart von Luft, mittlere Kurve: Erhitzung unter der Oberfläche von geschmolzenem Metall, rechte Kurve: Erhitzung ohne Gegenwart von Luft

The MOE and the MOR decreased with increasing heating time except for the results of AH at 160 °C in which both the values increased temporarily and then decreased. The relationships of the relative MOE and the relative MOR (the ratio of the heated to the unheated) with the WL for compressed specimens are shown in Figs. 12 and 13. The relationships were expressed by

$$\text{Relative MOE} = -2.78 \times 10^{-2} \text{WL} + 1.00, \quad (3)$$

$$\text{Relative MOR} = -4.98 \times 10^{-2} \text{WL} + 1.00. \quad (4)$$

Figures 14 and 15 show the relationship of the relative MOE and the relative MOR with the SR. From Eqs. (1), (3) and (4), the following relationships were obtained.

$$\text{Relative MOE} = -\frac{0.47}{\text{SR} + 4.00} + 1.01, \quad (5)$$

$$\text{Relative MOR} = -\frac{0.84}{\text{SR} + 4.00} + 1.01. \quad (6)$$

The relative MOE and the relative MOR at the perfect fixation were 0.89 and 0.81, respectively.

The relationships of the MOE and the MOR with the density (D) for the uncompressed and compressed of both, unheated and heated specimens are shown in Figs. 16 and

17. Although the MOE and the MOR of all heated specimens decreased in both uncompressed and compressed specimens with increasing heating time and temperature, there was still an excellent correlation between MOE or MOR and D .

Figure 18 shows the relationship between the DC determined by X-ray diffraction measurement for the heated wood powder and treating temperature. The DC decreased uniformly with increasing temperature. Figure 19 shows the IR spectra of heated wood powder. Significant changes in the bands assigned to the CO and COOH groups at 1736 cm^{-1} , 1719 cm^{-1} and 1698 cm^{-1} were detected. Absorptions of these bands increased significantly above 180 °C. As mechanisms of the fixation of compressive deformation, the following may be considered: (1) the crystallization of the microfibrils, (2) the reduction of hygroscopicity of the cell wall polymers, especially hemicelluloses, (3) the cross linking formation in the cell wall polymers, and (4) the release of the stresses stored in both, the microfibrils and the matrix. The results of the mechanical properties and X-ray diffraction showed that mechanism (1) was not dominant. Although an excellent correlation was observed between the ASE and the SR, the perfect fixation was achieved at relatively low ASEs. This

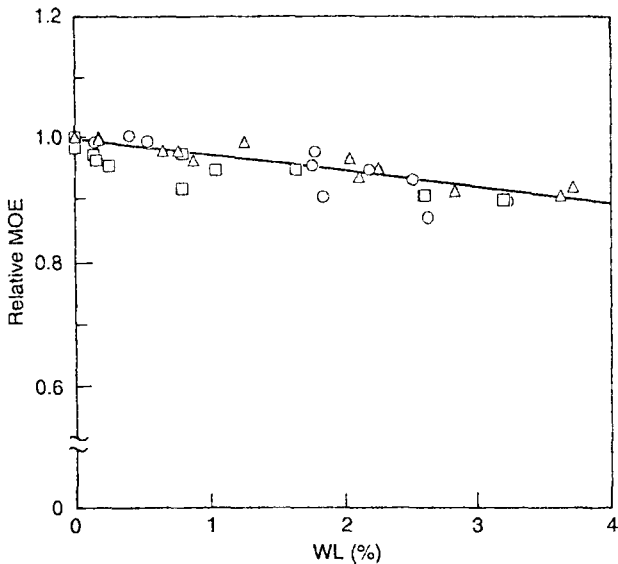


Fig. 12. The relationship between the relative MOE and the weight loss (WL) of compressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
 Bild 12. Die Beziehung zwischen dem relativen MOE und dem Gewichtsverlust von verformtem Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

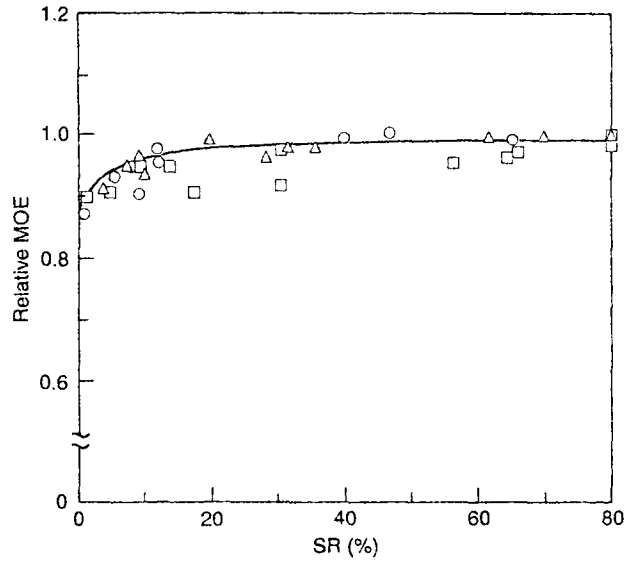


Fig. 14. The relationship between the relative MOE and the strain recovery (SR) of compressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
 Bild 14. Die Beziehung zwischen dem relativen MOE und der Rückbildung der Verformung von Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

result suggested that the reduction of hygroscopicity of the cell wall polymers is not the main mechanism. Our previous study showed that the deformation of the compressed sugi specimens was completely fixed by the treatment with formaldehyde vapor (Inoue, 1994). This fact showed that the formation of cross-linkings between the wood components was effective in fixing the deformation. However, a cross-linking reaction was disproved because swelling in pyridine and in an 18-percent aqueous

solution of sodium hydroxide was not reduced, unlike the swelling in water by the heat treatment (Seborg, 1953). The result of IR spectra suggested that the stresses stored in the matrix and the microfibrils are released by the degradation of the cell wall polymers. From these results, it was supposed that the fixation of compressive deformation by heating resulted from the release of the stresses stored in both the microfibrils and the matrix as well as the reduction of hygroscopicity of the cell wall polymers.

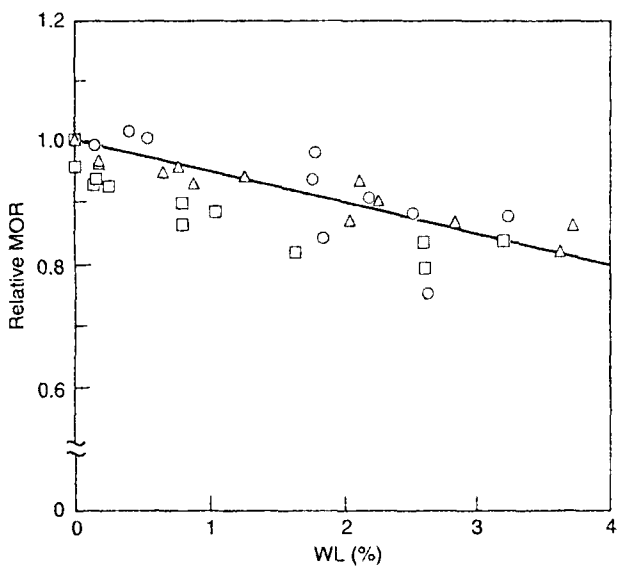


Fig. 13. The relationship between the relative MOR and the weight loss (WL) of compressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
 Bild 13. Die Beziehung zwischen dem relativen MOR und dem Gewichtsverlust von verformtem Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

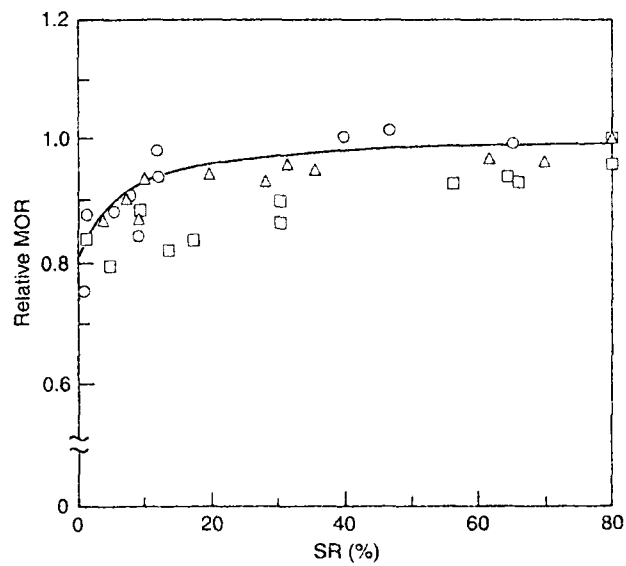


Fig. 15. The relationship between the relative MOR and the strain recovery (SR) of compressed sugi wood. ○: heating in the presence of air, △: heating beneath the surface of molten metal, □: heating in the absence of air
 Bild 15. Die Beziehung zwischen dem relativen MOR und der Rückbildung der Verformung von Sugiholz. ○: Erhitzen in Gegenwart von Luft, △: Erhitzung unter der Oberfläche von geschmolzenem Metall, □: Erhitzung ohne Gegenwart von Luft

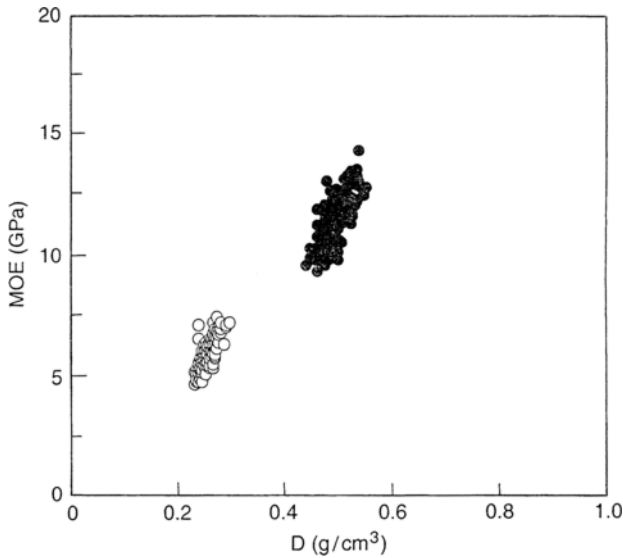


Fig. 16. The relationship between the MOE and the density (D) of uncompressed and compressed sugi woods. ○: uncompressed specimens, ●: compressed specimens.

Bild 16. Die Beziehung zwischen dem MOE und der Rohdichte von verformtem und unverformtem Sugiholz. ○: unverformte Proben, ●: verformte Proben

4

Conclusions

To utilize sugi wood (*Cryptomeria japonica* D. Don), a typical Japanese softwood with a low density, as a substitute for commercial high density hardwoods, the permanent fixation of deformation of compressed specimens was attempted by heating and the changes in mechanical properties of compressed specimens were evaluated.

In the loading-unloading cycle test for the radial compression of wet specimens, most of the strain was recovered after unloading at any strain level beyond the yield point, but subsequent loadings never followed the previous path, indicating a reduction in stiffness. No indica-

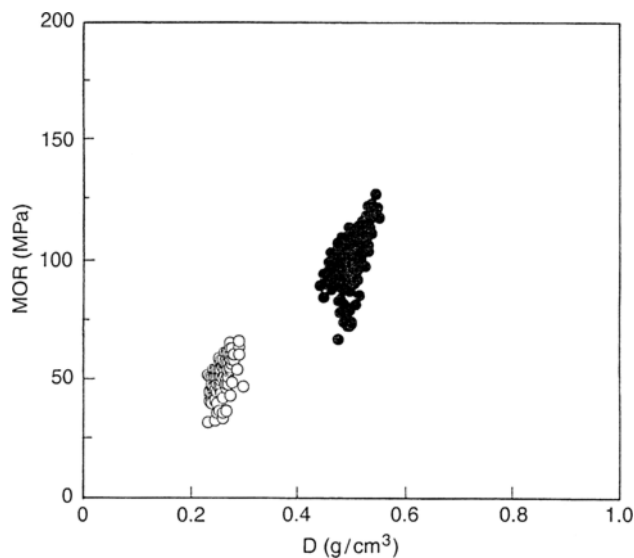


Fig. 17. The relationship between the MOR and the density (D) of uncompressed and compressed sugi woods. ○: uncompressed specimens, ●: compressed specimens

Bild 17. Die Beziehung zwischen dem MOR und der Rohdichte von verformtem und unverformtem Sugiholz. ○: unverformte Proben, ●: verformte Proben

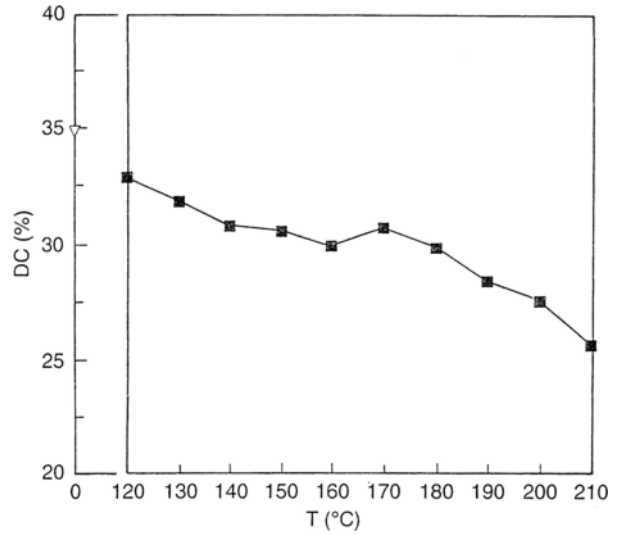


Fig. 18. The relationship between the degree of crystallinity (DC) determined by X-ray diffraction measurement and the treating temperature (T) for sugi wood powder heated in the presence of air for 20 h. ▽: unheated specimens, ■: heated specimens

Bild 18. Die Beziehung zwischen dem Kristallinitätsgrad, bestimmt durch Röntgenbeugung und der Temperatur (T) für Sugiholzmehl, das über 20 Stunden in Gegenwart von Luft erhitzt wurde. ▽: nicht erhitze Proben, ■: erhitze Proben

tions of damage such as a split in the middle lamella or between layers of the cell wall were obtained by scanning electron microscopy. However, the irreversibility of the deformation process suggested the occurrence of some structural changes inside the cell wall.

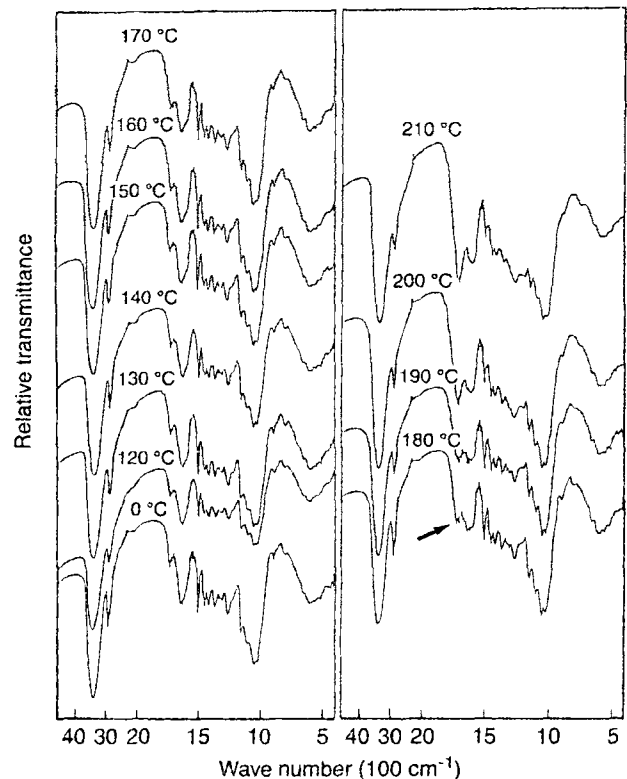


Fig. 19. The IR spectra of wood powder heated in the presence of air for 20 h at indicated temperatures

Bild 19. IR-Spektren von Holzmehl, welches in Gegenwart von Luft bei angegebenen Temperaturen für 20 Std. erhitzt wurde

When a compressed wet specimen was dried under restraint, the deformation was fixed in the deformed state. The elastic deformations of the microfibrils and the matrix in the deformed state are frozen by the reformation of hydrogen bonds between the molecules of the cell wall polymers during drying. However, most of the deformation is recovered due to the liberation of energy-elastic strains stored in the microfibrils and entropy-elastic molecular movements in the matrix by boiling.

The compressive stress relaxation tests of oven-dried specimens during heating suggested that heating while under deformation was effective for the permanent fixation of deformation. The fixation of compressed specimens was examined by three heating methods, beneath the surface of molten metal, and in the presence or absence of air. The relationship between the recovery of deformation and the weight loss was expressed by a hyperbolic equation regardless of heating methods, and the perfect fixation was attained at about 4.0% weight loss. The retentions of the modulus of elasticity and the modulus of rupture for compressed specimens by heating at the perfect fixation were 89% and 81%, respectively. The results of the mechanical tests of the X-ray diffraction and of the IR absorption suggested that the fixation of deformation by heating resulted from the release of the stresses stored in both the microfibrils and the matrix, from the degradation of the cell wall polymers, as well as from the reduction of hygroscopicity of the cell wall polymers.

References

- Fujimoto H (1992) Weathering behavior of chemically modified wood with a maleic acid glycerol (MG) mixture. *New Zealand FRI Bull.* 176: 87-96
- Hillis WE, Rozsa AN (1978) The softening temperatures of wood. *Hozforsch.* 32(2): 68-73
- Inoue M, Norimoto M, Otsuka Y, Yamada T (1990) Surface compression of coniferous wood lumber I. A new technique to compress the surface layer. *Mokuzai Gakkaishi.* 36(11): 969-975
- Inoue M, Norimoto M, Tanahashi M, Rowell RM (1993) Steam or heat fixation of compressed wood. *Wood and Fiber Sci.* 25(3): 224-235
- Inoue M, Minato K, Norimoto M (1994) Permanent fixation of compressive deformation of wood by crosslinking. *Mokuzai Gakkaishi.* 40(9): 931-936
- Itoh T, Ishihara S (1997) Compressive deformation of wood using hot rollpress and its fixation by glyoxal resin. *Mokuzai Gakkaishi.* 43(1): 52-60
- Seborg RM, Stamm AJ (1945) Heat stabilized compressed wood (Staypak). *Mech. Eng.* 67(1): 25-31
- Seborg RM, Tarkow H, Stamm AJ (1953) Effect of heat upon the dimensional stabilization of wood. *J. Forest Prod. Res. Soc.* 3(3): 59-67
- Stamm AJ, Hansen LA (1937) Minimizing wood shrinkage and swelling. Effect of heating in various gases. *Ind. Eng. Chem.* 29(7): 831-833
- Stamm AJ (1964) *Wood and Cellulose Science.* The Ronald Press Com., New York
- Tiemann HD (1920) *The kiln drying of lumber.* Lippincott, J. P. Co., Philadelphia