FIXATION OF COMPRESSIVE DEFORMATION IN WOOD BY PRE-STEAMING

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INOUE, M., SEKINO, N., MOROOKA, T., ROWELL, R. M. & NORIMOTO, M. 2008. Fixation of compressive deformation in wood by pre-steaming. Wood block specimens pre-steamed at 120–220 °C for 5–20 min were compressed in the radial direction. The recovery of set decreased with increasing pre-steaming temperature and time. The reduction of set recovery correlated with the amount of weight loss in steaming irrespective of pre-steaming temperature and time. The weight loss for the highest level of fixation of compressive deformation was about 7.5% that was obtained by pre-steaming at 210–220 °C for 10 min. Moreover, the set recovery of the pre-steamed specimen decreased as pressing temperature and time increased. The compressibility of wood block increased with increasing pre-steaming temperature and time. Large transverse compression and stress relaxation tests showed that Young's modulus and yield stresses were reduced while the stress relaxation magnitude was increased by more severe pre-steaming condition. Scanning electron micrographs showed some cell structure destruction at large deformations in specimens steamed at above 180 °C. This suggests that the wood becomes brittle due to steaming. Creep test data indicate that fluidity decreased with increasing steaming time and temperature. Pre-steam fixation of compressive deformation is due to degradation of the microfibril framework, as well as the viscous flow of the matrix components resulting from the scission of their cross-linked network.

Keywords: Set recovery, SEM, visco-elastic properties

INOUE, M., SEKINO, N., MOROOKA, T., ROWELL, R. M. & NORIMOTO, M. 2008. Proses pengewapan untuk menstabilkan kecacatan bentuk kayu akibat mampatan. Spesimen blok kayu yang diwap terlebih dahulu pada suhu 120–220 °C selama 5–20 min dimampatkan pada arah jejari. Pemulihan set berkurang dengan pertambahan suhu dan masa. Penurunan pemulihan set berkorelasi dengan kehilangan berat semasa proses pengewapan tanpa mengira suhu dan masa. Kehilangan berat bagi penstabilan kecacatan mampatan paling tinggi adalah kira-kira 7.5% yang diperoleh dengan mengewap pada 210–220 °C selama 10 min. Tambahan lagi, pemulihan set untuk spesimen yang diwap menurun apabila suhu penekanan dan masa bertambah. Ketermampatan blok kayu bertambah dengan bertambahnya suhu dan masa. Ujian mampatan melintang dan santaian tegasan menunjukkan bahawa modulus Young dan tegasan alah menurun manakala magnitud santaian tegasan bertambah dengan keadaan mengewap yang lebih teruk. Pada mampatan tinggi, mikrograf elektron imbasan menunjukkan kemusnahan struktur sel dalam spesimen yang diwap lebih daripada 180 °C. Ini mencadangkan bahawa kayu menjadi rapuh apabila diwap. Data ujian rayap menunjukkan bahawa kebendaliran menurun dengan bertambahnya masa dan suhu pengewapan. Penstabilan kecacatan mampatan dengan mengewap adalah disebabkan oleh pendegradan rangka mikrofibril serta aliran likat komponen matriks akibat daripada putusnya rangkaian silang.

INTRODUCTION

Compression of wood was investigated to increase the strength properties of fast-growing tree species. The permanent fixation of large deformation as a result of compression is very important in the utilization of compressed wood as an engineering material. In previous studies, attempts were made to fix the compressed wood by resin treatments (Inoue *et al.* 1991, 1993a, 1993b), chemical treatment (Inoue & Norimoto 1991, Inone *et al.* 1994), post-heating (Inoue *et al.* 1991) and post-steaming (Inoue *et al.* 1993c). The physical processes such as steaming or

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heating are preferred as they do not require any chemical.

Recent research has focused on pre-steaming as a technique to fix large deformation in wood resulting from compression. Previous studies (Heebink & Hefty 1969, Tomimura & Matsuda 1986, Hsu *et al.* 1988) have shown that pre-steaming is effective in reducing the thickness swelling of panel products such as waferboard and flakeboard. For example, Hsu *et al.* (1988) found that the thickness swelling of waferboard, after 72 hours of water soaking, was reduced from 38 to 13% with a 5-min pre-steam treatment at 1.55 MPa. However, the effects of pre-steaming was found to vary from earlier studies and the mechanism is not exactly known, due to the complexity of the composite material. This is because the total thickness swelling of panel products is affected not only by the recovery of compressed chips, but also the breakage of some or all of the adhesive bonds.

The purpose of this study was to investigate the effects and the mechanisms of pre-steaming on the set recovery of compressive deformation in wood. This is important for dimensional stability of compressed wood products and also to better understand the mechanism of thickness swelling in pre-steamed panel products.

MATERIALS AND METHODS

Wood specimens and steam treatment

Wood specimens were prepared from sugi *Cryptomeria japonica* and akamatsu *Pinus densiflora* with dimensions of 20 × 20 × 20 mm for compression, stress relaxation and recovery tests, and 10 (longitudinal direction) × 160 (radial)× 5 (tangential) mm for bending creep tests. The mean specific gravity and annual ring width were 0.36 and 1.7 mm for sugi respectively, and 0.46 and 4.2 mm respectively for akamatsu. The specimens were steamed in an autoclave by introducing high-pressure steam at temperatures ranging from 120 to 220 °C (at 10 °C interval) for 10 min.

Compression set and recovery

Pre-steamed specimens, either dry or wet, were compressed in the radial direction, by hot pressing, to about 50% of their original thickness. The press temperature was either 20, 100 or 180 °C and pressing times, 10, 30, 60, 120, 300 and 1800 s. In order to measure the set recovery by moisture and heat, the compressed specimens were first soaked in water until saturation (30 min under reduced pressure, followed by 210 min at atmospheric pressure), then placed in boiling water (98 °C) for 30 min, and the final thickness after oven-drying was recorded. The percentage of the set recovery is defined by the following expression:

$$\text{Recovery of set} = \frac{l_R - l_C}{l_O - l_C} \times 100\ (\%)$$

where l_O and l_C are the oven-dry thickness before and after compression respectively. l_R is the oven-dry thickness after the recovery test.

Examination using scanning electron microscope

Cross-section samples were cut with a sharp knife from the pre-steamed and compressed specimens after boiling. A surface of the section was coated with gold and examined with a scanning electron microscope.

Compression and stress relaxation tests

To determine the effects of pre-steaming on the compressibility of wood, the stress–strain relationship in compression along the radial direction was obtained at 20 °C (air-dry and wet conditions) and 80 °C (wet condition). The stress was measured continuously for 30 min while the specimen was under compression in order to obtain the stress relaxation values. A universal testing machine equipped with a water bath was used.

Creep tests

A three-point bending creep test was conducted on the steamed wet specimens. The span was 100 mm with a loading weight of 200 gf. The load was removed after 60 min, followed by creep recovery test for another 60 min.

RESULTS AND DISCUSSION

Fixation effects

The wood cell wall is composed of cellulose microfibril embedded in a matrix of lignin and hemicellulose. Since cellulose is partly a crystalline high polymer and lignin, three dimensional cross-linked structure, wood components are normally thought to be deformed elastically without plastic flow even if the deformation exceeds the yield strain. Therefore, the untreated compressed wood recovered to almost its original state when the matrix was softened again through re-moistening and heating. The set recovery is thought to be due to the release of the energy-elastic strain stored in the microfibril and entropy-elastic molecular movements in the matrix.

Figure 1 shows the effects of pre-steaming temperature on the set recovery of the compressed solid sugi after boiling. The set recovery was almost constant at lower pre-steaming temperature (below 170 °C). However, recovery began to decrease dramatically when the temperature exceeded 170 °C. Consequently, the set deformation was almost completely fixed by pre-steaming at 220 °C for 10 min.

A definite correlation existed between set recovery (SR) and weight loss (WL) for pre-steamed specimens at 140 to 210 °C (Figure 2). The linear regression line for these two parameters is expressed by the equation: SR = 10.8WL + 80.6. From the equation, the maximum weight loss for complete fixation of compressive deformation would be about 7.5%, which was obtained by steaming at 210–220 °C for 10 min. The amount of weight loss during the steaming process is related to the degradation of wood components. Therefore, reduction of set recovery in large deformation seems to result from the decomposition of wood substances.

Press time and temperature were found to have almost no effect on the set recovery of non-steamed specimens (Figure 3). For pre-steamed specimens, however, the degree of set recovery generally decreased with the extension of press time and an increase in press temperature. In the set recovery after water soaking, it was observed that in the specimens pre-steamed at 220 °C for 10 min, the fixation was achieved in a very short press time, i.e. between 10–30 s at 180 °C press temperature.

Fixation mechanisms

In order to prevent the set recovery of compressed wood after water soaking and boiling, we have proposed the following three mechanisms. The first mechanism is preventing the re-softening of wood by conversion of wood components to less water accessible components, since the set does not recover unless re-softening of the matrix occurs. The second mechanism is to form covalent cross-links between the wood components in the deformed state. The third mechanism is to release the elastic stresses and strains stored in the microfibrils and matrix. In the case of pre-steaming process, it is not possible to form new chemical covalent bonds after deformation. Hence, the mechanisms of

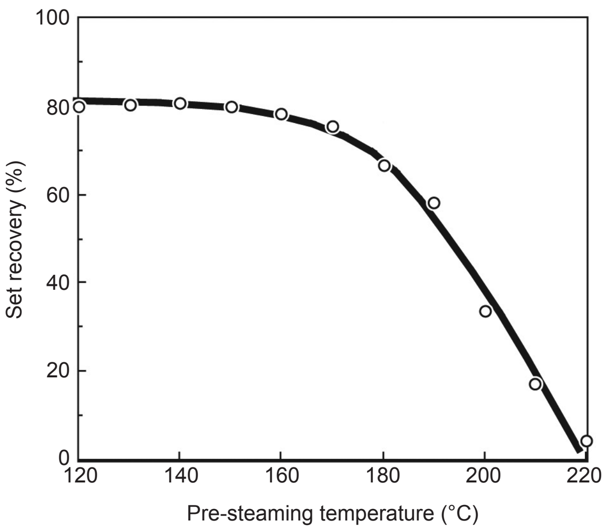

Figure 1 Effects of pre-steaming for 10 min on the set recovery of sugi compressed wood caused by boiling

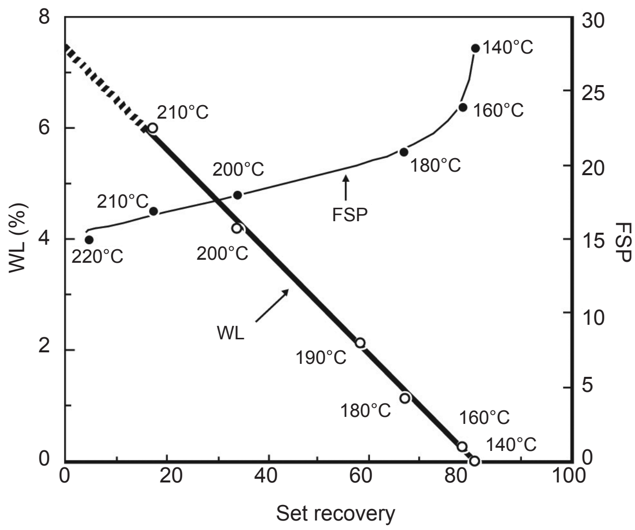

Figure 2 Relationships between weight loss rate (WL) and fibre saturation point (FSP) of the specimens after steaming for 10 min and set recovery by boiling

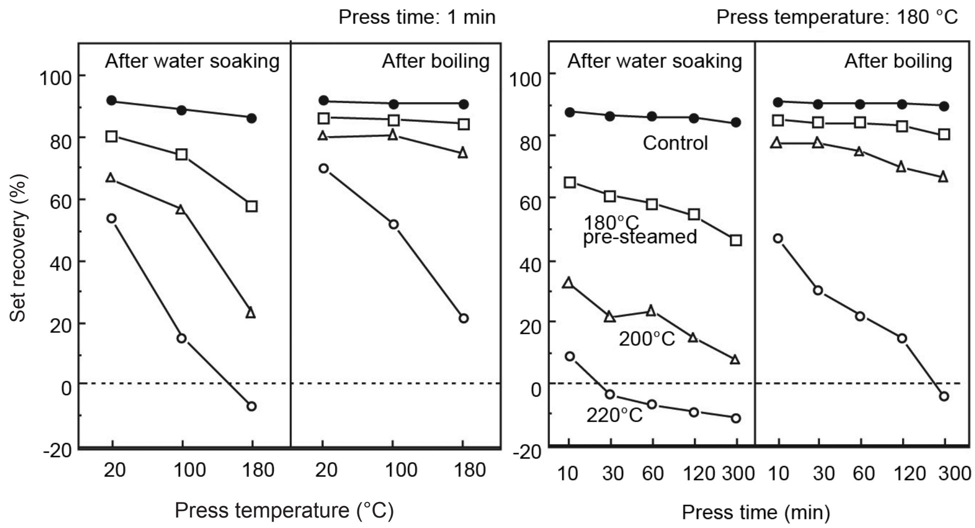

Figure 3 Effects of press time and temperature on set recovery for pre-steamed compressed wood

fixation by pre-steaming is based on the first and third mechanisms, i.e. reduction of accessibility to moisture and stresses stored by deformation.

As shown in Figure 2, with regard to a reduction of hygroscopicity after steaming, the fibre saturation point decreased with increasing pre-steaming temperature, resulting in a lower set recovery after boiling. With reference to Figure 3, the difference between set recovery of pre-steamed and control specimens was larger in water soaking than in boiling. This is further evidence that a reduction in hygroscopicity of wood components occurred after pre-steaming. The above facts imply that the first mechanism, i.e. a reduction of hygroscopicity could be one of the mechanisms behind the reduction of set recovery by pre-steaming.

In the second fixation mechanism, recovery stress reduction is explicable from the following two aspects: one is the brittle failure of the cell wall during deformation, the other is the viscous flow of wood components during and after deformation.

In relation to these considerations, it was necessary to first elucidate the brittle failure of the cell structure caused by large deformation. The cross-sections of untreated, compressed and recovered sugi specimens obtained by boiling of the compressed wood were examined under a scanning electron microscope (SEM). The cell walls were compressed without cell wall failure and lumen volume was greatly reduced (Figure 4b) as compared with the control (Figure 4a). A little residual strain existed, but the cell structure of non-steamed specimen was recovered almost to its original state, with no visible cracks, checks or disconnections of cell structure as observed in Figure 4c. On the other hand, much of the residual deformation was observed in compressed wood pre-steamed at 200 and 220 °C as shown in Figure 4d and 4e respectively. Most of the cells appeared to be deformed without considerable damage. However, there were some disconnections of cell walls and exfoliation at the middle lamella as shown by arrows in the figure. The samples that were pre-steamed at 220 °C showed cell wall damage to a large extent and many pores were formed in the cell walls, due to the extraction of decomposed wood components during steaming and recovery process. From these observations, the wood cell wall seemed to have been converted into a rather brittle material by pre-steaming, which led to a marked destruction of cell structure caused by the stress concentration during pressing. This then results in the reduction of built up stresses during compression.

To determine the compressibility of the steamed wood blocks, stress–strain and stress relaxation diagrams in Figure 5 were obtained from large transverse compression test. Like stress–strain curves for other cellular materials, the stress–strain diagram shows a linear elastic regime at low stress, followed by a long collapse plateau regime with a roughly constant stress, leading into a final regime with a steeply rising stress. When wood is compressed, the cell walls are first bent in a linear elastic manner, but when

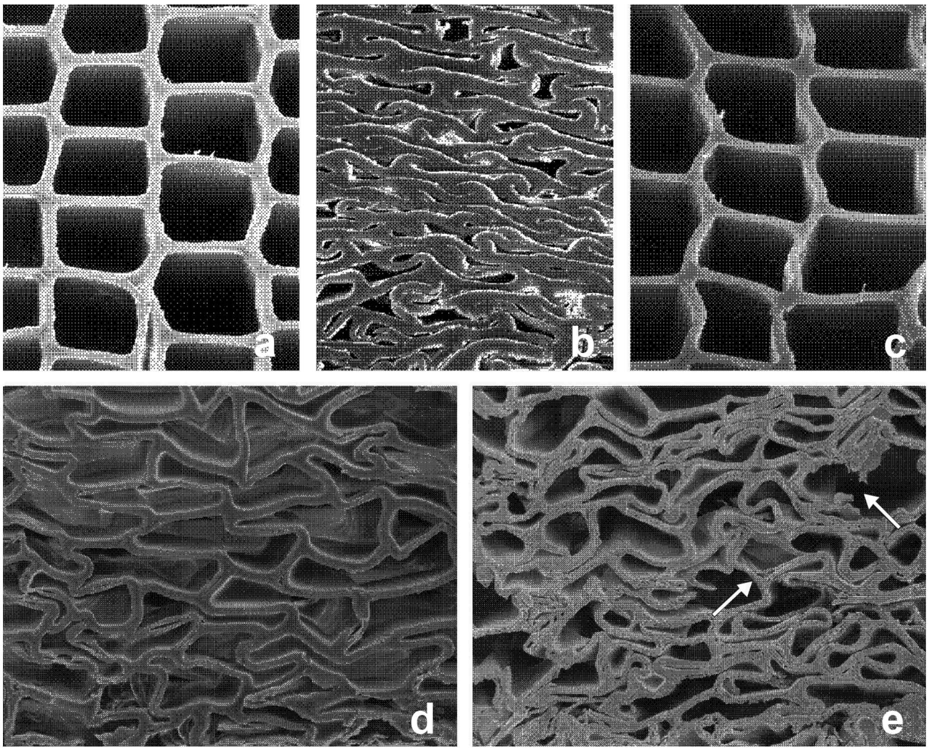

Figure 4 Scanning electron micrograph of cross section of untreated sugi (a), non-steamed compressed wood (b), and recovered specimens by boiling for untreated (c), pre-steamed at 200 °C (d) and 220 °C (e)

a critical stress is reached, the cells begin to collapse by elastic buckling. Eventually, at high strains, the cells have collapsed so much that the opposing cell walls touch each other and further deformation compresses the cell wall material itself. This gives the final steeply rising portion of the stress–strain curve. It can be seen that with harsher pre-steaming condition, the densification stress tends to decrease while the strain is not changed. The compressive Young's modulus and yield stress derived from Figure 5 decreased with increasing steaming temperature as shown in Figure 6.

We propose that stress–strain relationship in large transverse compression of wood be represented by the following equations with parameters C and K (Liu *et al.* 1993):

$$\varepsilon > \varepsilon y \qquad \frac{\sigma}{\sigma y} = 1 + C \left(\frac{\varepsilon d}{\varepsilon d - \frac{\sigma y}{E}} - 1 \right)$$

$$\sigma = E \varepsilon \varepsilon$$

$$\varepsilon d = 1 - K \left(\frac{\rho}{\rho s} \right)$$

where σy, εy, E, ρ, ρS, εd are yield stress, yield strain, Young's modulus, density of wood block, density of wood cell wall and strain at the cell lumen respectively.

Parameter K is related to Poison's ratio at the region of large deformation. Parameter C indicates the degree of increasing stress at the plateau regime of stress–strain curves and it usually has no connection with the visco-elastic behaviour of wood. The numerical values of fitted parameter C are shown in Figure 7. The values of C decreased with harsher pre-steaming condition, which shows that the compressibility and stress relaxation of wood while pressing are increased by pre-steaming process.

Stress relaxation curves after the compression test are shown in the right side of Figure 5. For all pre-steaming conditions, the stress decreased dramatically in the first several minutes. The residual stress up to 30 min of relaxation decreased with increasing pre-steaming temperature. In this study, since it took more than two minutes to compress the specimens, much of the stress relaxation had already occurred during this deformation process. The relationship between the yield stress and residual stress based on the

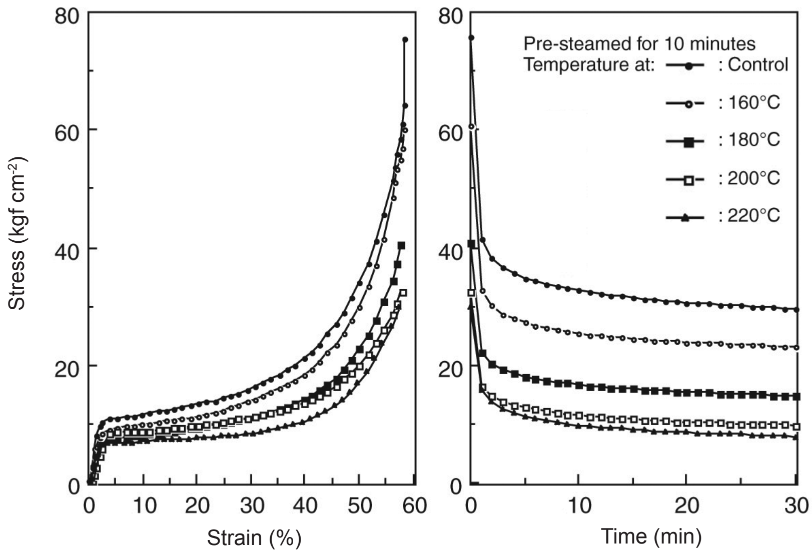

Figure 5 Stress–strain and stress relaxation diagrams for pre-steamed wood

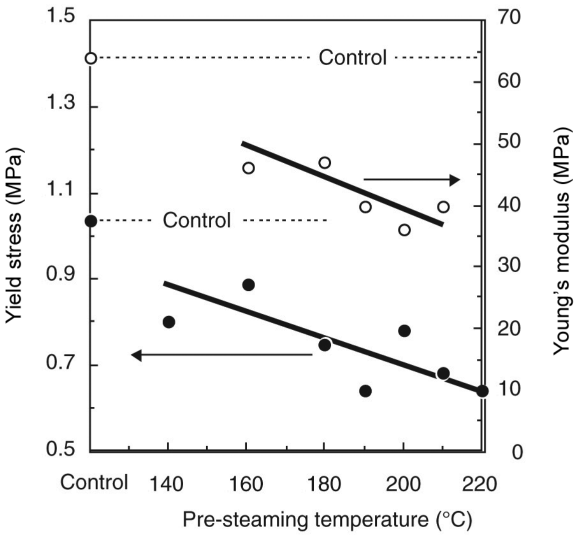

Figure 6 Effect of steaming for 10 min on yield stress (o) and Young's modulus (●) in radial compression test

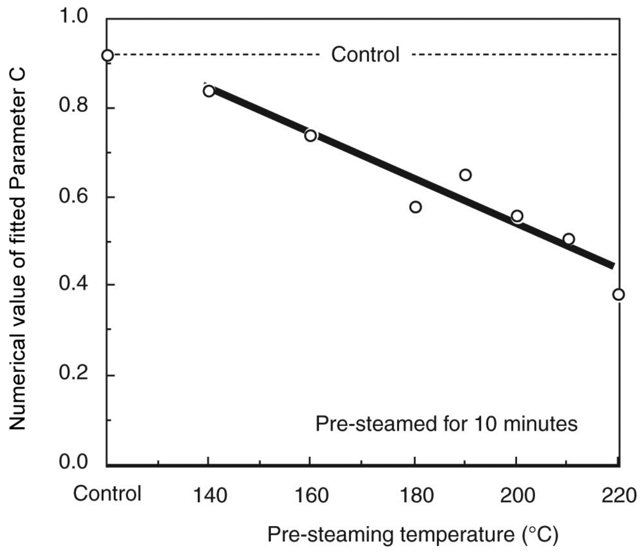

Figure 7 Effects of pre-steaming for 10 min on parameter C in radial compression test

untreated specimen is shown in Figure 8. When there is no change in the visco-elastic behaviour of wood substance, there must be a correlation between yield stress and residual stress after relaxation. However, the results in the figure show that the reduction in the residual stress is higher than in the yield stress, indicating that the stress relaxation magnitude is accelerated by pre-steaming.

Figure 9 shows the radial bending creep and recovery curves of wet sugi. Creep compliance J(t) is composed of the sum of elasticity J_0, retardation Jd (t), and plastic flow t/η as shown in the following equation.

$$J(t) = J0 + Jd\varphi\ (T) + \frac{t}{\eta}$$

Suppose that the plastic behaviour originates only from the viscous flow, viscosity of the specimens can be determined by the following expression through the measurements of the creep and its recovery (Takemura 1966).

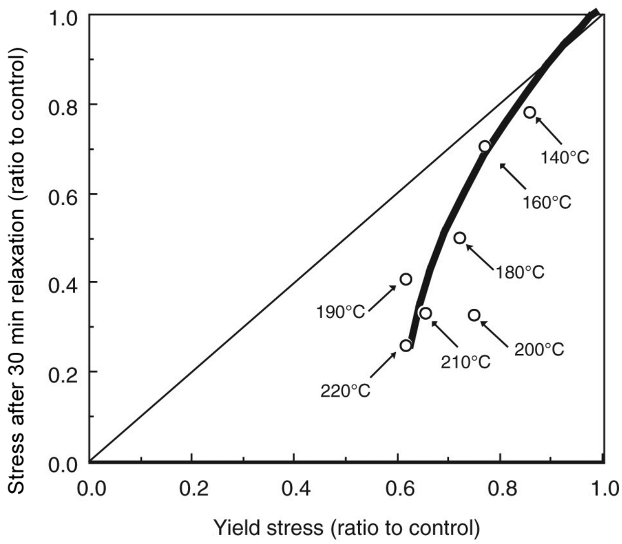

Figure 8 Relationship between stress after 30 min relaxation and yield stress

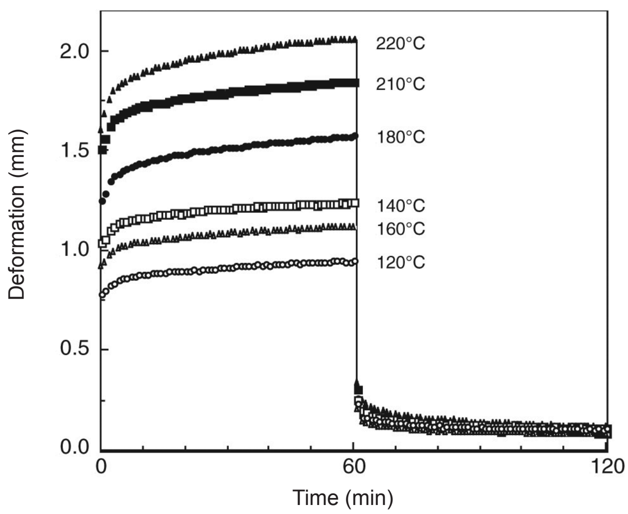

Figure 9 Effect of pre-steaming for 10 min on radial bending creep and creep recovery in wet condition

$$J(t) = J_{rec}(t) = \frac{t}{\eta}\ \underset{t}{\varphi\ (t_0 - t)} - \varphi\ (t_0)$$

where $\psi(t)$ is retardation compliance and t_0 the time at unloading. The resulting plots at treating temperature from $J(t)$ to $J_{rec}(t)$ are shown in Figure 10. In the figure, the value of $J(t) - J_{rec}(t)$ increased with time. It seems that they increase linearly above 20 min within the treating temperature. It should be noted that the terms $(t_0\text{-}t)\text{-}(t_0)$ in the above expression is known to approach zero after measurements for a long time, indicating linear relationship between $J(t) - J_{rec}(t)$ and t. Such being the case, the slope appearing in the measurements after 20 min is thought to correspond to fluidity $1/\eta$.

Figure 11 shows fluidity versus pre-steaming temperature where fluidity is based on the control specimen. From the figure, fluidity increased as the pre-steaming temperature increased. This fact suggests that the viscous flow may occur in wood substances as a result of the release of stress in the microfibril and matrix portion during pre-steaming.

CONCLUSIONS

Pre-steaming is effective in the fixation of compressed solid wood. The set recovery of the deformation generally decreased with increasing pre-steaming temperature and time, and it is closely correlated to the percentage

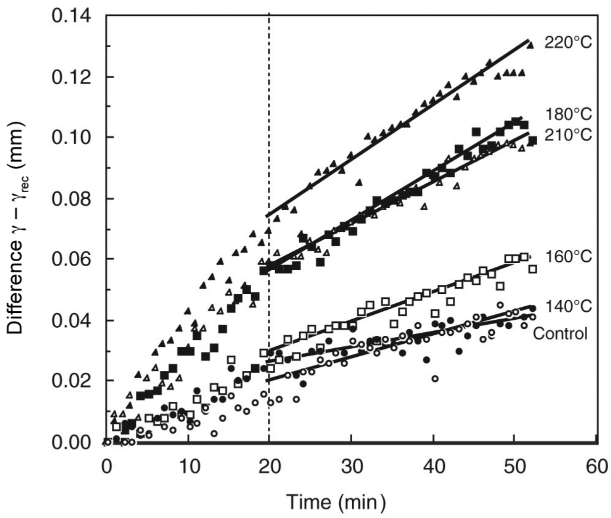

Figure 10 Difference between creep deformation (γ) and creep recovery (γ_{rec}) related to loading duration

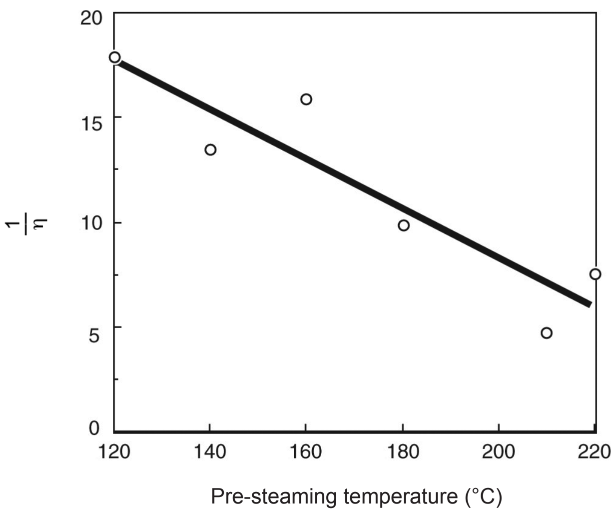

Figure 11 Change in fluidity by steaming for 10 min

of weight loss that occurred during the pre-steaming process. Pre-steaming increased the compressibility of wood and reduced the level of stress stored by compression. Based on the SEM observation, the cell wall was smoothly compressed on the whole. However, a few cracks, snaps and disconnections of the cell walls were observed. The stress relaxation and creep tests confirmed that pre-steaming process promotes the viscous flow of wood substances, resulting in the reduction of the stored stress. From these facts, it can be concluded that pre-steamed wood can be plastically deformed with the brittle failure of the cell wall and *in situ* viscous flow.

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