Ikuho Iida · Koichi Murase · Yutaka Ishimaru

# Stress relaxation of wood during the elevating and lowering processes of temperature and the set after relaxation

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Abstract Stress relaxation of water-saturated wood was examined at various temperatures and during the processes of elevation and lowering of temperature. The difference between relaxation at a lower temperature and that at a higher temperature was almost same as the increase in relaxation during temperature elevation. Similar results were obtained regardless of the elevating speed of the temperature and regardless of preheating the samples. On the other hand, relaxation behavior during temperature lowering was quite different from that during temperature elevation. The relaxation moduli during the process of lowering the temperature scarcely changed, although those at various constant temperatures decreased with increases in temperature. Marked sets occurred after relaxation measurements under temperature lowering, whereas only slight sets occurred during the process of elevating the temperature or at a constant temperature of 80°C. Furthermore, it was found that the sets that occurred during the process of temperature reduction were almost recovered when the samples were heated again in water. The mechanism of the relaxation behaviors observed in the present study under nonequilibrium temperature is discussed in relation to the occurrence of set after relaxation measurements.

Key words Wood · Stress relaxation · Temperature · Nonequilibrium state · Cooling set

## Introduction

The viscoelastic properties of wood during the processes of changing temperature and moisture content are important

I. Iida (⊠) · K. Murase · Y. Ishimaru Laboratory of Wood Technology, Kyoto Prefectural University, Kyoto 606-8522, Japan Tel. +81-75-703-5639; Fax +81-75-703-5639 e-mail: i\_iida@kpu.ac.jp

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for wood manufacture. However, the viscoelastic behavior of wood during the process of changing temperature has not been studied extensively, although viscoelastic behavior during the process of changing moisture content has been discussed in detail.<sup>1-6</sup>

Kitahara and Yukawa<sup>7</sup> reported the behavior of bending creep in the longitudinal direction of wood with elevation and lowering temperature. Arima<sup>8-10</sup> studied compressive creep in the radial direction, and Schniewind<sup>11</sup> examined tensile creep in the tangential direction.

All of these authors reported that larger creep deformations occurred with elevation of temperature than at the highest constant temperature examined. They interpreted this to mean that wood has higher fluidity during the process of temperature elevation than at the highest constant temperature, which is attributed to temperature gradients in wood samples. However, a temperature gradient in wood would not positively affect creep deformation because the temperature in the inner layer during the process of temperature elevation would always be lower than the environmental temperature with which any part of the sample would be equilibrated at constant temperature. Consequently, creep deformation during the process of temperature elevation cannot occur beyond that under the highest constant temperature due to temperature gradients in samples.

Moreover, the above results were principally discussed without consideration for an increase in instantaneous deformations (elastic deformations) with elevating temperature.<sup>7</sup> That is, instantaneous deformations increase with temperature elevation because the deformations during the process of temperature elevation include elastic deformation that increases with temperature. Therefore, creep deformations during the process of temperature elevation should be larger than those at constant temperature. Arima<sup>8</sup> discussed the increased amount of elastic deformation during temperature elevation, but the accuracy of his measurement is doubtful because accurate measurements of instantaneous deformation from creep experiments are difficult. Consequently, it cannot be determined whether the fluidity of wood during temperature elevation is higher than that at constant temperature.

Therefore, the viscoelastic properties during the process of temperature changes have not been sufficiently determined and the mechanisms involved not yet clarified. The aim of this study was to clarify the viscoelastic properties of wood under constant, elevating, and lowering temperatures and then to define the mechanisms responsible for these behaviors.

# **Materials and methods**

#### Test specimens

Wood samples were obtained from the heartwood of a log of hinoki (*Chamaecyparis obtusa*). To measure stress relaxation, more than 100 wood samples [12 (radial direction, R)  $\times$ 1 (longitudinal direction, L)  $\times$ 0.4 (tangential direction, T) cm] were successively cut from several sticks with a cross section of 12 (R)  $\times$ 1 (L) cm and a length of about 40 cm. These specimens were oven-dried and then saturated with water under vacuum. Other samples [12 (R)  $\times$ 1 (L)  $\times$ 0.4 (T) cm] were prepared to examine the effects of the temperature gradient on stress relaxation, and additional samples of the same size and shape were heated in water to investigate the effects of heating.

#### Apparatus and test conditions

Figure 1 shows the apparatus used for measuring stress relaxation. Test samples were given an initial deflection by moving upward a load cell that was connected to samples with wire and installed in the axis of a mobile system; the deflections were observed with a laser displacement meter. The loads were measured with a load cell, and the rates of elevation and reduction of temperature were controlled by a temperature controller (model SU; Chino, Japan). Test samples were supported at two points 8cm apart, and the initial deflection was applied to the center of the span. Stress relaxation was measured in water.



**Fig. 1.** Apparatus for stress relaxation measurements during bending. A, digital recorder; B, recorder; C, amplifier; D, laser deformation meter; E, load cell; F, thermometer; G, temperature control sensor; H, heater; J, support stand of sample; S, test sample; L, cooler; M, programmable temperature controller; K, fan; N, waterbath

Measurement of stress relaxation

Six temperatures  $(20^\circ, 40^\circ, 50^\circ, 60^\circ, 70^\circ, 80^\circ C)$  were chosen for measurements of stress relaxation at constant temperature. Stress relaxation during the processes of elevating and lowering temperature was measured under five temperature ranges:  $20^\circ-40^\circ C$ ,  $20^\circ-60^\circ C$ ,  $20^\circ-80^\circ C$ ,  $40^\circ-60^\circ C$ ,  $60^\circ-80^\circ C$ . The initial deflection was mainly 1 mm followed by deflections of 2 and 3 mm. Stress relaxation was measured for 10 h. For the initial 4 h relaxation was measured at the initial constant temperature, then for 4 h with elevating or lowering the temperature, and for a final 2 h at the final temperature. The effects of temperature elevation rate and of preheating samples on stress relaxation were also investigated. Elevation rates of  $0.25^\circ C/min$  and  $1.00^\circ C/min$  were selected; and some samples preheated at  $80^\circ C$  for about 30 min were slowly cooled to room temperature.

Measurement of residual deflections after stress relaxation measurement

Immediately after relaxation measurements the test samples were unloaded, and the residual deflections were measured at 6 and 12h after unloading. The amount of sets was estimated as the ratio of the residual deflection to the initial applied deflection.

## **Results and discussion**

Stress relaxation at constant temperature and during temperature elevation and reduction

Figure 2 shows stress relaxation curves under four temperature conditions. A and B show relaxation curves at constant



**Fig. 2.** Stress relaxation curves at constant temperatures of  $20^{\circ}$ C (*A*) and  $80^{\circ}$ C (*B*) and during the processes of temperature elevation from  $20^{\circ}$ C to  $80^{\circ}$ C (*C*) and temperature reduction from  $80^{\circ}$ C to  $20^{\circ}$ C (*D*). For curves *C* and *D* the temperature was maintained at  $20^{\circ}$ C or  $80^{\circ}$ C for the initial 240 min, followed by elevation and reduction of temperature until 480 minutes, and maintained at the final temperatures from 480 to 600 minutes, respectively

temperatures of 20°C and 80°C, respectively. C shows the relaxation curve during the process of temperature elevation from 20°C to 80°C; and D shows it during temperature reduction from 80°C to 20°C. These curves varied markedly with temperature conditions. During the period of temperature elevation from 240 to 420 min (C), the relaxation modulus  $(E_1)$  decreased markedly. On the other hand, during the process of lowering temperature (D),  $E_t$  was nearly constant despite the reduction in temperature. These results indicated that the stress relaxation behavior was fundamentally different for elevating and lowering temperature. Furthermore, the total decrease in  $E_t$  during the process of temperature elevation from 20°C to 80°C was almost equivalent to the difference of  $E_{\rm t}$  at 600 min after beginning the measurement at constant temperatures of 20°C and 80°C.

Figure 3 shows the relations between the differences in  $E_t$  at 20°C (A) and at some higher temperatures (40°, 60°, 80°C) (B) at 600 min and the difference between A and the final  $E_t$  during the process of temperature elevation from 20°C to the higher temperature (B'). All the plots in Fig. 3 are almost linear, with a slope of 45°. This indicates that the relaxation moduli during the process of temperature elevation from 20°C to a specific higher temperature were almost equivalent to those at the higher temperature 600min after beginning the relaxation measurements.

In previous studies<sup>7-11</sup> creep deformations during the process of temperature elevation were always larger than those at any constant temperature over the range of temperatures used.

Kitahara and Yukawa<sup>7</sup> reported that creep deformations were larger during the process of temperature elevation than at constant temperature and attributed this result to



**Fig. 3.** Comparison of the differences in total relaxation under two constant temperatures and increases in relaxation during temperature elevation. *A*, relaxation modulus at 600 min under a constant temperature of  $20^{\circ}$ C; *B*, relaxation modulus at 600 min under constant temperatures of  $40^{\circ}$ ,  $60^{\circ}$ , and  $80^{\circ}$ C; *B'*, relaxation moduli at 600 min after beginning of measurements during temperature elevation from  $20^{\circ}$ C to  $80^{\circ}$ C. Figures in parentheses show the initial deflections in millimeters

the temperature gradient in the test specimens. However, a temperature gradient in wood cannot lead to more creep deformation; rather, it should decrease the deformation because the temperature of the inner part of the wood specimens during temperature elevation is lower than the environmental temperature at which the sample would equilibrate at a constant temperature. Furthermore, they discussed their results without considering an increase in instantaneous deformations (elastic deformations) with elevating temperature.<sup>7</sup> That is, instantaneous deformations increase with temperature elevation because the deformations during the process of temperature elevation include elastic deformation, which increases with temperature. As elastic deformations increase with temperature elevation, the creep deformation during this process includes a component of the increase in elastic deformation. However, creep deformation at constant temperature does not include elastic deformation.

Arima<sup>8</sup> showed that creep deformations during the process of temperature elevation were larger than those at constant temperatures above 60°C, and the difference between them diminished if wood samples were preheated. Based on these results, he inferred that the larger creep deformations during temperature elevation were attributable to growth stress and the residual stress in wood due to the preliminary history.

The previous studies described above indicated that creep deformations during the process of temperature elevation are apparently larger than those under conditions of constant temperature. However, this does not neccessarily mean that true creep deformations (fluidity) of wood during the process of temperature elevation are larger than those at constant temperature. Indeed, the results of the present study show in Fig. 3 that the relaxation moduli during temperature elevation were almost equivalent to those at constant temperatures after a sufficient relaxation time. This indicated that the fluidity of wood at a specific temperature during the process of temperature elevation is similar to that at the same constant temperature.

Figure 4 shows the changes in behavior of  $E_t$  with time during the process of temperature elevation at different rates, and Fig. 5 shows the differences in  $E_{\rm r}$  of untreated and preheated samples during temperature elevation. The equilibrium values of  $E_t$  were independent of the rate of temperature elevation and of preheating. In addition, Fig. 5 indicates that the relaxation of the preheated samples was greater than that of the untreated samples during a period at a constant temperature from 0 to 240 min, although the total relaxations during the whole process were similar to each other. This suggested that the inner structure of the cell wall changed to an unstable structure as a result of cooling after the preheating treatment. Consequently, the relaxation of preheated samples was greater than that of untreated samples during the initial period at constant temperature owing to the unstable cell wall structure. During the process of temperature elevation after this period, relaxation of the pretreated samples was smaller than that of the unheated samples because the preheated samples retained the structure at higher temperatures to some extent.



Fig. 4. Stress relaxation curves during the process of temperature elevation from 20°C to 80°C with different rates



Fig. 5. Stress relaxation curves of samples untreated and preheated in water at  $80^{\circ}$ C during the process of temperature elevation from  $20^{\circ}$ C to  $80^{\circ}$ C. Stress relaxations were measured at  $20^{\circ}$ C for the initial 240 min and then were subjected to elevation or reduction of temperature until 480 min, then maintained at  $80^{\circ}$ C from 480 to 600 min

Thus, we concluded that relaxation at a certain temperature during the process of temperature elevation is similar to that at the same constant temperature, regardless of the rate of increase in temperature, the temperature range measured, or the preheating of the sample.

It is necessary to discuss the inconsistencies between previous results<sup>7-11</sup> and those of the present study. The large creep deformations during the process of elevating the temperature obtained in the previous studies were attributable to the increase in instantaneous deformation (elastic deformations) with elevating temperature and insufficient accuracy of the instantaneous deformations. Elastic deformation increases with increasing temperature, and therefore creep deformation during temperature elevation includes a component of increases in elastic deformation with increasing temperature. On the other hand, creep de-



Fig. 6. Stress relaxation moduli during various processes of temperature elevation and reduction

formation at constant temperatures does not include these elastic deformations. It has been suggested that the temperature gradient in wood samples is responsible for the large creep deformations. However, as described above, the temperature gradient itself in samples during the temperature elevation process cannot cause a large creep deformation.

Stamm<sup>12</sup> reported that the fiber saturation point (FSP) of wood decreased by about 0.1% with elevation of temperature by 1°C, and thus a decrease in FSP accompanied by temperature elevation may cause large creep deformation in a manner similar to that under a nonequilibrium state of moisture.<sup>1-3</sup> However, it has recently been suggested that decreases in FSP with temperature elevation are far smaller based on the observation of only a small decrease in swelling of wood in water with temperature elevation.<sup>13,14</sup> Ishikawa<sup>15</sup> also found that there was a tendency toward an increase in FSP with temperature elevation at temperatures above 100°C. Based on these results, the decrease in FSP with elevation of temperature can be considered to be so small that a large increase in creep deformation cannot occur as a result.

Figure 6 shows stress relaxation curves during elevation and lowering of temperature as a function of temperature. The relaxation modulus  $E_t$  decreased markedly during the process of temperature elevation. On the other hand, during the processes of temperature reduction  $E_t$  remained almost constant, regardless of the temperature range measured. These observations indicated that the relaxation behaviors during both processes are quite different.

The larger decrease in  $E_t$  during temperature elevation can be considered to be due to a decrease in elasticity with elevation of temperature. On the other hand, relaxation  $(E_t)$  during the process of temperature reduction remained almost constant, possibly as a result of increases in fluidity at the same temperature during temperature reduction in comparison to the process of temperature elevation. In this

context, the results shown in Fig. 5 indicate that the relaxation of the preheated samples was larger than that of untreated samples during the initial period at constant temperature because of the unstable structure in the preheated samples. This suggests that the fluidity of wood at a specific temperature during the process of temperature reduction is higher than that at the same constant temperature on the basis of its unstable state caused by cooling. Therefore, relaxation behavior during the process of temperature reduction should not be considered to be regulated only by decreases in fluidity. Relaxation during the process of temperature reduction may occur as a result of a decrease in elasticity relative to that at constant temperature on the basis of the unstable structure in wood caused by cooling. Thus, the mechanism of relaxation during the process of temperature reduction cannot be discussed further at present.

However, relaxation behavior during the process of temperature reduction is thought to be intimately related to the cooling set that occurs during the process. The residual deflections after relaxation measurement are described in the next section.

#### Set after stress relaxation

Figure 7 shows residual deflections (sets) after relaxation measurements under various temperature conditions. They were calculated as the ratio of residual deflection to initially applied deflection.

Larger sets occurred during the process of temperature reduction ( $80^{\circ}$ C to  $20^{\circ}$ C) than during the process of temperature elevation ( $20^{\circ}$ C to  $80^{\circ}$ C) or at the highest constant temperature ( $80^{\circ}$ C) in the temperature range examined. Moreover, the set that occurred at  $80^{\circ}$ C diminished with time left in water at that temperature. Recoveries of the sets that occurred during relaxation measurements with temperature elevation from  $20^{\circ}$ C to  $80^{\circ}$ C and at a constant temperature of  $80^{\circ}$ C by heat treatment in water are shown



Fig. 7. Set occurred after relaxation measurement.  $80-20^{\circ}C$ , set during lowering of temperature from  $80^{\circ}$ C to  $20^{\circ}$ C;  $20-80^{\circ}$ C, set during elevation of temperature from  $20^{\circ}$ C to  $80^{\circ}$ C;  $80^{\circ}$ C, set immediately after measurement at  $80^{\circ}$ C;  $80^{\circ}$ C (*6hr.*), residual set when samples were kept in water at  $80^{\circ}$ C for 6h after measurement at  $80^{\circ}$ C;  $80^{\circ}$ C (*12 hr.*), Residual set when samples were kept in water at  $80^{\circ}$ C for 12h after measurement at  $80^{\circ}$ C

in Fig. 8. The sets after heat treatment were similar with each treatment, although a set that occurred during the process of temperature elevation was much larger than that occurring at a constant temperature of 80°C. These results indicated that marked sets did not occur during the process of temperature elevation but occurred to a larger extent during the lowering of temperature, and that these sets could be significantly recovered by heating in water.

Based on the result of sets and the differences between stress relaxation behavior during the processes of temperature elevation and reduction, the larger relaxation during temperature elevation was suggested to occur as the result of the decrease in elasticity and an increase in fluidity with temperature elevation. On the other hand, the relaxation behavior found during the process of temperature reduction ( $E_t$  remained almost constant) was probably caused by decreases in the mobility of the molecules of wood constituents with decreasing temperature and secondary bonding between the molecules in the strained state. Thus, a greater set also occurred during the process of temperature reduction. However, as mentioned above, a decrease in elasticity due to the unstable structure caused by cooling possibly relate to the relaxation behavior during the process of temperature reduction. The mechanism of stress relaxation during elevation and lowering of temperature will be discussed in the next paper using a viscoelastic model in which elastic and viscous elements are given physical meanings.

#### Conclusions

The stress relaxation of water-saturated wood in the radial direction was measured at various temperatures and during the processes of temperature elevation and reduction in the range 20°–80°C. Sets after the relaxation measurements were also determined. The extent of stress relaxation at a specific temperature during the process of temperature



Fig. 8. Amounts of set after stress relaxation measurement (*filled bars*) and residual set after heating in water (*open bars*). Error bars show standard deviations

elevation was quite similar to the difference of relaxations at the two constant temperatures, differing from the conclusions of the previous studies. The relaxation moduli remained constant during temperature reduction, although extensive decreases were observed during the process of temperature elevation. Only slight sets occurred during the process of temperature elevation. However, sets occurred to a greater extent during temperature reduction and could be markedly recovered by heating in water.

The greater relaxation during the process of temperature elevation was attributable to a decrease in elasticity and increase in fluidity with temperature elevation. On the other hand, the relaxation behavior during the process of temperature reduction (i.e.,  $E_t$  remained almost constant) was probably caused by increases in the mobility of the molecules of wood constituents with decreasing temperature and secondary bonding between the molecules in the strained state.

# References

- Armstrong LD, Christensen GN (1961) Effect of moisture changes on creep in wood. Nature 191:869–870
- Gibson EJ (1965) Creep of wood: role of water and effect of changing moisture content. Nature 206:213–215
- Hearman RFS, Paton JM (1964) Moisture content changes and creep of wood. For Prod J 14:357–359
- 4. Hisada T (1979) Creep and set behavior of wood relating to kiln drying. II. Effects of stress level on tensile creep of

wood during drying (in japanese). Mokuzai Gakkaishi 25:697--706

- Mukudai J, Yata S (1986) Modeling and simulation of viscoelastic behavior (tensile strain) of wood under moisture content. Wood Sci Technol 21:49–63
- Mukudai J, Yata S (1988) Verification of Mukudai's mechanosorptive model. Wood Sci Technol 22:43–58
- Kitahara K, Yukawa K (1964) The influence of the change of temperature on creep in bending (in japanese). Mokuzai Gakkaishi 10:169–175
- Arima T (1972) Creep in process of temperature changes. I. Creep in process of constant, elevated and decreased temperature (in japanese). Mokuzai Gakkaishi 18:349–353
- Arima T (1972) Creep in process of temperature changes II. History effect on creep of wood in process of temperature changes (in japanese). Mokuzai Gakkaishi 18:377–380
- Arima T (1973) Creep in process of temperature changes: prediction of creep curves in process of elevated temperature (in japanese). Mokuzai Gakkaishi 19:75–79
- Shniewind A (1966) On the influence of moisture content changes on the creep of beech wood perpendicular to the grain including the effects of temperature and temperature changes. Holz Rof Werkstoff 24:87–98
- Stamm AJ (1964) Wood and cellulose science. Ronald Press, New York, pp 145–149
- Ishimaru Y (1999) Wood and water during the temperature range below 100°C. In: Proceedings of the symposium for the study of rheological properties of wood of the Japan Wood Research Society (in japanese), pp 1–4
- 14. Ishimaru Y, Narimoto S, Iida I (2000) Mechanical properties of wood swollen in organic liquids having two or more functional groups for hydrogen bonding in a molecule. J Wood Sci 47: 171–177
- 15. Ishikawa A (1999) Wood moisture content under the high temperature by the steaming treatment. In: Proceedings of the symposium for the study of rheological properties of wood of the Japan Wood Research Society (in japanese), pp 32–37