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Yoichi Kojima · Hiroyuki Yamamoto

Effect of moisture content on the longitudinal tensile creep behavior of wood

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Abstract In our previous report, we investigated the effect of the microfibril angle (MFA) in the middle layer of the secondary wall (S_2) on the longitudinal creep behavior of a thin homogeneous earlywood specimen sugi. In the present study, we investigated the role of moisture on the tensile creep behavior of wood. We discuss the creep behavior of the wood cell wall from the viewpoint of the composite structure of the cell wall and the properties of the constituent materials. A microtomed thin specimen of earlywood of sugi (Cryptomeria japonica D.Don) was used for the longitudinal tensile creep test. Creep tests were conducted at three moisture stages (oven-dry, air-dry, fiber saturation point) over a broad range of MFA. Results showed that the longitudinal tensile creep behavior was highly dependent on both the moisture content and the MFA. With a small MFA, the variation in the creep function among the three moisture states was very small. For a large MFA, the variation in the creep function was larger. At low moisture contents, the magnitude of the creep function was very small, while at high moisture content, it was very large except for the case of specimens with very small MFA. Those results show that the longitudinal tensile creep behavior was directly affected by the fine composite structure and the internal properties of the cell wall constituents.

Key words Viscoelastic behavior \cdot Tensile creep \cdot Wood cell wall \cdot Moisture content \cdot Mechanical property

Introduction

To clarify the origin of the viscoelastic properties of wood from the viewpoint of the cell wall fine structure, it is neces-

Y. Kojima · H. Yamamoto (⊠) School of Bioagricultural Sciences, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8601, Japan Tel. +81-52-789-4152; Fax +81-52-789-4150 e-mail: hiro@agr.nagoya-u.ac.jp sary to examine the longitudinal tensile test using a homogeneous clear specimen. This is because it is considered that the mechanical properties determined from a longitudinal tensile test would reflect the fine structure of the cell wall directly.¹⁻³

In our previous report, we discussed the relationship between longitudinal tensile creep behavior and the microfibril angle (MFA) of the middle layer of the secondary wall (S_2) using the results of the tensile creep test with small specimens of sugi (*Cryptomeria japonica* D.Don), and simulations using a simplified viscoelastic model.⁴ These revealed that the longitudinal tensile creep behavior is highly dependent on MFA. That is, the magnitude of the longitudinal creep function tended to increase with MFA. This shows that longitudinal tensile creep behavior is strongly affected by the composite structure of the cell wall, that is, the two-phase structure of the wall layer comprising the rigid framework of cellulose microfibril (CMF) and the matrix of lignin–hemicellulose compounds.

It is well known that the physical properties of wood, e.g., the elastic modulus, the anisotropic shrinkage, heat conductivity, and others, are affected by the moisture in the cell wall.⁵ Concerning the relationship between the viscoelastic behavior and the moisture content in the cell wall, many reports refer to bending or compression tests, especially in relation to the mechanosorptive creep properties.⁶⁻⁹ It is also reasonable to expect that the moisture gives a certain influence on the longitudinal tensile viscoelastic behavior in the steady moisture condition; however, no positive demonstration has yet been obtained. In our previous report, the tensile creep tests were conducted when the moisture content was around the fiber saturation point (FSP),⁴ and it is challenging to know whether the obtained results would stand over various moisture contents.

The aim of this study is to clarify the longitudinal tensile creep behavior of wood, especially in relation to the moisture content in the steady moisture condition, over a broad range of the MFA, using a thin specimen of sugi earlywood. Moreover, we discuss the effect of the moisture content on the viscoelastic behavior of the wood cell wall constituents using a simplified viscoelastic model consisting of three parameters, involving a Voigt element (two parameters) and an independent spring in series.

Material and methods

Material

A 40-year-old, 20-cm-diameter at breast height (DBH) sugi (*Cryptomeria japonica* D.Don) growing in the Nagoya University experimental forest, was studied. Blocks were cut from the sapwood of the vertical stem, some from juvenile wood. After cutting, the blocks were boiled in hot water for 10min to saturate them. Then, we focused on the earlywood region of one annual ring in one block. Four homogeneous tangential sections ($70 \times 8 \times 0.2$ mm in the L, T, R directions) were prepared using a sliding microtome, and were used for both the tensile test and to measure the MFA. An earlywood specimen that was used to measure oven-dried density was cut from the same annual rings as the specimen used for the creep test.

The longitudinal tensile creep test

In general, the strain gauge method is used to detect the strain of deformation induced in a tensile test. In this study, the specimens were very thin (0.2mm). As we reported previously, a specimen must be at least 0.6mm thick to eliminate the effects of the rigidity of the strain gauge and the glue when the strain gauge method is used to detect mechanical strain.⁴ Because our specimens were 0.2 mm thick, the strain gauge method was not suitable for detecting creep strain. To detect creep strain, we used a traveling microscope with an xy-microstage to directly measure the distance between two dots marked on the grain. To prevent slippage at the clamps during the creep test, pieces of sandpaper were attached to each edge of the specimen using quick-drying glue (CC-33A, Kyowa). Then, the specimen was put in a small air-conditioned cabinet. In this study, the moisture content of the specimen was controlled at 100%, 76%, and 0% relative humidity (RH), at 20°C, using H_2O , aqueous NaCl, and P₂O₅ powder, respectively. The moisture contents of the specimens were conditioned to ovendry (MC2%), air-dry (MC15%), and the FSP (MC25%) at 20°C. The creep test was performed in an airtight chamber at 20°C, in which the air was circulated using a microelectric fan, and the RH was controlled using the three materials mentioned above. After the specimen had been conditioned in the cabinet for a few days, it was attached to a hand-made tensile testing machine in the chamber. Two spots 20-30mm apart along the grain were marked with black paint to measure the displacement caused by the tensile load. To measure the distance between the two dots, a traveling microscope with an xy-microstage (0.001-mm accuracy) was used. A load of 14.2 MPa, corresponding to the proportional limit of wet sugi earlywood, was applied to the specimen as



Fig. 1. A simplified viscoelastic model of wood for simulating the longitudinal creep behavior. Spring 1 and spring 2 have elastic moduli E_1 and E_2 , respectively, and dashpot2 has viscosity coefficient η_2

a dead load. The initial displacement was measured soon after applying the load and was considered as the instantaneous displacement. Then, the displacement was recorded every few hours for several hundred hours. After the creep test had been completed, a time-creep compliance curve was plotted. Creep tests were performed for a broad range of MFAs.

Determining microfibril angle

After the creep test had been performed, the MFA in the S_2 layer of each specimen was measured using an X-ray diffractometer (XD-D1w, Shimadzu).¹⁰ The MFA was the same as that of the specimen prepared for MFA measurement.

Measuring wood density

The wood density was determined using a gravimetric method with mercury impregnation. The density of each specimen was determined after the prepared specimen had reached a constant weight in an oven at 105°C for 24h. To measure the oven-dried volume, the mercury displacement technique was used.

Simulating the creep properties

A simplified model explaining the longitudinal creep properties is shown in Fig. 1. The model consists of an independent spring (spring 1) and a Voigt element (spring 2 and dashpot2) in series. The elastic moduli of spring 1 and spring 2, and the viscosity coefficient of dashpot2 are denoted by E_1 , E_2 , and η_2 , respectively. Based on this creep model, the following equation was derived:⁴

$$J^{W}(t) = \frac{1}{E_{1}^{W}} + \frac{1}{E_{2}^{W}} \left(1 - e^{-t/\lambda_{2}^{W}}\right), \quad \lambda_{2}^{W} = \lambda_{2} = \frac{\eta_{2}}{E_{2}}$$
(1)

where $J^{W}(t)$, t, E_{1}^{W} , E_{2}^{W} , λ_{2}^{W} , and η_{2}^{W} stand for the substantial creep compliance of the cell wall in the longitudinal direction, time, the substantial elastic modulus of spring 1 and spring 2, the retardation time, and the substantial viscoelastic coefficient of dashpot2, respectively.

We tried to determine the values of the unknown parameters $(E_1^{W}, E_2^{W}, \eta_2^{W}, \lambda_2^{W})$ to simulate the experimental results quantitatively. Finally, we discuss the physical meaning of the estimated values.

Results and discussion

Longitudinal tensile creep properties

The mechanical properties of wood are affected by its density, as is the viscoelastic property. To understand the viscoelastic behavior of wood as a cell wall property, it is necessary to transform J(t) into a substantial value by considering the effect of the density of the specimen. To transform the longitudinal Young's modulus of wood, $E_{\rm L}$, into the substantial Young's modulus of the wood cell wall, $E_{\rm L}^{\rm W}$, we can use the following formula:

$$E_{\rm L}^{\rm W} = E_{\rm L} \frac{\rho^{\rm W}}{\rho_0},$$

where ρ^{W} is the density of the cell wall, and ρ_{0} is that of oven-dried wood. For linear viscoelastic materials, the substantial creep compliance of the cell wall in the longitudinal direction, $J^{W}(t)$, is calculated as follows:

$$J^{\mathrm{w}}(t) = J(t)\frac{\rho_0}{\rho^{\mathrm{w}}}.$$

Figure 2 shows the time dependency of the substantial creep compliance of the cell wall $J^{W}(t)$ in relation to the MFA. The three curves in each part of the figure show the results for the three moisture conditions (oven-dry, air-dry, FSP). As Fig. 2 shows, the instantaneous compliance appeared immediately after the load was applied. Subsequently, the total creep compliance increased with time and peaked after several hundred hours. These observations concur with various experimental reports on bending creep properties under steady moisture conditions using wood beams.^{11,12} Because the instantaneous compliance (which equals instantaneous deformation) depends strongly on the MFA and moisture content, it is difficult to discuss the effects of the MFA and moisture content on the creep behavior using the results shown in Fig. 2. Therefore, we need to calculate the substantial creep function of the cell wall, $\varphi^{W}(t)$, which is calculated by subtracting the initial instantaneous compliance from $J^{W}(t)$ using the following expression:

$$\varphi^{\mathrm{w}}(t) = J^{\mathrm{w}}(t) - \frac{1}{E_1^{\mathrm{w}}}.$$

Figure 3 shows the substantial creep function of the cell wall, $\varphi^{W}(t)$. First, we will discuss the relationship between the moisture content and the longitudinal tensile creep behavior. It is clear that the moisture content affects the variation in the creep function, as shown in Fig. 3. In the low moisture content region (oven-dry), the variation in the



Fig. 2. The moisture dependencies of the substantial creep compliance of the cell wall in the longitudinal direction $J^{W}(t)$

creep function was smaller than at other moisture contents. As the moisture content increased, the variation in the creep function also increased. That is, the longitudinal tensile creep behavior was clearly dependent on the moisture content. Furthermore, the dependency of the moisture content was remarkable in the specimen with a large MFA, while in the specimen with the smallest MFA (12.0°), the dependency on the moisture content was not clear. This is because longitudinal tensile creep behavior strongly reflects the properties of the wood cell wall constituents. Because the longitudinal tensile creep behavior for a small MFA was affected by CMFs, which may have little affinity to moisture, the variation in the creep function was very small, even



Fig. 3. The moisture dependencies of the substantial creep function of the cell wall in the longitudinal direction $\phi^{W}(t)$

if the moisture content changed. Conversely, for a large MFA, because the matrix substance in the cell wall, which may strongly attract moisture, affected the longitudinal tensile creep behavior, the variation in the creep function was more distinct when the moisture content changed. Navi et al.¹ discussed the longitudinal tensile mechanosorptive creep behavior. When we calculated the creep function from the part of the steady moisture state in the mechanosorptive behavior revealed by Navi et al., the result nearly corresponded to our measurement result.

Next, we discuss the relationship between the MFA and the longitudinal tensile creep behavior. Figure 4 shows the relationship between the MFA and the substantial creep



Fig. 4. The microfibril angle (MFA) dependencies of the substantial creep function in the longitudinal direction $\phi^{W}(t)$. *Open triangles*, MFA 12.0°; *filled triangles*, MFA 20.4°; *open circles*, MFA 29.8°; *filled circles*, MFA 44.1°

function of the cell wall. In Fig. 4, it is clear that the variation in the creep function was very small in specimens with small MFA. As MFA increased, the variation became larger. This shows that the longitudinal tensile creep properties of wood are highly dependent on the MFA. This is because the viscoelastic behavior in response to the longitudinal tensile load directly reflects the properties of the cell wall constituents and their arrangement in the cell wall. In the specimens with a small MFA, because the longitudinal creep deformation is affected by the CMF, which is much more viscous than the matrix substance in the cell wall constituents, the variation in the creep function becomes very small. By contrast, at large MFA, behavior of the matrix substance in the cell wall becomes more clear, because the mechanical contribution of the CMF decreases. Because the matrix substance is less viscous than the CMF, the

creep function becomes several times larger than at a small MFA.

Navi et al.¹ investigated the effect of the transient moisture on the longitudinal tensile creep behavior of spruce wood portion. From their results regarding the longitudinal creep deformation, one can estimate the longitudinal creep function of wood portion under the steady moisture condition in the air-dried state, which gave a similar magnitude to that obtained in the present investigation using a sugi specimen with a small MFA.

Simulated results using the simplified viscoelastic model

The observed $J^{W}(t)$ was simulated using the simplified viscoelastic model formulated as Eq. 1. It is guite natural to consider the values of the parameters in the simulation to reflect intrinsic information about the composite structure of the cell wall and internal properties of its constituents. Figure 5 shows the dependency of the fitted values of E_1^{W} , E_2^{W}, η_2^{W} , and λ_2^{W} on the MFA. It is obvious that E_1^{W} stands for the substantial Young's modulus of wood cell wall. As seen in Fig. 5, the value of E_1^W decreased concavely as MFA increased, which quantitatively concurs with previous results using clear wood specimens or isolated wood fiber.¹³⁻¹⁶ The dependencies of the simulated values of E_2^{W} and η_2^{W} on the MFA were greater than that of E_1^{W} , as shown in Fig. 5. The values of E_2^{W} and η_2^{W} tended to be small when the MFA exceeded 20°, and became very large in the region of smaller MFAs. The values of $E_2^{\rm W}$ and $\eta_2^{\rm W}$ increased as the moisture content decreased in the region where the MFA exceeded 20°, although the values were smallest for the oven-dried specimens with the smallest MFA. The reason for this is not clear from this simplified viscoelastic model. The value of E_2^{W} is an indicator of the smallness of the creep deformation. The value of E_2^{W} in the region of small MFA is much larger than that in the large MFA region. In the region of small MFAs, the longitudinal creep deformation is affected by the CMF, which is much more viscous than the matrix substance in the cell wall constituents. Conversely, in the region of large MFAs, the longitudinal creep deformation is affected by the matrix substance, which is much less viscous than the CMF. The values of $\eta_2^{\rm w}$ and $\lambda_2^{\rm w}$ are indicators of the smallness of the creep rate. The value of η_2^{W} is clearly dependent on the MFA. In the small MFA region, the value of η_2^{W} is much larger than that in the large MFA region, because the creep rate is reduced by the rigid CMF. Conversely, the value of η_2^{W} decreases as MFA increases, because the longitudinal creep deformation is markedly affected by the matrix substance in the region of large MFAs. The value of λ_2^{W} is not clearly dependent on the MFA. This can be explained as follows. The value of λ_2^{W} is calculated as the ratio of η_2^{W} and E_2^{W} . The MFA dependency of η_2^{W} was similar to that of E_2^{W} . Therefore, the value of λ_2^{W} is roughly constant regardless of the MFA.

Thus, the parameters are highly dependent on the MFA because tensile creep deformation is possibly affected by the arrangement of the cell wall constituents, namely the CMF framework and matrix substance. To discuss this idea



Fig. 5. The MFA dependencies of calculated values of the parameters in the simplified model

more quantitatively, we must construct a realistic wood fiber model having a multilayered cell wall that considers the composite and fine structures of the cell wall lamella.

Conclusions

This study examined the dependence of the longitudinal tensile creep behavior of thin specimens of sugi on the moisture content in relation to the MFA. We found that the longitudinal tensile creep behavior is highly dependent on both the moisture content and the MFA. In specimens with a large MFA, there was a large difference in the variation of the creep function among the three moisture content states. In the specimen with a small MFA, there was very little difference in the creep function among the three moisture content states, because the creep deformation in the specimen with a small MFA is affected by the hydrophobic and very viscous CMF. Conversely, in the specimen with a large MFA, the creep deformation is affected by the hydrophilic matrix substance, which is much less viscous than the CMF. This is why the longitudinal tensile creep behavior and the other mechanical properties are directly affected by the behavior of the cell wall constituents and their arrangement in the cell wall.

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