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ORIGINAL ARTICLE

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Effect of microfibril angle on the longitudinal tensile creep behavior of wood

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Abstract In this report, we undertook studies of the viscoelastic properties of wood from the viewpoint of the fine structure and properties of the constituent materials in the wood cell wall. To measure the mechanical properties of the wood as the behavior of the cell wall, it is required to perform the longitudinal tensile test using a homogeneous specimen. In this study, microtomed specimens of sugi (*Cryptomeria japonica* D.Don) earlywood were used for the creep test, which were conducted at the fiber saturation point. The substantial creep compliance of the cell wall was simulated using a simplified viscoelastic model consisting of a Voigt element and an independent spring in series. Based on the experimental results, the values of the parameters were optimized. The results were as follows: (1) the longitudinal tensile creep deformation tends to increase with the elapsed time, similar to the bending creep behavior; (2) the magnitude of the longitudinal creep function increases with MFA; and (3) each parameter in the simplified viscoelastic model is markedly affected by the MFA. Based on these results, the mechanism of the longitudinal tensile creep deformation of wood is discussed.

Key words Viscoelastic behavior · Tensile creep · Wood cell wall · Microfibril angle · Mechanical property

Introduction

It is very important to clarify the viscoelastic properties of the wood cell wall in order to use forest products as structural members in buildings or furniture. Based on such a practical background, studies of the viscoelastic properties of wood began on a large scale in the 1960s, and included studies of mechano-sorptive creep and stress relaxation.[1,2] Subsequently, there have been many reports on the viscoelastic properties of wood.[3–13] Most of these reports involved bending or compression tests using wood beams because the results using wood beams are practically important for building or furniture engineering. By contrast, there are few reports of longitudinal tensile tests.[13] This is because the structural member for building is usually subjected not to tensile force but to bending or compressive load. Furthermore, the viscoelastic measurements required for the longitudinal tensile tests take a very long time to perform, and it is difficult to detect small deformations caused by the longitudinal tensile load.

To understand and explain the origin of the viscoelastic properties of the wood, it is necessary to comprehend the viscoelastic properties of wood from the viewpoint of the fine structures and properties of the constituent materials in the wood cell wall. It is considered that the mechanical properties determined from a longitudinal tensile test using a homogeneous specimen would reflect the fine structure of the cell wall.[13,14] Therefore, it is necessary to measure the longitudinal viscoelastic properties of wood using a thin specimen which is approximately regarded as a single wood fiber.

Among the fine structures of the wood cell wall, it is likely that the microfibril angle (MFA) of the middle layer of the secondary wall (S2) plays a very important role in controlling various mechanical properties of wood and the wood cell wall, e.g., the longitudinal growth stress, the drying shrinkage, and the Young's modulus of the wood.[14–16] It is reasonable to expect that the MFA gives a certain influence on the viscoelastic properties of wood, however, no positive verification has yet been obtained.

This study examined the longitudinal tensile creep properties of wood, especially in relation to the MFA, using a thin specimen of sugi (*Cryptomeria japonica* D.Don) earlywood. We adopted a simplified viscoelastic model consisting of three parameters, that is, a Voigt element and an independent spring in series, and evaluated their values by simulating the experimental results. The physical meanings

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indicated by the parameters are discussed especially in relation to the MFA.

Materials and methods

Materials

A 40-year-old, 20-cm-DBH sugi (*Cryptomeria japonica* D.Don) growing in Nagoya University experimental forest, was studied. Blocks were cut from the sapwood of the vertical stem. Some blocks were cut from the juvenile wood. After cutting the blocks, they were boiled in hot water for 10 min to saturate them. Then, homogeneous tangential sections (70 × 8 × 0.2 mm in L × T × R directions) were prepared from the earlywood region of each block using a sliding microtome. The sections were then used for the tensile test. An earlywood specimen used to measure the oven-dried density was cut from the same annual rings as the specimens used for the creep test.

Determining the creep strain

In general, the strain gauge method is used to detect the strain of deformation induced by the tensile test. The effect of the thickness of the earlywood specimen prepared from the sapwood region (70 × 8 × 0.2–0.8 mm in L × T × R directions) on the apparent longitudinal Young's modulus was measured using the strain gauge method with wet specimens. The mean value of the MFA of the specimen was 15.5° (SD 1.22, $n = 4$). The result is shown in Fig. 1, which illustrates that the apparent Young's modulus is larger in specimens that are 0.2–0.5 mm thick than in those that are over 0.6 mm thick. This suggests that the effects of the rigidity of the strain gauge and the quick-drying glue become manifest in specimens less than 0.5 mm thick. This means that specimens must be at least 0.6 mm thick to eliminate the effect of the rigidity of the strain gauge and glue when the strain gauge method is used to detect the mechanical strain. In this study, homogeneous flat-sawn specimens of earlywood were used for the creep test. Because the specimens were often less than 0.5 mm thick, it is improper to adopt the strain gauge method to detect the creep strain. Therefore, we measured the distance between two dots marked on the grain using a traveling microscope with an xy-microstage to directly observe the creep strain.

Determining the creep load

The shape of the stress–strain curve was investigated to determine the proportional limit. The proportional limit was 12–15 MPa (average 14.0 MPa) regardless of the thickness of the specimen, as shown in Fig. 2. Therefore, a load of 14.2 MPa, corresponding to the proportional limit of the normal earlywood specimen, was used for the creep load.

Longitudinal tensile creep test

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. The moisture content of the specimen was controlled using water at 100% relative humidity (RH) at 20°C. The creep test was performed in an airtight chamber, in which the air was circulated by a microelectric fan and RH was controlled with water at 20°C. After the specimen was conditioned in the cabinet for a few days, it was attached to a handmade tensile testing machine in the chamber. Two spots, 20–30 mm 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

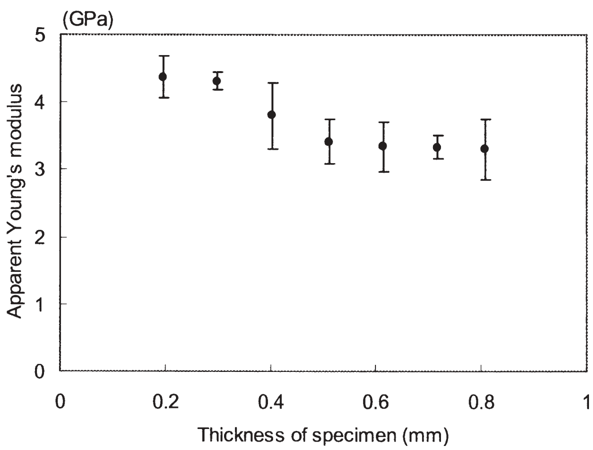

Fig. 1. The relationship between the thickness of the sugi earlywood specimen and the apparent longitudinal Young's modulus measured using the strain gauge method. Each point represents average values, the error bar shows the standard deviation. All tests were conducted under wet conditions

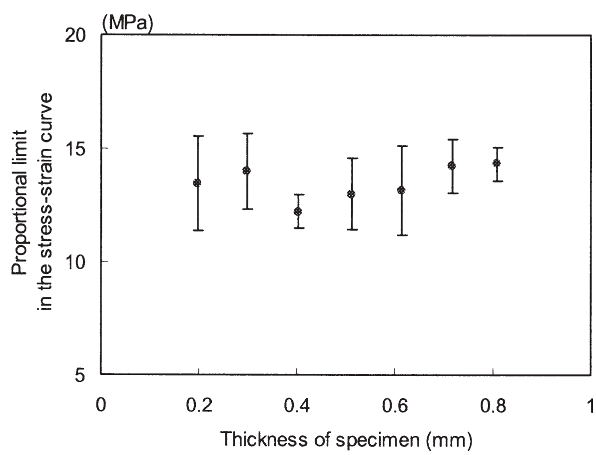

Fig. 2. The relationship between the thickness of the sugi earlywood specimen and the proportional limit in the stress–strain curve. Each point represents average values, the error bar shows the standard deviation. All tests were conducted under wet conditions

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 a dead load. The initial displacement was measured soon after applying the load and was considered an instantaneous displacement. Then, the displacement was detected every few hours for several hundred hours. After the creep test was finished, a time–creep compliance curve was obtained. The creep tests were performed for a broad range of MFA.

Determining microfibril angle

After the creep test was performed, the MFA in the S2 layer of each specimen was measured using an X-ray diffractometer (XD-D1w, Shimadzu).[17]

Measuring wood density

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

Simulating creep properties

A simplified model explaining the longitudinal creep properties is shown in Fig. 3. The model consists of an independent spring (spring1) and a Voigt element (spring2 and dashpot2) in series. The elastic modulus of spring1 and spring2, and the viscosity coefficient of dashpot2 are denoted by E_1, E_2, η_2, respectively. Using this creep model, the following equations are derived:

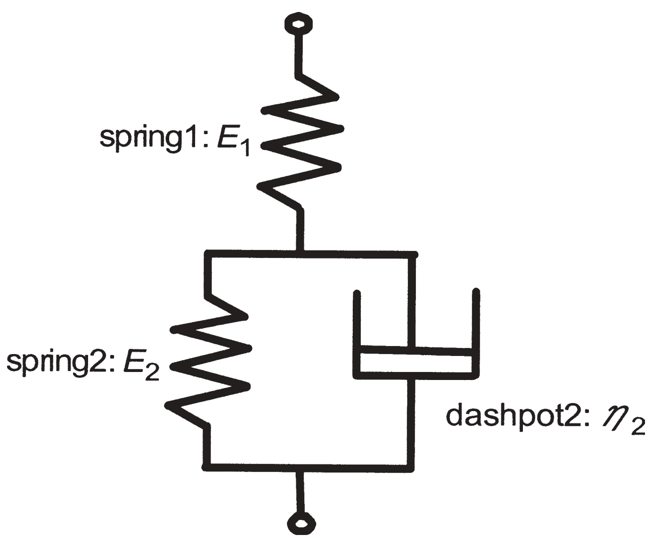

Fig. 3. A simplified viscoelastic model of wood for simulating the longitudinal creep behavior. Spring1 and spring2 have elastic moduli E_1 and E_2, respectively, and dashpot2 has viscosity coefficient η_2

$$\varepsilon(t) = \sigma\left[\frac{1}{E_1} + \frac{1}{E_2}\left(1 - e^{-t/\lambda_2}\right)\right], \qquad \lambda_2 = \frac{\eta_2}{E_2} \tag{1}$$

$$J(t) = \frac{1}{E_1} + \frac{1}{E_2}\left(1 - e^{-t/\lambda_2}\right), \qquad \lambda_2 = \frac{\eta_2}{E_2} \tag{2}$$

where $\varepsilon(t)$, $J(t)$, σ, t, and λ_2 denote the creep strain, creep compliance, loaded stress, time, and the retardation time, respectively.

Then, the parameters E_1, E_2, and η_2 are transformed as follows:

$$E_1 \to E_1^{\mathrm{W}}\left(=\frac{\rho^{\mathrm{W}}}{\rho_0}E_1\right),$$

$$E_2 \to E_2^{\mathrm{W}}\left(=\frac{\rho^{\mathrm{W}}}{\rho_0}E_2\right),$$

$$\eta_2 \to \eta_2^{\mathrm{W}}\left(=\frac{\rho^{\mathrm{W}}}{\rho_0}\eta_2\right)$$

where ρ^{W}, ρ_0, E_1^{W}, E_2^{W}, and η_2^{W} represent the density of the cell wall, the density of oven-dried wood, the substantial elastic modulus of spring1 and spring2, and the substantial viscosity coefficient of dashpot2, respectively. We can rewrite Eq. 2 as:

$$J^{\mathrm{W}}(t) = \frac{1}{E_1^{\mathrm{W}}} + \frac{1}{E_2^{\mathrm{W}}}\left(1 - e^{-t/\lambda_2^{\mathrm{W}}}\right), \quad \lambda_2^{\mathrm{W}} = \lambda_2 = \frac{\eta_2}{E_2} \tag{3}$$

It is quite natural to consider $J^{\mathrm{W}}(t)$ as the substantial creep compliance of the cell wall in the longitudinal direction. We tried to determine the values of the unknown parameters (E_1^{W}, E_2^{W}, η_2^{W}, λ_2^{W}) to simulate the experimental results quantitatively. The physical meanings of the estimated values and their dependencies on the MFA are discussed.

Results and discussion

Longitudinal tensile creep properties

Figure 4 shows the time dependency of the longitudinal tensile creep compliance $J(t)$, of the wood. As seen in Fig. 4, 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 the bending creep properties under a steady moisture condition using wood beams.[18,19] The magnitude of the creep deformation tended to increase with the MFA.

The $J(t)$ curve in Fig. 4 does not consider the densities of the specimen. 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_L, into the substantial Young's

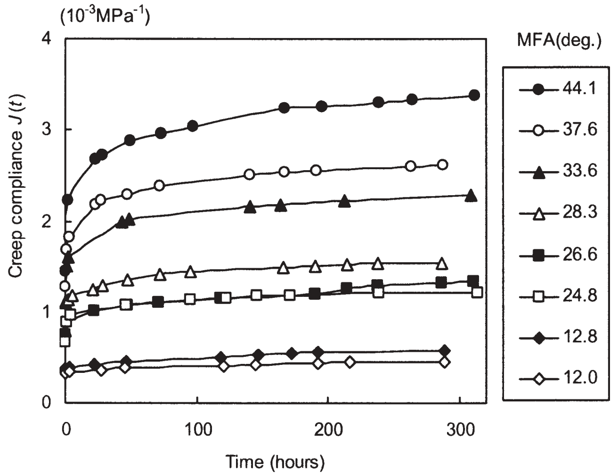

Fig. 4. The time dependency of the longitudinal tensile creep compliance $J(t)$ of the sugi earlywood specimen. All creep tests were conducted at the fiber saturation point (moisture content: 25%). The creep load was 14.2 MPa, which corresponds to the proportional limit of earlywood in sugi (wet)

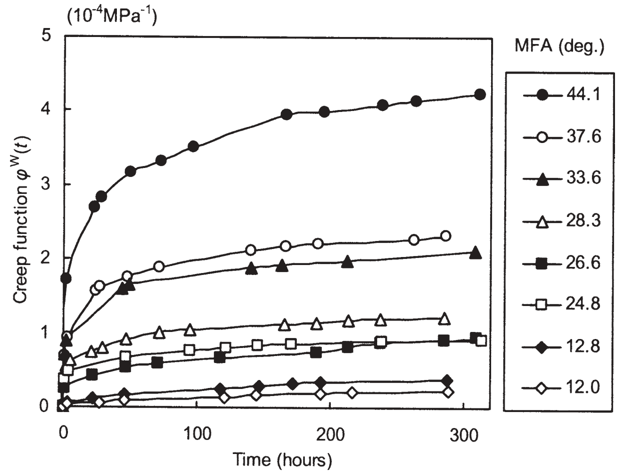

Fig. 5. The substantial creep function of the cell wall $\varphi^{W}(t)$. All creep tests were conducted at the fiber saturation point (moisture content: 25%). The creep load was 14.2 MPa, which corresponds to the proportional limit of earlywood in sugi (wet)

modulus of the wood cell wall, E_L^W, we can use the following formula:

$$E_L^W = E_L \frac{\rho^W}{\rho_0}$$

where, ρ^W is the density of the cell wall, and ρ_0 is the density 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:

$$J^W(t) = J(t)\frac{\rho_0}{\rho^W}$$

Figure 5 shows the substantial creep function of the cell wall, $\varphi^W(t)$, which is calculated by subtracting the initial instantaneous compliance ($1/E_1^W$) from $J^W(t)$. For a small MFA, the creep deformation remained very small for a few hundred hours in the creep test. As MFA increased, the creep strain became larger and amounted to several percent for specimens with MFA $> 30°$. This shows that the longitudinal tensile creep properties of wood are highly dependent on the MFA. This can be explained by the composite structures of the wood cell wall. The MFA in the S2 layer has a very important effect on the mechanical properties of wood, e.g., the origin of the longitudinal Young's modulus[14,20–23] and the anisotropic swelling and shrinkage.[15,24–26] A small MFA means that the cellulose microfibril (CMF) bundles are arranged nearly parallel in the longitudinal direction of the cell. Therefore, the longitudinal tensile creep properties are dominated mainly by the CMF in the S2 layer. Because the viscosity of the CMF is much larger than that of the matrix substance, the creep function becomes very small at a small MFA. By contrast, at a large MFA, the matrix substance in the cell wall affects the longitudinal tensile creep behavior, 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.

Simulated results using the simplified viscoelastic model

The observed $J^W(t)$ was simulated using the simplified viscoelastic model formulated as Eq. 3. It is quite reasonable to consider that the values of the parameters in the simulation reflect intrinsic information on the fine structure and internal properties of the cell wall constituents. Figure 6 shows the dependency of the fitted values of the E_1^W, E_2^W, η_2^W, and λ_2^W on the MFA. As seen in Fig. 6a, the value of E_1^W decreased concavely as the MFA increased, which concurs with the previous results reported by Cave,[20] Sobue and Asano,[21] Page et al.,[22] and Norimoto et al.[23] The MFA dependencies of the simulated values of E_2^W and η_2^W were steeper than that of E_1^W, as shown in Fig. 6b,c. 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 at large MFA. This is why, in the region of small MFA, the longitudinal creep deformation was affected by the CMF, which is much more viscous than the matrix substance in the cell wall constituents. Conversely, in the region of large MFA, the longitudinal creep deformation is affected by the matrix substance, which is much less viscous than the CMF. The values of η_2^W and λ_2^W are indicators of the smallness of the creep rate. As shown in Fig. 6c,d, these two values are clearly dependent on the MFA. For small MFA, the values of η_2^W and λ_2^W are much larger than at large MFA, because the creep rate is affected mainly by the CMF. Conversely, the values of η_2^W and λ_2^W decrease as MFA increases, because the longitudinal creep rate is markedly affected by the matrix substance in the region of large MFA.

We estimated the unknown parameters of the simplified model by comparing the simulation with the experimental

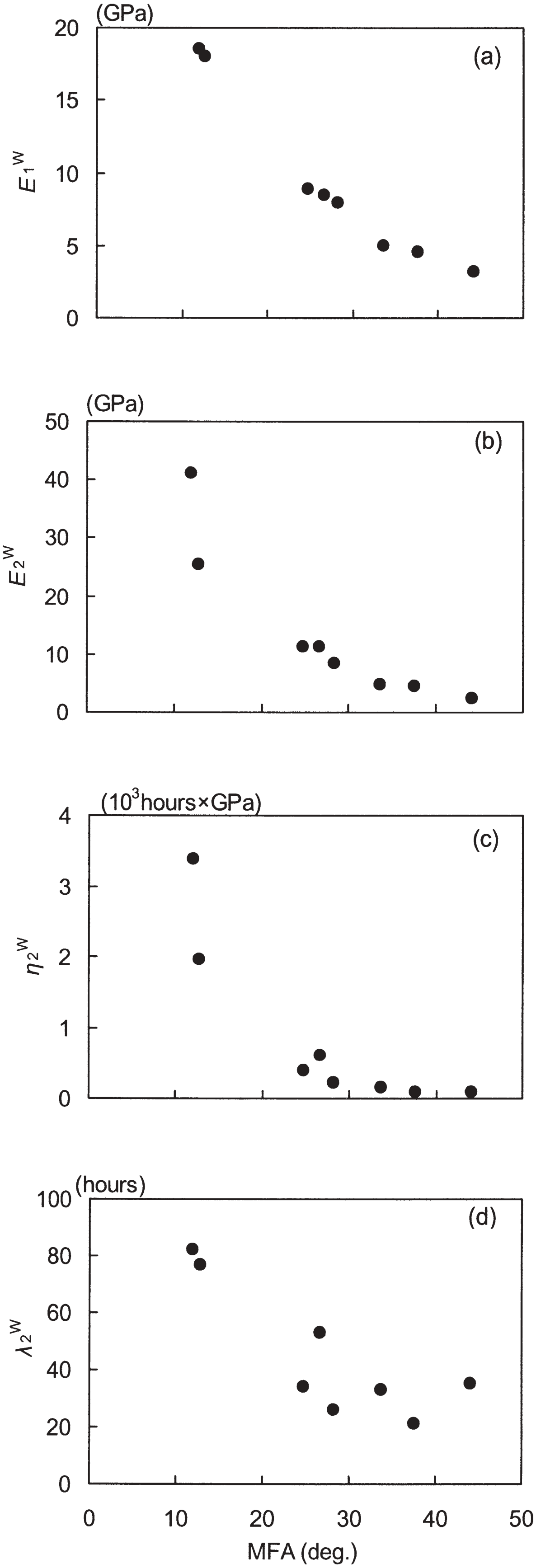

Fig. 6a–d. The microfibril angle (*MFA*) dependency of calculated values of the parameters in the simplified model. **a** E_1^W, **b** E_2^W, **c** η_2^W, **d** λ_2^W

results. The parameters are highly dependent on the MFA. This is because the tensile creep deformation is affected by the arrangement of the cell wall constituents, namely the CMF framework and matrix substance. To clarify the effect of the cell wall constituents on the longitudinal tensile creep property of wood, we must construct a multi-layered cell wall model considering the composite and fine structures of the cell wall lamella.

Conclusions

This article examined the dependence of the longitudinal tensile creep behavior of thin specimens of sugi on MFA. Then, we determined the values of parameters in a simplified viscoelastic model, and discussed the physical meanings of those values. We found that the longitudinal tensile creep deformation is highly dependent on the MFA and each parameter in the simplified model is clearly affected by the MFA. These results suggest that in the region of small MFA, the creep deformation becomes much smaller and is affected by the CMF, which is very viscous. Conversely, in the region of large MFA, the creep deformation becomes larger and is affected by the matrix substance, which is much less viscous than the CMF.

To clarify the effect of the cell wall constituents on the longitudinal tensile creep property in detail, we need to construct a multi-layered cell wall model that considers the composite and fine structures of the cell wall lamella.

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