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Thermal softening properties of various wood species within an annual ring

Hiroaki Horiyama[1], Yuka Miyoshi[2], Keisuke Kojiro[1*] and Yuzo Furuta[1]

Abstract

Dynamic mechanical analysis (DMA) measurements of water-saturated earlywood (EW) and latewood (LW) of various wood species in the temperature range from 0 to 100 °C were focused to clarify the differences in thermal softening properties within an annual ring. The following results were obtained. The peak of tanδ caused by micro-Brownian motion of lignin was observed in both EW and LW all species. For softwoods, the peak temperatures of tanδ of EW appeared at higher temperatures than those of LW. For hardwoods, in the other hand, the peak temperatures of tanδ were slightly different between EW and LW in the diffuse-porous wood, whereas the temperatures were almost the same in the ring-porous wood. It was found that the difference in peak temperature of tanδ between EW and LW varied greatly among species. The difference in peak temperature between EW and LW was relatively large for softwoods. In addition, the thermal softening properties both in the radial and tangential directions differed depending on the species, so this suggested that there was anisotropy in the thermal softening properties depending on the species.

Keywords Earlywood, Latewood, Dynamic mechanical analysis, Lignin, Thermal softening, Anisotropy

Introduction

It has long been known that the mechanical properties of industrial materials such as metals and plastics vary greatly depending on the differences in the thermodynamic states of the atoms and molecules. Furuta et al. reported that the mechanical properties of wood also varied greatly with differences in the thermodynamic states of the molecules [1, 2]. For example, it has been revealed that elastic modulus decreases and loss modulus increases in water-saturated wood due to quenching or drying history [1, 2]. In the case of wood placed in an environment for a long time, the molecules remain in a stable state in terms of humidity and temperature. However, when environment changes rapidly like quenching or drying, molecule arrangements in cell wall cannot conform to the new environment and falls into thermodynamic unequilibrium state. Understanding the thermal softening properties of wood is considered important for understanding the molecular state of wood. Dynamic mechanical analysis (DMA) of water-saturated wood in the temperature range from 0 to 100 °C are often used as a method to determine the thermal softening properties of wood and bamboo [3–8]. Furuta et al. reported that the peak of tanδ obtained by dynamic viscoelasticity measurements from 0 to 100 °C is due to micro-Brownian motion of lignin [3]. The peak of tanδ in rapidly cooled and/or dried water-saturated wood has been reported to be lower than that of raw wood [1, 2]. The peak temperature of tanδ, which was lowered by these histories, increased over time with water immersion [2]. Thus, the history-induced changes in mechanical properties are closely related to the thermodynamic state of the steric structure of lignin.

In softwood, the peaks of tanδ are found from 80 to 90 °C, whereas in hardwood, that is found from 60 to

*Correspondence:
Keisuke Kojiro
kojiro@kpu.ac.jp
[1] Graduate School of Life and Environmental Sciences, Kyoto Prefectural University, Kyoto 606-8522, Japan
[2] Forestry and Forest Products Research Institute, Tsukuba, Ibaraki 305-8687, Japan

70 °C at 0.05 Hz [3]. Softwood lignin is composed of only guaiacyl-propane units, while hardwood lignin is composed of guaiacyl and syringyl-propane units. It is generally believed that the degree of condensation of softwood lignin is higher than those of hardwood lignin [9]. It was considered that the difference in the peak temperature of tanδ between hardwood and softwood is due to the difference in the cross-linking density derived from the structure of lignin in the study of Furuta et al. [3]. On the other hand, the lignin concentration and ratio of syringyl lignin to guaiacyl lignin in wood vary with each tissue. It has been reported that the distribution of lignin in the cell wells of vessel, fiber and ray cell in Birch wood by observation with the ultraviolet microscopy and bromination technique with the SEM–EDXA system [10, 11]. It has been reported that guaiacyl lignin is enriched in the S2 layer of vessels, syringyl lignin in the S2 layer of wood fibers, and guaiacyl lignin is enriched in the corner intercellular layer between various cells [11]. Watanabe and Fukazawa reported that in the genus maple, wood fibers were syringyl-rich in except species, but the vessels were quite variable from species to species [12]. In particular, trees in temperate regions have annual ring structure, which is more pronounced in conifers. There have been many studies on the annual ring structure of coniferous trees, especially the structure of earlywood (EW) and latewood (LW) [13–16]. The change in physical properties within an annual ring is mostly explained by the difference in density [17]. It has been reported that lignin concentrations in the cell wall and intercellular layer differ between EW and LW of conifers [10, 11]. However, the relationship between lignin structure and thermal softening properties within an annual ring has not been investigated. Our previous studies have shown that EW and LW of Douglas fir (*Pseudotsuga menziesii*) have different thermal softening properties [8]. The peak of tanδ was found at around 95 °C for EW and around 90 °C for LW [8]. It was found that the difference in thermal softening properties between EW and LW could not be explained only by the difference in specific density [8]. In other words, the microstructure may be different within an annual ring. However, it is not clear whether similar trends can be obtained in other softwoods, or whether the thermal softening properties of hardwoods vary within an annual ring. Hardwoods are known to have different ratios of lignin monomers in the vessels and fibers [12]. Therefore, thermal softening properties of hardwoods are also expected to vary widely within an annual ring.

As described above, it is extremely likely that the thermal softening properties of various wood species differ within an annual ring. In other words, the microstructure of each species differs within a single annual ring. However, previous studies have been based on the results of DMA measurements in the R direction to understand the microstructure. It is important to investigate the thermal softening properties of wood within an annual ring in order to control microstructure, which is a major issue in the use of wood as an industrial material. In this study, dynamic viscoelasticity measurements of EW and LW of various wood species were performed using DMA in order to investigate the differences in thermal softening properties of various wood species within annual rings.

Materials and methods

Materials

Table 1 shows the wood species used for the DMA. Softwoods such as Douglas fir were selected for their clear visual distinction between EW and LW, and for their large latewood width. Hardwoods were selected that differ vessel distribution, such as ring-porous and diffuse-porous. The sample sizes for EW and LW were 1.2 mm

Table 1 Wood species used for the dynamic mechanical analysis

Features of anatomy	Species	Density (g/cm^3)
Softwood		
Gradual transition	*Abies sachalinensis* (Fr. Schmidt) masters	0.42
	Pinus radiata D.Don	0.47
Abrupt transition	*Pseudotsuga menziesii* (Mirb.) Franco	0.50
	Pinus densiflora Siebold et Zucc.	0.60
	Cedrus deodara (Roxb.) G.Don	0.63
Hardwood		
Ring-porous wood	*Albizia julibrissin* Durazz.	0.47
	Castanea crenata Siebold et Zucc.	0.54
Diffuse-porous wood	*Zanthoxylum ailanthoides* Siebold et Zucc	0.44
	Populus alba L.	0.47

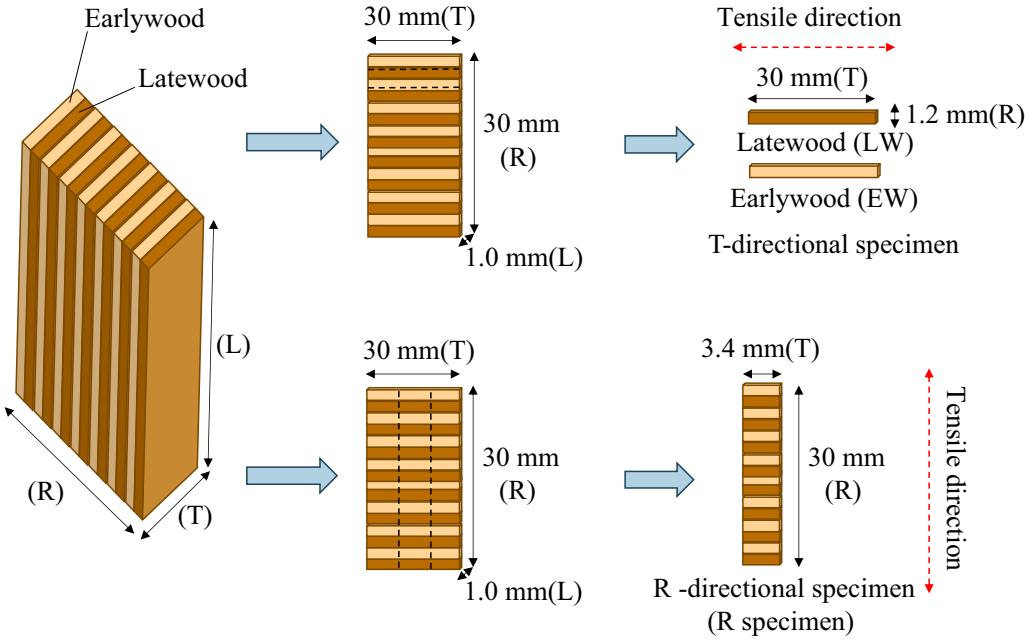

Fig. 1 Schematic diagram of sample shape of dynamic mechanical analysis (DMA)

in the radial (R) direction, 30 mm in the tangential (T) direction, and 1.0 mm in the longitudinal (L) direction. R-directional specimen (R specimen) were also measured as a comparison control. The sample sizes for R specimen were 30 mm in the R direction, 1.2 mm in the T direction, and 1.0 mm in the L direction. The schematic diagram of samples is shown in Fig. 1. The samples were boiled for two hours in distilled water and then annealed to room temperature in those to make them saturated.

Dynamic mechanical analysis (DMA)

Temperature dependence of dynamic elastic modulus (E'), loss modulus (E''), and tanδ were measured by the tensile forced oscillation method using a dynamic mechanical analyzer (DMS6100, Seiko Instruments, Chiba, Japan). The samples swollen by the distilled water were measured in the distilled water at a temperature range from 20 to 95 °C. The heating and cooling rate was 1 °C/min. Frequencies for the measurement were 0.5, 1.0, 2.0, 5.0, and 10 Hz. The span was 12 mm, and the displacement amplitude was 5 μm. As shown in Fig. 1, the tensile direction was in the radial or tangential direction. The tensile direction was tangential and radial. As shown in Fig. 2, results were obtained in the second heating process to uniform the heating and cooling histories [3].

Scanning electron microscope (SEM)

The cross-section of the samples was planed using a sliding microtome. The samples were dried at 105 °C for 24 h, and then the cross-section was observed using SEM (TM3030 Plus Miniscope, Hitachi High-Technologies, Tokyo, Japan) at an accelerating voltage of 15 kV.

Results and discussion

Figure 3 shows SEM images of the wood used in this study. It can be seen that the specimens used in this study are normal wood, neither reaction wood nor starved

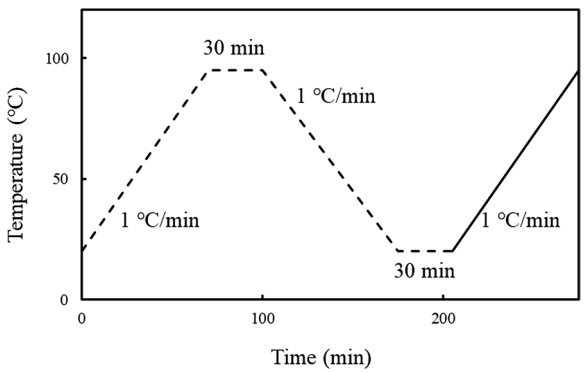

Fig. 2 Temperature program for measurements of dynamic viscoelastic properties from 20 to 95 °C. Solid line corresponds to the temperature program used in Figs. 3, 4, 5 and 6; dashed line corresponds to the pretreatment process to uniform heating and cooling histories

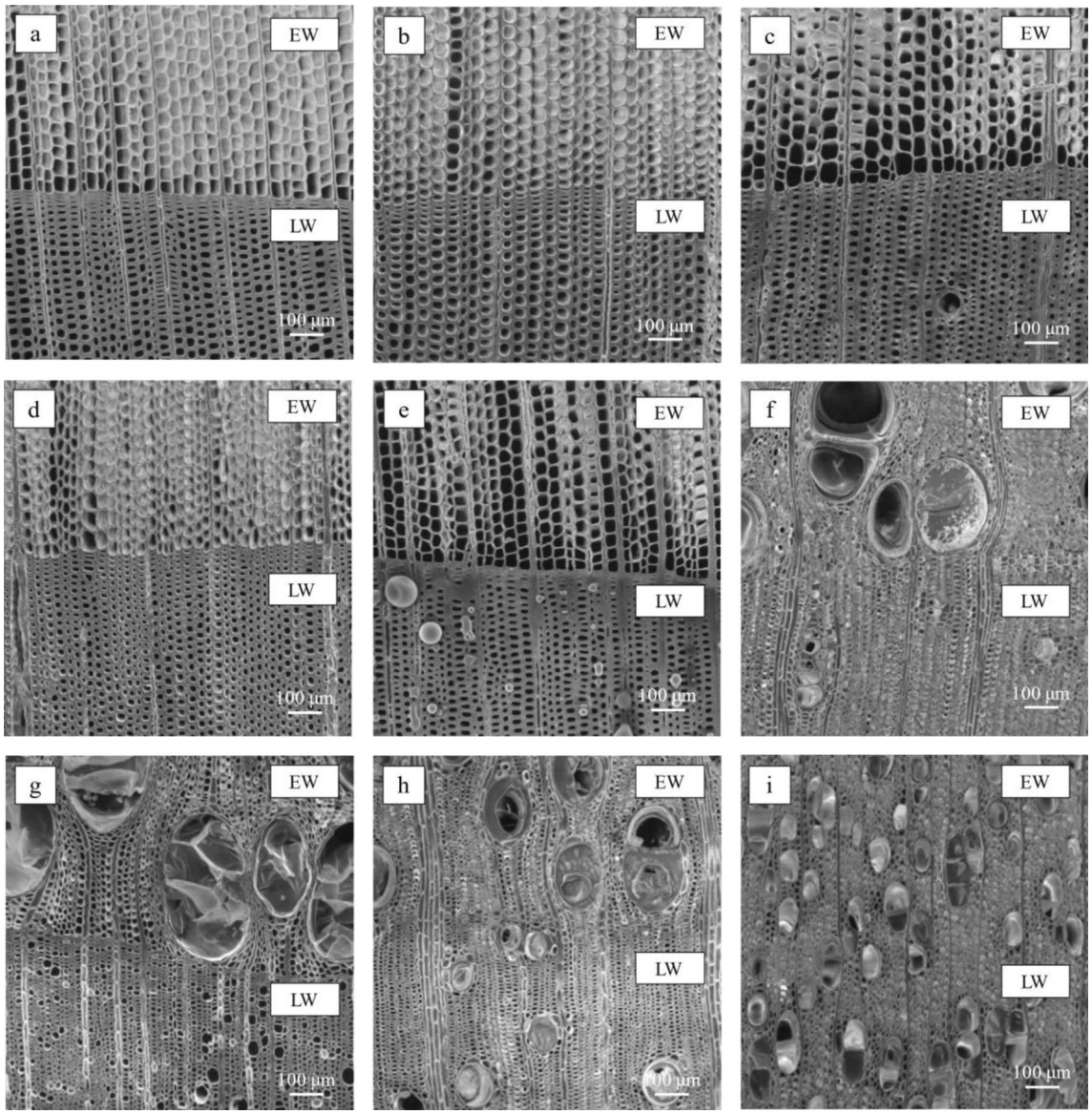

Fig. 3 SEM images of some kinds of wood used in this study. **a**–**e**, softwoods. **a**, *P. densiflora*. **b**, *P. radiata*. **c**, *P. menziesii*. **d**, *A. sachalinensis*. **e**, *C. deodara*. **f**–**i**, hardwoods. **f**, *A. julibrissin*. **g**, *C. crenata*. **h**, *Z. ailanthoides*. **i**, *P. alba*

wood. *Cedrus deodara* was found to spew extractable components like carbohydrates, as shown in Fig. 3e. Narrow vessel were abundant in LW of g (*Castanea crenata*) and h (*Zanthoxylum ailanthoides*) is a diffuse-porous wood, but the vessel in LW were slightly smaller and less numerous than those in EW. As mentioned above, it has been reported that guaiacyl lignin is enriched in the S2 layer of vessels, syringyl lignin in the S2 layer of wood fibers, and guaiacyl lignin in the corner intercellular layer between various cells [11, 12]. Based in the above, it is possible that the hardwoods used in this study also exhibit lignin heterogeneity within an annual ring.

Figures 4 and 5 show the temperature dependence of E' in the tangential and radial direction of various wood swollen by water at 0.5 and 10 Hz. E' of EW and LW decreased with increasing temperature in the range from 30 to 100 °C. E' decreased with increasing temperature in the range from 30 to 100 °C in both R and T directions for all tree species. In all species except c (*Pseudotsuga menziesii*) and h (*Zanthoxylum ailanthoides*), E' of the R specimens showed the largest value; in the T direction, E' of LW tended to be larger than E' of EW for all species. No significant difference was observed at the measurement frequency of 10 Hz, except that E' was larger

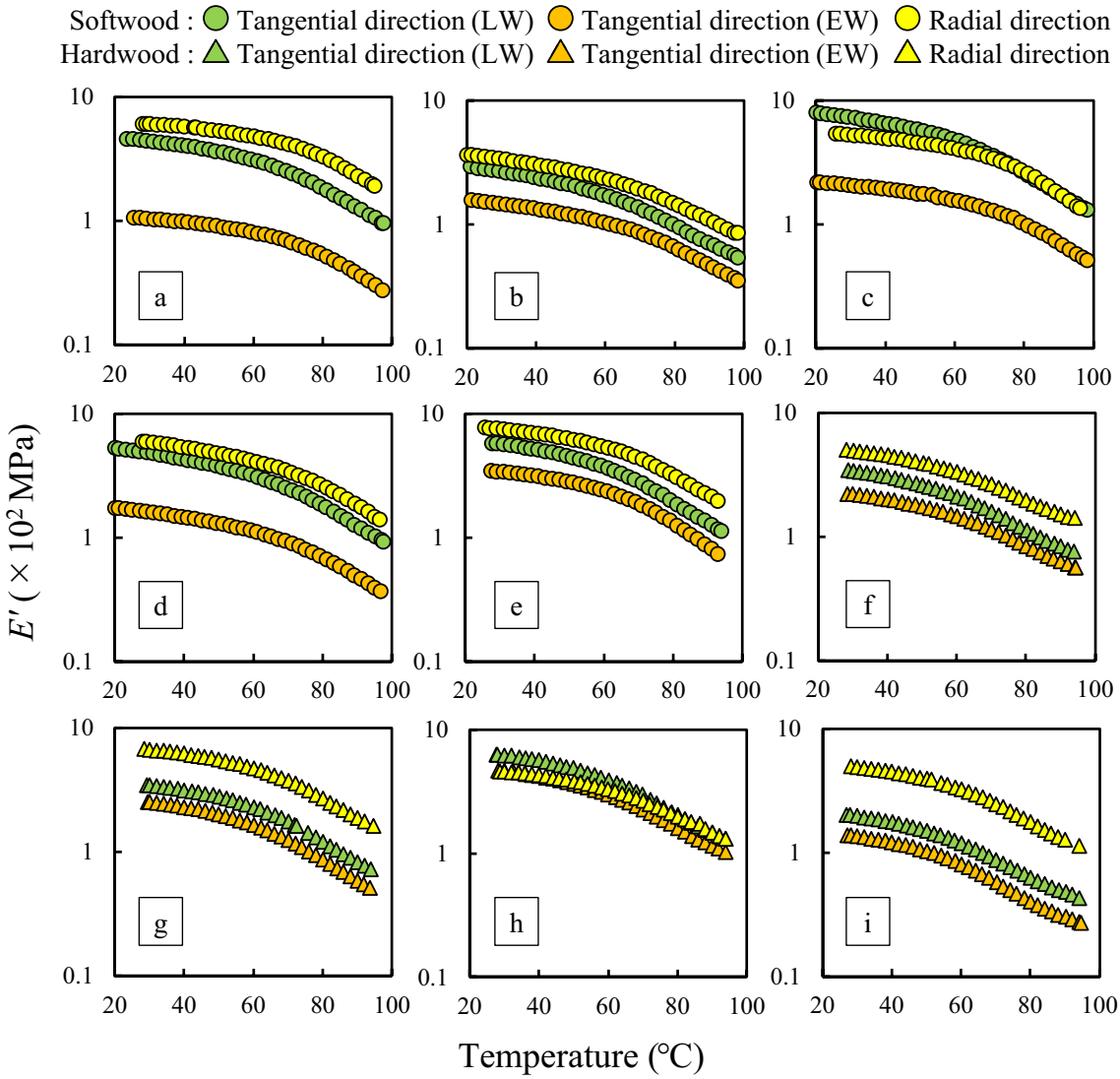

Fig. 4 Temperature dependence of E' in tangential and radial direction of some kind of wood swollen by water at 0.5 Hz. Green filled circles and triangle, LW; orange filled circles and triangle, EW; yellow filled circles and triangle, R specimen. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

than at 0.5 Hz. Figures 6 and 7 show the temperature dependence of tanδ in the T and R direction of various wood swollen by water at 0.5 and 10 Hz. For softwoods, the temperature dependence of tanδ of R specimens was almost the same as that of EW. For hardwoods, the temperature dependence of tanδ of EW was almost the same as that of LW. The tanδ values measured at 10 Hz were greater for all measurement directions for all species compared to 0.5 Hz. At a frequency of 0.5 Hz, the peaks of tanδ were observed except of R specimens for c (*Pseudotsuga menziesii*) and a (*Abies sachalinensis*). A tendency for the peak of tanδ to shift toward higher temperatures was obtained at a frequency of 10 Hz. As a result, there were some tree species for which the peak of tanδ could not be observed in the range from 0 to 100 °C at a frequency of 10 Hz.

Furuta et al. reported that the peak in tanδ obtained from dynamic viscoelasticity measurements of water-saturated wood from 0 to 100 °C is due to micro-Brownian motion of lignin [3]. They also reported that the tanδ peaks of softwoods and hardwoods can suggest differences in lignin structure, etc. by organizing them by tanδ peak [3]. Therefore, from the obtained results of temperature dependence of tanδ, we focused on the peak of tanδ and organized the results. Figure 8 shows relationships between the peak value of tanδ and the peak temperature of tanδ of EW, LW and R specimens of various wood measured at 0.5 Hz. In softwood, the

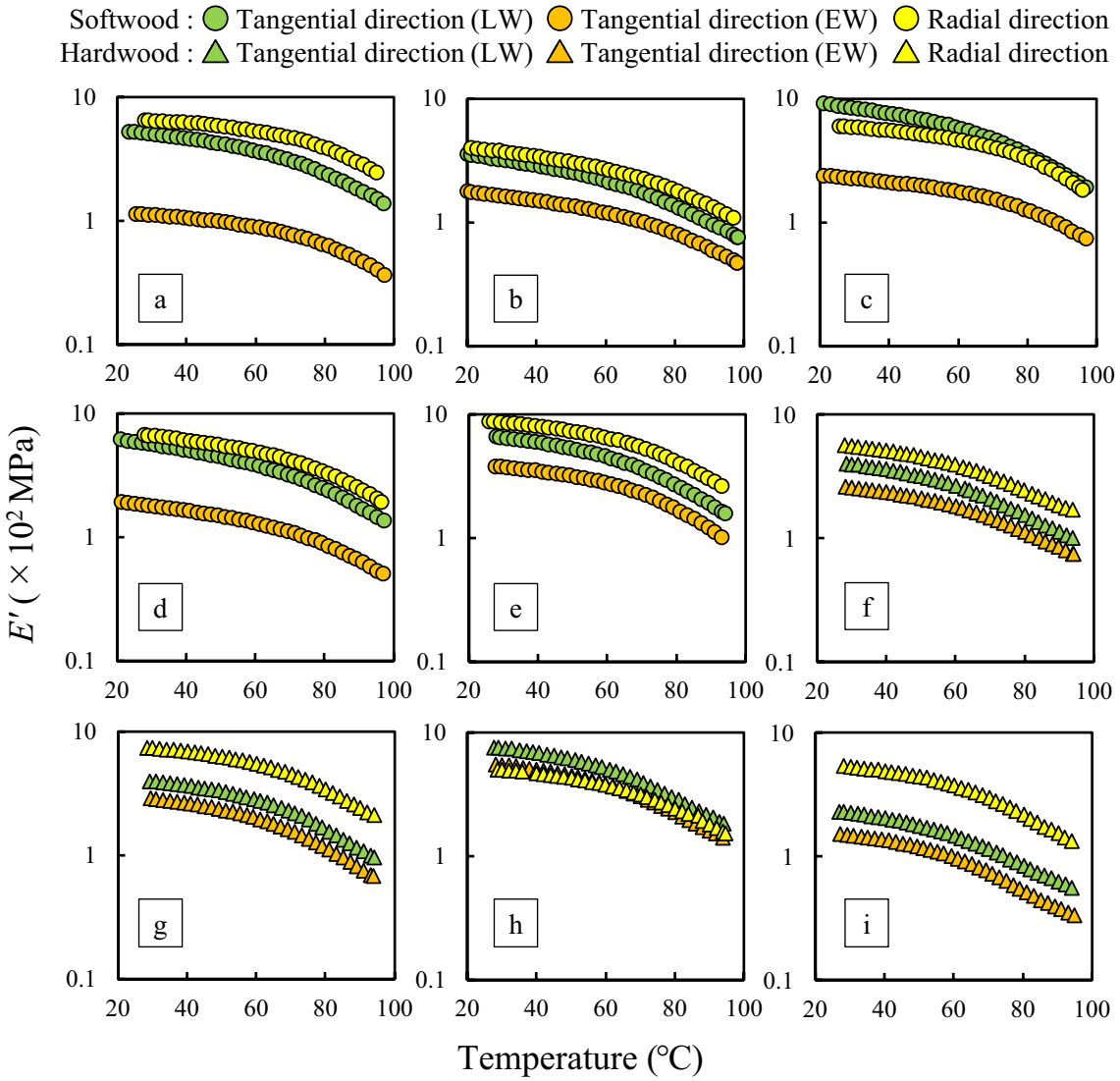

Fig. 5 Temperature dependence of E' in tangential and radial direction of some kind of wood swollen by water at 10 Hz. Green filled circles and triangle, LW; orange filled circles and triangle, EW; yellow filled circles and triangle, R specimen. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

peaks of tanδ are found from 85 to 95 °C, whereas in hardwood, those are found from 75 to 85 °C at 0.5 Hz. For softwoods, the peak temperature of tanδ of EW at 0.5 Hz appeared at higher temperatures than those of LW as in the previous report [8]. In addition, the peak temperature of tanδ of R specimens was similar to those of EW except for *Pseudotsuga menziesii* and *Abies sachalinensis*, for which the peak tanδ of R specimens was not observed. For hardwoods, the peak temperature of tanδ at 0.5 Hz was slightly different between EW and LW in the diffuse-porous wood, whereas the temperatures were almost the same in the ring-porous wood. It is known that as the degree of condensation of polymeric materials increases, the peak temperature of tanδ shifts significantly to the high temperature side [18]. The results of dynamic viscoelasticity measurements of delignified hinoki (R specimen) reported that the stronger the degree of delignification, the greater the decrease in E' at lower temperatures, and the tendency for the peak temperature of tanδ to shift to lower temperatures [19]. Similarly, it has been reported that the peak temperature of tanδ for hinoki wood also shifts to the lower temperature side with the progress of delignification [20]. Seki et al. reported from nuclear magnetic resonance (NMR) and attenuated total reflection-infrared spectroscopy (ATR-IR) measurements

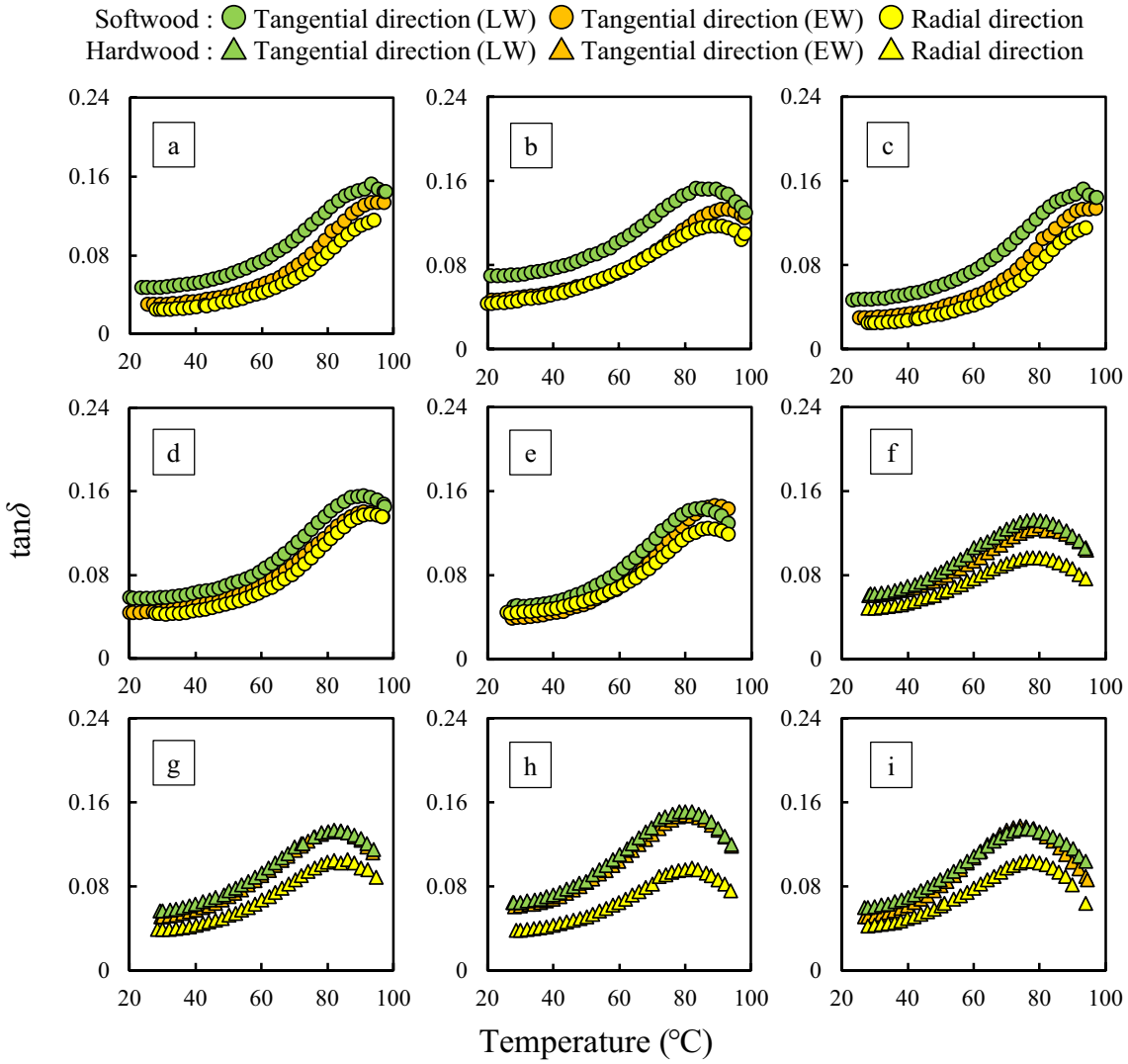

Fig. 6 Temperature dependence of tanδ in tangential and radial direction of some kind of wood swollen by water at 0.5 Hz. Green filled circles and triangle, LW; orange filled circles and triangle, EW; yellow filled circles and triangle, R specimen. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

that the benzene ring of the guaiacyl unit on the surface of the lignin unit is oxidized to form a carboxyl group in the initial stage of delignification (treatment time 10 min), although the mass loss is slight [20, 21]. Even in the initial stage of delignification, the thermo-softening temperature decreases by about 5 °C [20]. In other words, the peak temperature of tanδ decreases even if the structural change is not large enough to cleave the lignin unit. From this, it can be inferred that the lignin structure has a greater effect on the peak tanδ temperature than the amount of lignin. One possible reason for the difference in the peak temperature of tanδ between EW and LW is that the lignin in softwoods seems to consist mainly of guaiacyl-propane units, but even if the same guaiacyl-propane units are present, the lignin structure, such as cross-link density, may differ between EW and LW.

As mentioned above, the peak tanδ of water-saturated wood in the 0–100 °C range is believed to be derived from lignin. The degree to which the tanδ peak differs within an annual ring can be used as an indicator of microstructural differences. To investigated how tanδ peaks differ among species within an annual ring, Fig. 9 shows the relationships between the difference in the peak value of tanδ (those values are the peak value of tanδ EW minus those of LW) and the difference in the peak temperature of tanδ (those values are the peak temperature of tanδ EW minus those of LW) in T direction of various wood measured at 0.5 Hz. It was

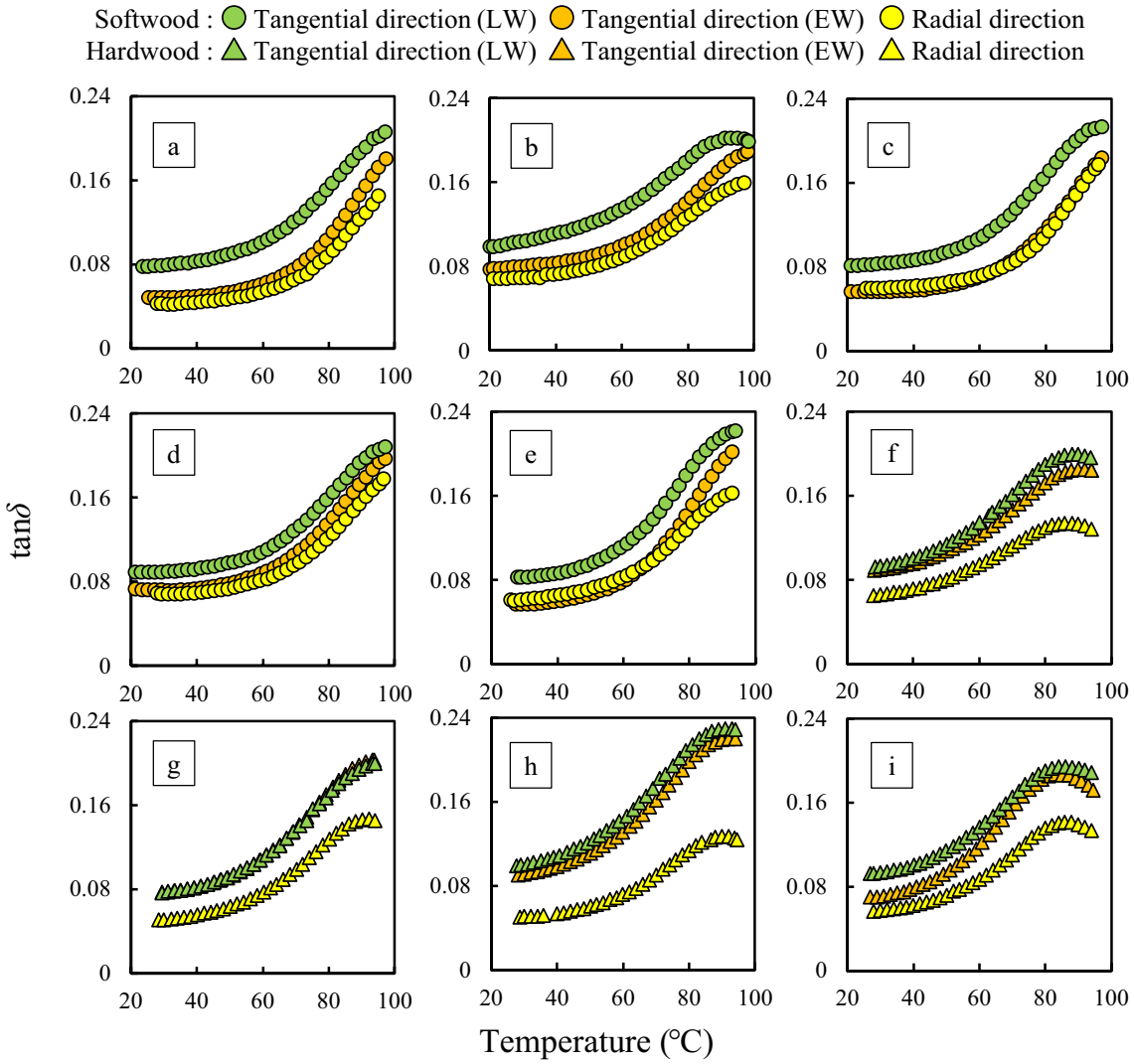

Fig. 7 Temperature dependence of tanδ in tangential and radial direction of some kind of wood swollen by water at 10 Hz. Green filled circles and triangle, LW; orange filled circles and triangle, EW; yellow filled circles and triangle, R specimen. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

found that the difference in the peak value of tanδ and the peak temperature of tanδ between EW and LW was smaller for hardwoods than for softwoods, regardless of species. On the other hand, for softwoods, the difference in the peak temperature of tanδ between EW and LW varied greatly among species. The species with particularly large peak temperature differences between EW and LW was c (*P. menziesii*), with a 5 °C temperature difference.

In previous studies, many DMA measurements have been performed in the R direction. As shown in Fig. 8, the thermal softening behavior differs significantly between the R and T directions. To investigated how tanδ peaks differ in the direction of measurement, Fig. 10 shows the relationships between the difference in the peak value of tanδ and the difference in the peak temperature of tanδ of the specimen in R direction and T direction (EW and LW) of various wood measured at 0.5 Hz. For both softwoods and hardwoods, the difference in the peak temperature of tanδ between the R and T directions was small. However, the peak value of tanδ was found to be larger in the T direction, especially for hardwoods. Furuta et al. reported that there was no difference in thermal softening properties in the RT plane for water-swollen Japanese cypress (*Chamaecyparis obtusa* Endl.), and no clear anisotropy existed [22]. However, the present results indicate that there is anisotropy in thermal softening properties even in the RT plane, depending on the species. These results suggest that when discussing thermal softening properties, it is better to take

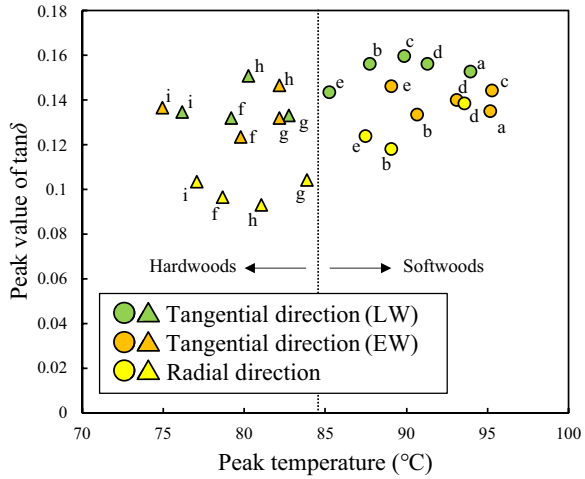

Fig. 8 Relationships between the peak value of tanδ and the peak temperature of tanδ of EW, LW and R specimen of various wood measured at 0.5 Hz. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

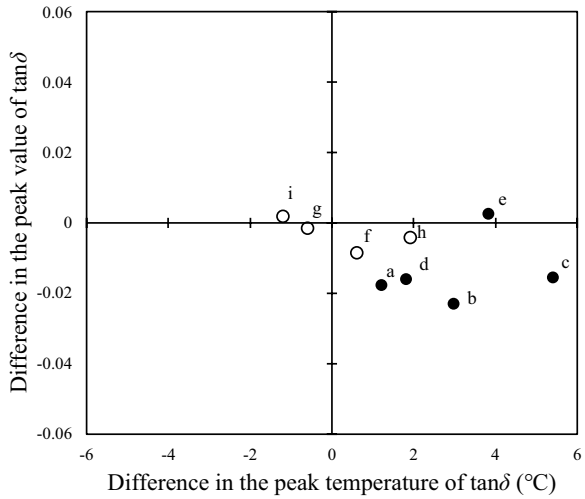

Fig. 9 Relationships between the difference in the peak value of tanδ and the difference in the peak temperature of tanδ of EW and LW of various wood measured at 0.5 Hz. These differences are EW values minus LW. Filled circles, softwoods; open circles, hardwoods. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

into consideration that different wood species have different the peak temperature of tanδ within an annual ring and anisotropy.

Conclusions

In this study, dynamic viscoelasticity measurements of EW and LW of various wood species were performed using DMA in order to investigate the differences in thermal softening properties of various wood species within annual rings. In addition, R specimens used in previous

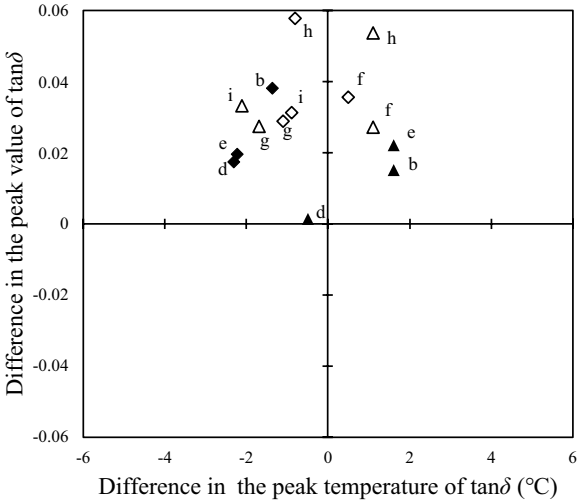

Fig. 10 Relationships between the difference in the peak value of tanδ and the difference in the peak temperature of tanδ of the specimen in R direction and T direction of various wood measured at 0.5 Hz. These differences are the specimen in R direction values minus EW or LW in T direction. Filled triangle and rhombus, softwoods; open triangle and rhombus, hardwoods; triangle, the difference between EW and R specimens; rhombus, the difference between LW and R specimens. **a**–**e**, Softwoods. **f**–**i**, Hardwoods. See Fig. 3 for more details

studies were also measured and compared with the results of EW and LW.

The following became clear as those results:

1. E' decreased with increasing temperature in the range from 30 to 100 °C in both R and T directions for all tree species. Except for some species, E' in the R direction was the largest, and in the T direction, E' in LW tended to be larger than E' in EW for all species.
2. For softwoods, the peaks temperature of tanδ for EW appeared at higher temperatures than those of LW, and the peaks temperature of tanδ in the R direction were similar to those of EW in the T direction. For hardwoods, the peaks temperature of tanδ were slightly different between EW and LW in the diffuse-porous wood, whereas the temperatures were almost the same in the ring-porous wood.
3. In softwoods, the peak temperature of tanδ tended to be higher for EW than for LW. On the other hand, no clear trend was observed for hardwoods. It was found that the difference in peak temperature of tanδ between EW and LW varied greatly among species. The peak temperature difference between EW and LW was particularly large for softwoods, with a maximum difference of 5 °C. The difference in the peak temperature of tanδ between the R and T directions

was small for both softwoods and hardwoods. However, the peak value of tanδ was larger in the T direction, especially for hardwoods.

Abbreviations

EW	Earlywood
LW	Latewood
DMA	Dynamic mechanical analysis
E'	Dynamic elastic modulus
E''	Loss modulus
SEM	Scanning electron microscope
NMR	Nuclear magnetic resonance
ATR-IR	Attenuated total reflection-infrared spectroscopy

Author contributions

HH designed the study, collected and analyzed data and wrote the initial draft of the manuscript. KK, YM and YF contributed to dynamic viscoelastic analysis and interpretation of data, and assisted in the preparation of the manuscript. All authors agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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