Basic Study on Controlling Deformation and Fracture Properties of Wood in Lateral Tension

2016

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Contents

General introduction

As a basis for efficient use of wood in accordance with the purpose, studies have been carried out in the field of wood physics. Recently, utilization method of wood is diversified, and new technologies and products are used practically and expanded by referring to the results of various studies. Such as study of the new wood-based materials, including cross laminated timber, development of new functional materials using cellulose nanofibers, studies for artificial drying technology of various wood species and by high-temperature setting method, and development of high processability technology of flow molding, three-dimensional molding and so on. In the future, physical properties of wood should be clarified from the aspects of biological and molecular structure in detail and results obtained should be applied to technologies for more advanced use of wood.

Therefore, several studies were conducted for controlling the property of wood in use. Among the these studies, exceptional deformation behavior of wood in lateral tension was shown before fracture. In the study for the deformation of wood, bending or compressing deformation was examined frequently, however, study about tensile deformation was very few and processing technology using tensile deformation positively was not seen at the present. The reason is considered that micro cracks generated in wood could be a starting point of the fracture and large deformation is difficult.

Thus, focusing on the lateral tensile deformation and fracture which were not studied enough previously, deformation and fracture properties were clarified and those mechanisms were discussed. In addition, the possibility of applying the mechanisms and results obtained from the experiments to the processing technology was also discussed.

This study is composed of a general introduction, four chapters and general conclusions. Purposes of the present study were as follows:

In chapter I, the importance of controlling physical properties of wood was described referring to our precedent studies in terms of utilization. From these studies, interesting results of lateral tensile deformation and fracture obtained in our experiment were focused. Therefore, study about deformation and fracture properties of wood in lateral tension was conducted in the following chapters.

In chapter II, for the purpose of clarifying the deformation and fracture properties of wood in lateral tension as a whole, effects of annual ring inclination against to tensile direction, moisture, temperature, density and anatomical characteristics on lateral tensile properties were examined. From the results obtained, the deformation and fracture mechanisms and the important factors for controlling those properties were discussed.

In chapter III, for the purpose of clarifying lateral tensile fracture mechanisms in detail, relationship between fractures and rheological properties of wood was discussed.

In chapter IV, for the purpose of controlling deformation of wood exactly and considering the possibility for increasing the deformation, effects of drying and quenching histories on lateral tensile properties were discussed.

In the general conclusions, from the above results the chapters, mechanism of deformation and fracture of wood in lateral tension was summarized and potential for applying the results obtained to processing technology of wood was discussed.

CHPTER I

Importance of controlling physical properties of wood

Mechanical and physical properties of wood significantly change due to the moisture content and heating temperature. Therefore, it is important to understand and control these properties for suitably and safely use of wood.

In this chapter, two studies which were conducted to control the performance of wood are introduced. In addition, the result of preliminary experiment that led to the start of main study in following chapters is also described.

Technological development for the control of humidity conditioning performance of slit materials made from Japanese cedar

It is an important subject to make effective use of the domestic wood in Japan. In particular, the technological developments of new use and application are needed for Japanese cedar which was planted in approximately 40 % of the area in Japan. In recent years, for effective utilization method of Japanese cedar, the functions of air purification [1] and humidity conditioning [2-4] of Japanese cedar have been noted. These features have been reported to be excellent in end grain surface. However, Japanese cedar materials generally are often used by exposing the edge grain or flat grain surface in room. Therefore, it is not possible to take advantage of their functions in end grain surface. Accordingly, to increase the exposed area of the end grain surface, the Japanese cedar material carved some slits in the fiber orthogonal direction in the flat grain surface which has already been put to practical use (Fig. I-1, Fig. I-2)[5]. So, the slit material of Japanese cedar is utilized in various products

Fig. I-1 Slit material of Japanese cedar. [5]

Fig. I -2 Usage examples of slit material of Japanese cedar. [5]

such as furniture and interior materials.

However, for slit material, it cannot be said that the way of optimum production has been established because the effects of the slit shape, drying conditions, and installation environment on moisture adsorption and desorption properties of slit samples were not sufficiently studied. To develop the technology for controlling the humidity conditioning performance of slit material made from Japanese cedar, the effects of the groove shape of slit samples, air convection, and heat treatment on moisture adsorption and desorption properties were studied.

The results obtained are as follows. The moisture adsorption and desorption rates of slit samples were increased with increase in end grain area of them. However, the moisture adsorption and desorption rates were not increased with increase in depth of concave, although the end grain area was increased. On the other hand, reduced rates of moisture absorption and desorption in the slit samples with deep concave were increased by air convection. The increase ratios of moisture adsorption and desorption rates were larger in samples with deep concave. Equilibrium moisture content was decreased with increase in heat treatment temperature [Fig. I-3]. Pore volume of micropores smaller than 0.6 nm was decreased with increases in heat treatment temperature [Fig. I-4]. Compromise position of processing conditions for slit material should be sought considering production and labor costs, drying conditions, and installation environment in addition to results obtained in this study.

Swelling behavior of cells in compressed Wood

In recent years, to increase density of soft wood such as Japanese cedar, many studies and technological development for compressed wood have been made. The compressive deformation is fixed temporarily when water-saturated

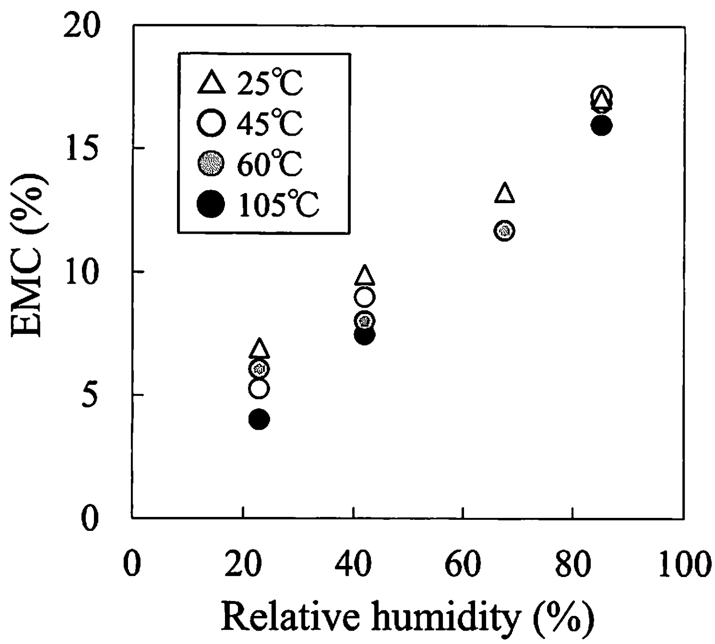

Fig. I-3 The relationship between heat treatment temperature and equilibrium moisture content (EMC) at each relative humidity.

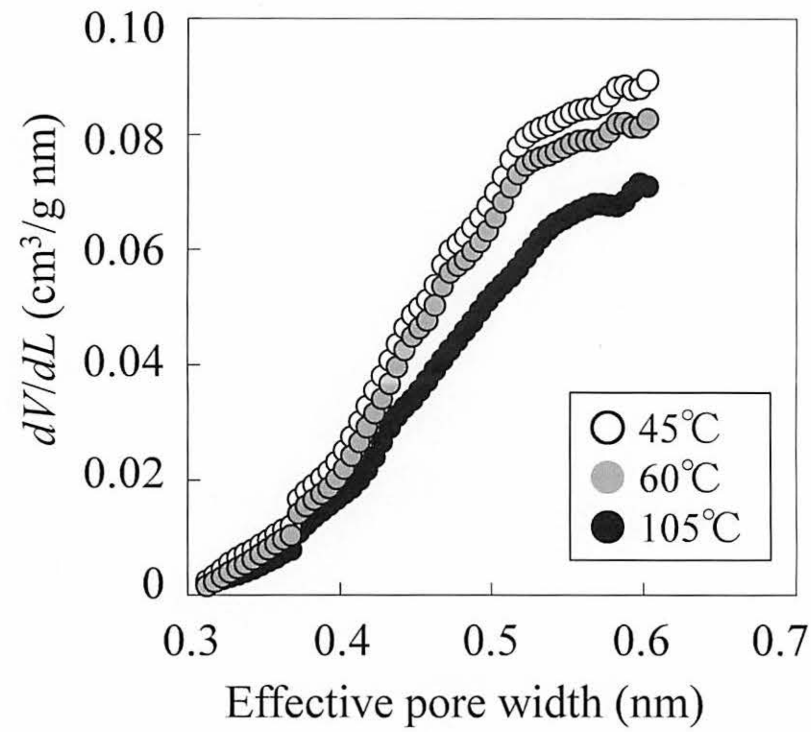

Fig. I-4 Influence of drying temperature on pore-size distribution determined by the adsorption of CO_2 with samples dried at different temperature.

wood is dried in the compressed state. However, the compressive deformation is almost recovered when the wood has absorbed the water again [6,7]. It is essential to fix the compressive deformation permanently when the compressed woods are used as materials. Therefore, to fix the compressive deformation permanently, many treatments such as heat treatment, steam treatment, and impregnation treatment of resin were tried [8-10]. Many studies to elucidate the mechanism of fixation have been done [11-14]. In addition, various studies about utilization of the compressed wood have been done. From these studies, it is clarified that tensile and bending strengths are increased with increase in compression rates [15,16].

While, it was reported that the dimensional stability of the compressed wood for the moisture was lower than that of uncompressed wood [17]. The dimensional stability of the compressed wood for the moisture is very important for use as materials. However, the study to improve the dimensional stability of compressed wood was not done. Therefore, swelling behavior of cell was observed in compressed wood and the relationship between cell shape and swelling behavior of cell was discussed. From the results obtained, it was considered that the mechanism of dimensional instability of the compressed wood by observing the shapes of water-saturated cells in detail.

The results obtained are as follows. The swelling in radial direction of wood compressed 50% was four times larger than that of uncompressed wood. In the visual fields of a microscope, four different deformation types of cell lumens were observed in the manner shown in Fig. I-5 and Fig. I-6. The increase in area of cell lumens is different among the four types. The increase in area of cell lumens is much different between S-shape cells and elongated shape of cells. In particular, the cells deformed to S-shape are hard to be increased the area of cell lumens by swelling. This result suggests that deformation types of cell lumens affect the swelling behaviors of compressed

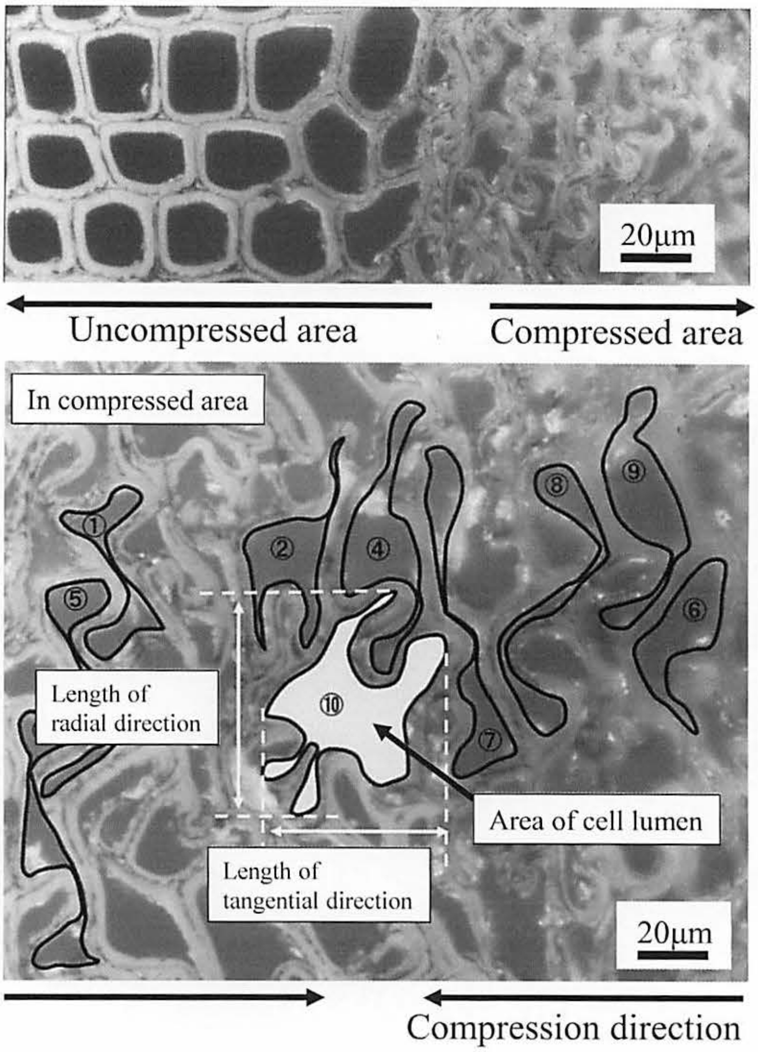

Fig. I-5 Images of measurements of compressed wood. Length and area of the specific cells were measured in uncompressed and compressed area before and after swelling. Top: cross sectional image of Japanese cedar which was used for the measurements. Bottom: example of measurement of cells in compressed area.

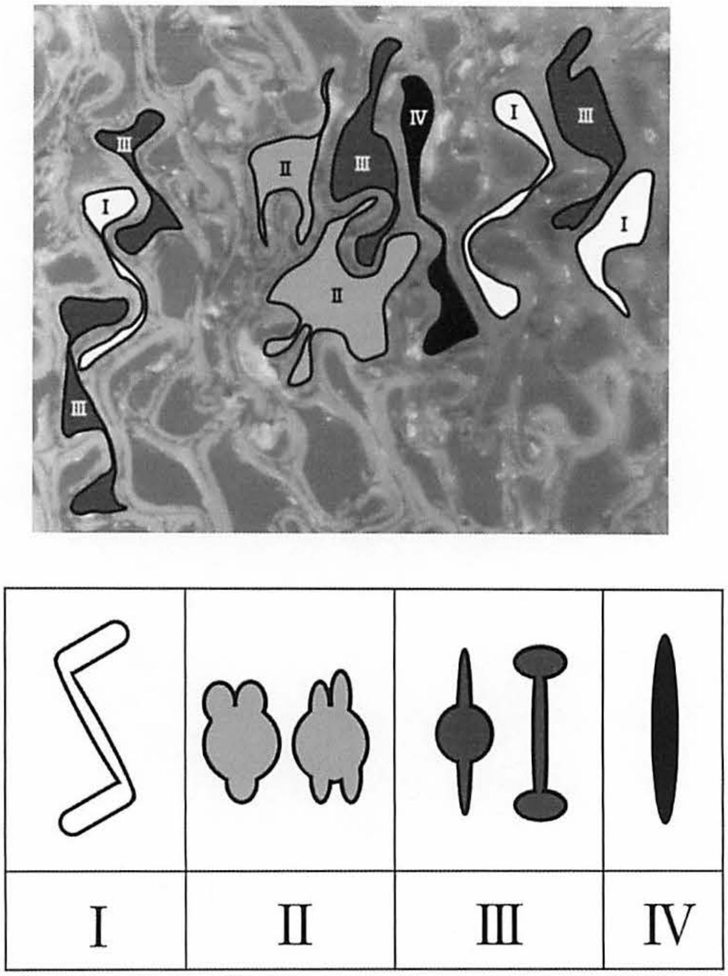

Fig. I-6 Image of measurement of cell shapes which was classified into four deformation types. Top: cross sectional image of Japanese cedar which was used for the measurements. Bottom: Image of four deformation types. S-shape cells were classified I. Cells having 3 or 4 projections were classified II. Cells with bulges in center or tip were classified III. Cells of elongated shape were classified IV.

cells. From the above results, it might be possible to improve the dimensional instability of the compressed wood by giving deformations as increasing the S-shape cells.

Tensile deformation properties of wood in lateral tension

To develop the processing technology such as deforming the wood like a chewing gum, some processing methods were tested in preliminary experiments. In the experiments, exceptional characteristic was shown in the deformation behavior in lateral tension.

Stress-strain curves of the specimens with different annual ring inclination against to tensile direction were shown in Fig. I-7. Strip shaped specimen with the annual ring inclinations of 0° (tangential direction), 45° (angle between tangential and radial direction), and 90° (radial direction) against the tensile direction were used. The specimen has dimensions of 1 mm (longitudinal direction) in thickness × 3.2 mm in width × 20 mm in length. The distance between specimen chucks was set to 10 mm. The deformation behavior and failure strain were different among the specimens with different annual ring inclination, and those characteristics were shown remarkably in higher temperature with wet condition. To investigate the cause of those deformation behaviors of water-swollen specimens at 80°C, the specimens were dried by keeping each tensile deformation in the load range that specimens were not broken. The cross sectional surface of the dried specimen was observed by scanning electron microscope (SEM). The some of SEM images are shown in Fig. I-8. From the images, different deformation patterns are found among the specimens with different annual ring inclination. Especially, in the specimen with the annual ring inclination of 45°, early wood cells show diamond shape. These different patterns of cell deformation should affect the deformation characteristic in lateral tension.

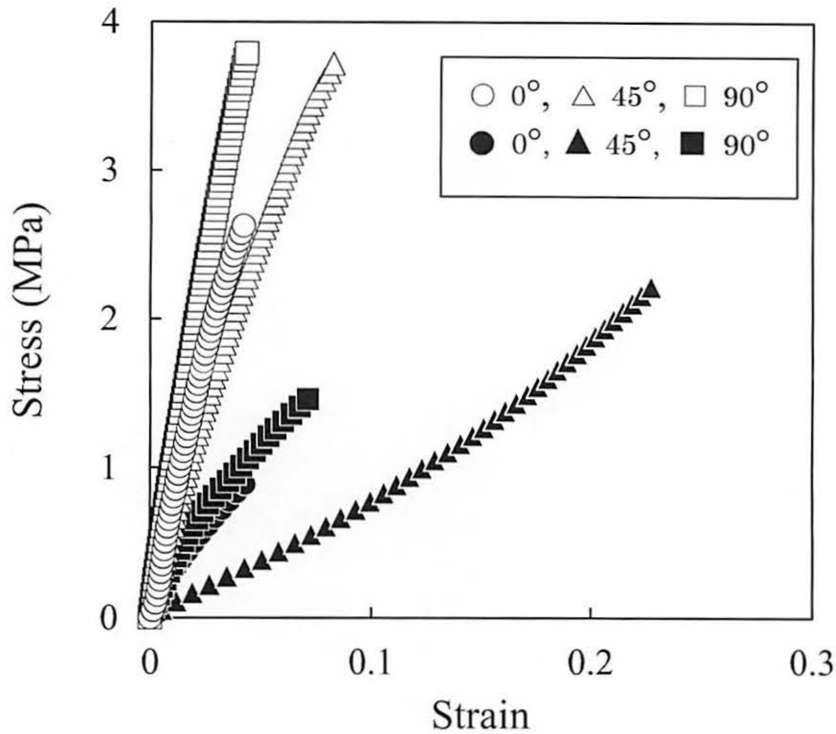

Fig. I-7 Stress-strain curves of the specimens with different annual ring inclination against to tensile direction. Open symbols are the results measured in wet condition at 25°C. Closed symbols are the results measured in wet condition at 80°C.

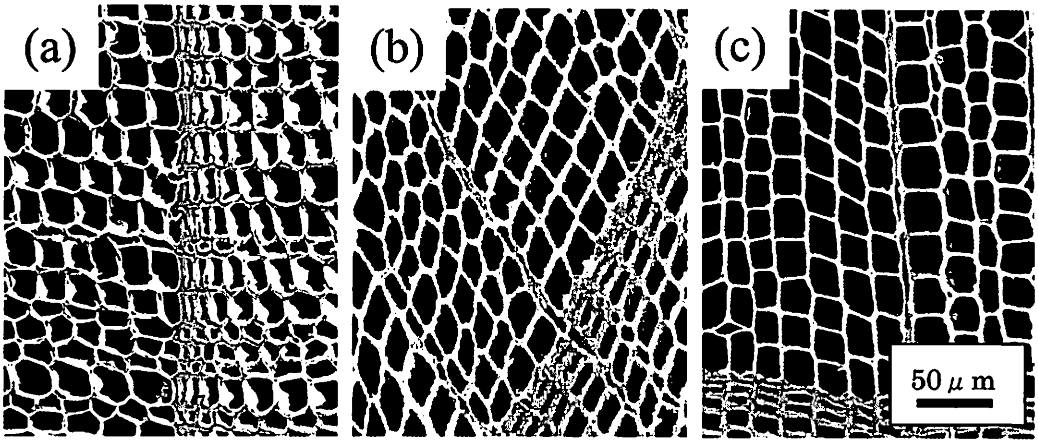

Fig. I-8 Scanning electron micrographs of dried Hinoki in cross section which kept the deformation given in water at 80°C. Micrographs show each specimen with annual ring inclinations of (a) 0°, (b) 45° and (c) 90° against tensile direction. The vertical direction of each photograph shows the tensile direction.

From the above results, it will be possible to increase the tensile deformation and develop the processing technology by considering temperature and moisture condition, and load method.

Furthermore, study about lateral tensile deformation of wood is insufficient and study that focused on increasing lateral tensile deformation was not found. If mechanisms of lateral tensile deformation and fracture and conditions which can increase the tensile deformation were elucidated, it might be possible to improve the deformation properties of wood and establish processing technologies utilizing tensile deformation positively. Therefore, basic study on controlling the deformation and fracture properties in lateral tension is carried out in following chapter.

CHPTERII

Factors affecting the deformation and fracture properties of wood in lateral tension

II-1 Introduction

A lot of researches on deformation processing of wood such as bending [18-23] and compressing [24-35] have been conducted. Recently, new processing methods of wood such as flow molding [36-40] and three-dimensional molding [41,42] are actively examined. However, these processing technologies of wood take advantage of the properties of compressing deformation or flow phenomenon between cells, and none of technologies for positively utilizing tensile deformation of wood are seen in domestic and foreign researches and industry. The following points can be considered as the reasons: Wood has a low ductility and when veneers are press molded into a tray or pasted on the curved surface, cracks often occur in large parts of the curvature. It is considered that the reasons for these problems are due to small lateral tensile deformation and strength. Therefore, first of all, it is necessary to understand the characteristics of the lateral tensile deformation and fracture of wood to improve the processability. And then, these studies should be applied to the various deformation processing technologies of wood.

In the some previous researches on the deformation or fracture properties in lateral tension, most researches discussed the elastic modulus or strength in some moisture and temperature condition, and few researches discussed lateral tensile strain [43-46]. On the other hand, Fujita reported the results of lateral tensile strain of wood for the purpose of elucidating the mechanism of cracks occurring in drying [47-49]. He investigated lateral tensile strains of wood greatly differ depending on the angle to annual ring. However, the moisture and temperature conditions of wood were far from the conditions for improving the deformation performance of wood. In addition, in these previous researches, wood species, measurement conditions, specimen sizes and conditioning methods are various because their aims were different each other. Therefore, it is hard to quantitatively and qualitatively evaluate the lateral tensile deformation or fracture property with a high precision by comparing those results.

In order to establish the bases of science and technology about tensile deformation processing, maximum tensile deformation and basic data as an index for controlling tensile deformation property should be measured. Therefore, the same wood species and similar shapes of specimens are measured accurately changing the conditions of moisture, temperature and inclination to annual ring because the results obtained can be compared each other. And it is necessary to elucidate the mechanism which can comprehensively explain the results associating with the measurement condition. Then, it will be possible to develop the processing technology such as increasing the maximum tensile deformation referring to the mechanism.

From the above, in this chapter, mechanical properties concerning to lateral tensile failure strain were measured in different inclination to annual ring, moisture and temperature for the similar shape specimens from various wood species having different anatomical characteristics. Then, from the results obtained, the deformation and fracture mechanisms were discussed in terms of thermal-softening and anatomy, and the important factors were discussed to increase the lateral tensile deformation.

II-2 Materials and methods

II-2-1 Materials for tensile breaking test of Hinoki

The specimens were determined to be of Hinoki (*Chamaecyparis obtusa* Endl.), and the block-shaped specimens with the annual ring inclinations of 0° (tangential direction), 45° (angle between tangential and radial direction), and 90° (radial direction) against the tensile direction were collected from the sapwood part. The reason for using Hinoki was that it was suitable as a model material for wood because the annual rings are uniform and dense. Thin cross sections with the thickness of about 0.1 mm (longitudinal direction) and with the dimensions of 3.2 mm in width × 20 mm in length were cut using a sliding microtome and used for the tensile breaking test. This shape of section was selected because the most reliable results were obtained within the load range of the test device in pretest, and the origin of the fracture could be clearly observed at a cellular level. Furthermore, the thin-sections were prepared with extreme care not to generate scratches or knife marks which could be a starting point of the fracture.

II-2-2 Tensile breaking test of Hinoki

A thermal mechanical testing machine (manufactured by Seiko Instruments Co., Ltd.; TMA/SS6100) was used for tensile breaking tests. The measurements were carried out in water or in dried air kept at a constant temperature of 25°C and 80°C. The distance between specimen chucks was set to 10 mm, and the specimens were measured with the applied load rate of 1 N/min until they were fractured. The measurement results were adopted only for the specimens fractured roughly at their central portion, whereas those showing exceptional deformation behaviors were excluded. The number of specimens whose results were adopted was 5 pieces per condition.

II-2-3 Materials of tensile breaking test and dynamic viscoelastic measurement of various wood species

The following wood species were selected in this examination considering anatomical characteristics of the annual ring: Hinoki (*Chamaecyparis obtusa* Endl.) from softwood, Yamaguruma (*Trochodendron aralioides* Sieb. et Zucc.) most of its xylem is composed of tracheids instead of vessels, Honoki (*Magnolia obovata* Thunb.) from diffuse-porous hardwood, Keyaki (*Zelkova serrata* Makino) from ring-porous hardwood and Arakashi (*Quercus glauca* Blume) from radial-porous hardwood. The density and annual ring width are as follow; 0.32 g/cm^3 and 1.1mm (Hinoki), 0.57 g/cm^3 and 0.8 mm (Yamaguruma), 0.46 g/cm^3 and 1.7 mm (Honoki), 0.82 g/cm^3 and 2.0 mm (Keyaki), 0.70 g/cm^3 and 3.0mm (Arakashi). Two sizes of specimens were prepared. The one was used in the tensile breaking test and the size of specimen was the same to II-2-1. The other was used in the dynamic viscoelasticity measurement and the size of specimen was 4 mm (R), 20 mm (T), 2 mm (L).

II-2-4 Tensile breaking test and dynamic viscoelasticity measurement of various wood species

In the tensile breaking test, the specimens of each species were measured in the same methods of II-2-2. The measurements were carried out only in water kept at a constant temperature of 25°C and 80°C. The measurement results were adopted only for the specimens fractured roughly at their central portion, whereas those showing exceptional deformation behaviors were excluded. The number of specimens whose results were adopted was 5 pieces per condition. The fracture specimens were observed by scanning electron microscope (SEM).

In the dynamic viscoelasticity measurement, an automatic dynamic viscoelastometer (Seiko Instruments Co. Ltd., DMS6100) was used. The measurements were conducted in water with 5 mm span and the measurement frequencies of 0.5, 1, 2, 5 and 10Hz. The data was obtained during the second temperature rising after the temperature fell from 98°C to 20°C at 1°C /min.

II-3 Results and discussion

II-3-1 Effects of tensile direction to the annual rings, moisture, and temperature on mechanical properties of Hinoki (*Chamaecyparis obtusa*) in lateral tension

Stress-strain curves

First, to examine the influence of annual ring inclination, moisture, and temperature on lateral tensile deformation property, deformation behavior of Hinoki was measured because of its typical anatomical character of annual ring. The typical stress-strain curves were shown in Fig. II-1 and Fig. II-2. Fig. II-1 shows the results measured in dried air at 25°C and 80°C and Fig. II-2 shows the results measured in water at 25°C and 80°C. From Fig. II-1, stress-strain curves of specimens with the annual ring inclination of 0° and 90° were rather linear and the slopes of these curves was almost the same at each temperature. Whereas, in the specimen with the annual ring inclination of 45°, the slopes of stress-strain curves were more gradual and the deformations at fracture were larger than those with the annual ring inclination of 0° and 90°. Fig. II-2 shows that the slopes of stress-strain curves at 25°C are steeper than those of 80°C, and these tendencies are seen in all of the specimens. Especially, in the specimen with the annual ring inclination of 45°, the deformations at fracture at 25°C and 80°C were larger than the other specimens, and failure strain was about 20% at 80°C. Furthermore, the behaviors of stress-strain curves of the samples with the annual ring inclination of 45° were different from the other

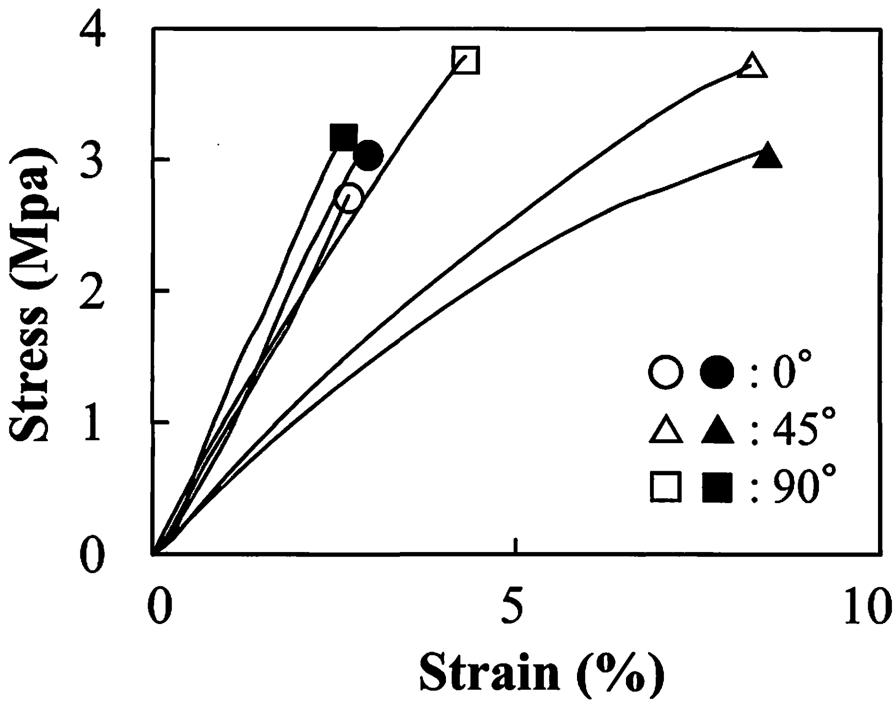

Fig. II-1 Typical stress-strain curves of specimens with the annual ring inclinations of 0°, 45° and 90° against the tensile direction measured in the dry condition at 25°C or 80°C. Open symbols are dry conditions at 25°C, closed symbols are dry conditions at 80°C. Symbols mean failure points.

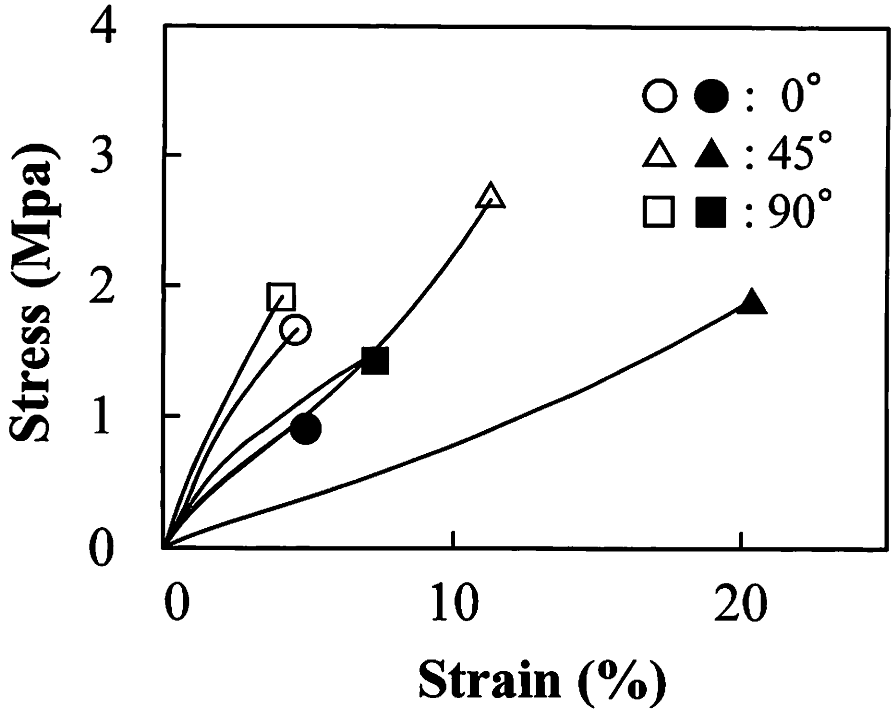

Fig. II-2 Typical stress-strain curves of specimens with the annual ring inclinations of 0°, 45° and 90° against the tensile direction measured in the wet condition at 25°C or 80°C. Open symbols are wet conditions at 25°C, closed symbols are wet conditions at 80°C. Symbols mean failure points.

specimens and the slopes of them were steeper with increasing the tensile strain. The reason for the specimens with the annual ring inclination of 45° having the characteristic behavior of stress-strain curve was considered as follows in terms of anatomy of Hinoki.

In the specimen with the annual ring inclination of 0°, late wood with thick cell wall and flat shape is arranged in parallel with the tensile direction. The cell wall of late wood is thicker than that of early wood and hard to deform, therefore, tensile stress applied to late wood cells is larger than early wood cells. Then, ray tissues with thin cell wall arranged in a direction perpendicular to the late wood are receiving large tensile stress in the lateral direction. Therefore, the deformation of specimen with the annual ring inclination of 0° would be restricted by late wood, and strength would depend on the strength of cell wall of ray tissues or the adhesive strength between the cells in late wood. In observation of fracture surface by an electron microscope, fractures tended to occur along the ray tissue and propagated cracks among ray tissues shown in Fig. II-3(a).

In the specimen with the annual ring inclination of 90°, ray tissue is arranged in parallel to the tensile direction. Therefore, it is considered that the deformation of specimen is restricted by ray tissue with small failure strain in its longitudinal direction and large stress works the ray tissue. In addition, a large stress should work the ray tissue arranged in the section of early wood which close to the annual ring boundary with low resistance to deformation because late wood and early wood are arranged in series. This occurs the fracture at this position. Observation by SEM revealed that fractures tended to occur along the annual ring boundary as shown in Fig. II-3(c). From the above, in the specimen with the annual ring inclination of 0° and 90°, it is considered that ray tissue and late wood restrict the deformation of specimen and the mechanical property of these tissues with small strain is reflected in tensile deformation

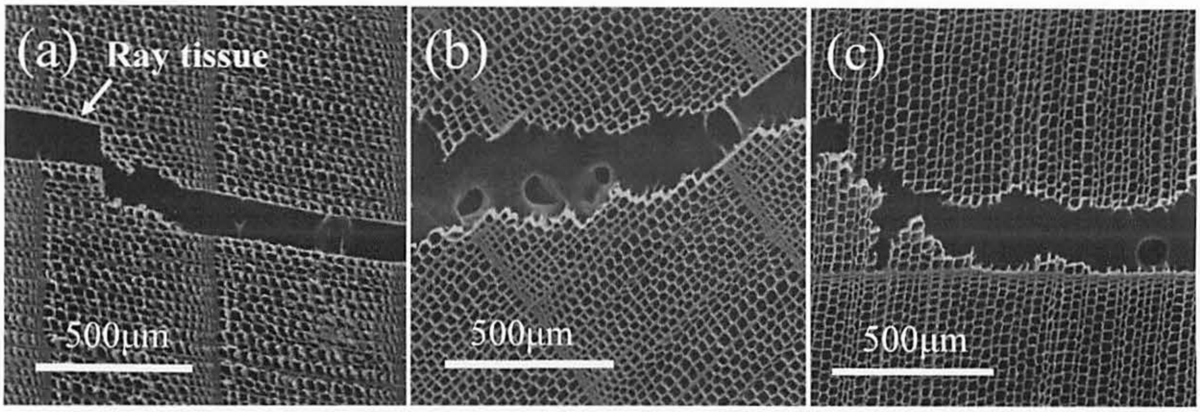

Fig. II-3 Scanning electron micrographs of fractured sections of various wood species in lateral tensile tests in dry condition at 25°C. Micrographs show each specimen with annual ring inclinations of (a)0°, (b)45° and (c)90° against the tensile direction. The vertical direction of each photograph shows the tensile direction.

property.

On the other hand, in the specimen with the annual ring inclination of 45°, ray tissue and late wood are arranged in the direction inclined at 45° toward the tensile direction. Therefore, these tissues do not restrict the deformation so much, and the early wood cells having an almost square shape relatively easily deform like a diamond shape by tensile force acting to the cells during increasing of tensile stress [50]. The fracture tended to occur along the ray tissues or intercellular layers between cells in the direction inclined at 45° toward the tensile direction because maximum stress worked in this direction shown in Fig. II-3(b). From the above, the angle between ray tissues or late wood and the tensile direction was reducing with increasing in tensile strain and tensile stress was increased rapidly because the tissues with higher elastic modulus had to receive a larger stress.

The results mentioned above suggest that the difference in the deformation mode of tissue affects the behavior of stress-strain curves.

Mechanical properties in lateral tension measured in several conditions

Next, to examine the influence of annual ring inclination on lateral tensile deformation and fracture, tensile failure strain, strength and elastic modulus were shown in Fig. II-4. In the specimen with the annual ring inclination of 45°, failure strains were the largest in all of the measurement conditions, and failure strains in wet condition were larger than those in dry condition in all of the specimens. The failure strains obtained at the annual ring inclination of 45° measured in wet condition at 80°C were about 9 times larger than the failure strains obtained at the annual ring inclination of 90° measured in dry condition at 80°C. The strengths in dry condition were larger than those in wet condition, and the strengths in dry condition were larger in order of the specimen with the annual ring inclination of 90°, 45° and 0°. In contrast, the strengths in wet

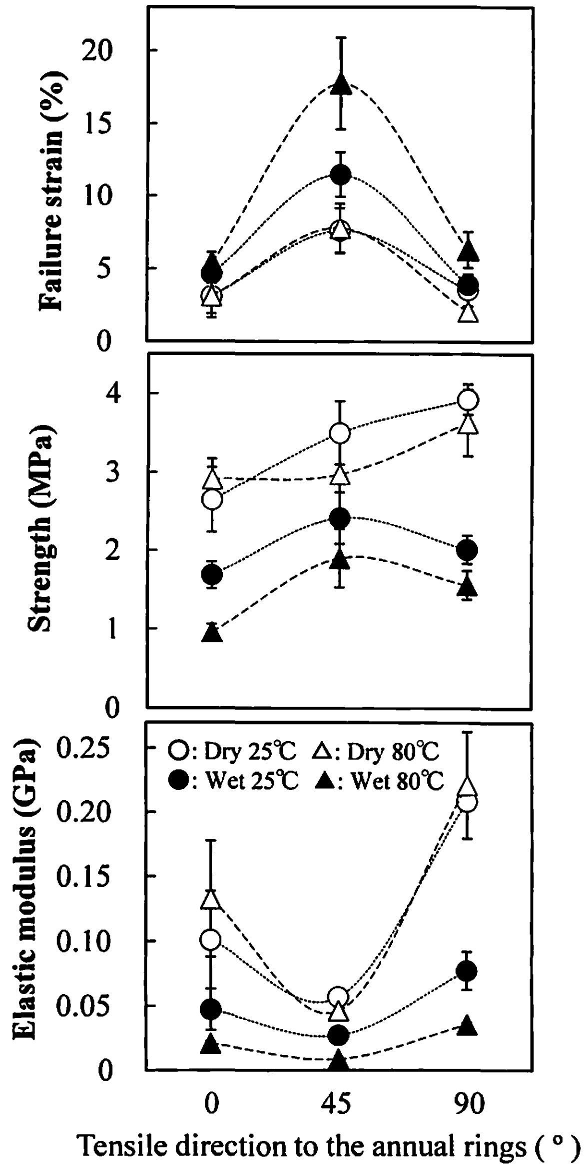

Fig. II-4 Mechanical properties in tension of lateral wood specimens with different annual ring inclination against the tensile direction. Open circle is dry condition at 25°C, open triangle is dry condition at 80°C, closed circle is wet condition at 25°C, closed triangle is wet condition at 80°C. Symbols mean the average value and error bars mean 95% confidence interval.

condition were larger in order of the specimen with the annual ring inclination of 45°, 90° and 0°. The elastic moduli in wet condition were smaller than those in dry condition. In all measurement conditions, the elastic moduli were larger in order of the annual ring inclination of 45°, 0° and 90°. In the specimens with the annual ring inclination of 90°, the cause that both of the strengths and elastic moduli were larger than those of the annual ring inclination of 0° was considered that mechanical properties of ray tissue in longitudinal affected the results.

Influence of moisture and temperature on tensile failure strain

To clarify the factors which increase the tensile deformation, the influence of moisture and temperature on tensile failure strain, meaning the amount of deformation before fracture, was discussed. The results of tensile failure strain are shown in Fig. II-5. In the specimens with the annual ring inclination of 0° and 45°, the failure strains were almost the same regardless of the temperatures in dry condition, however, in wet condition the failure strains at 80°C were larger than that of 25°C. The cause of these results can be discussed in terms of the molecular motion of wood components. It is known that oven-dried wood has mechanical relaxation caused by micro-Brownian motion of amorphous components above 200°C [51,52]. In addition, it is confirmed that elastic modulus is decreased and fluidity is increased by activating the molecular motion of wood components during the temperature rise from 100°C to 200°C [52,53]. Hence, the results of the elastic modulus and failure strain were almost the same at the 25°C and 80°C in dry condition, as shown in Fig. II-4. And, similar results for the elastic modulus and failure strain at 25°C and 80°C were attributable that the micro-Brownian motion of amorphous components did not occur sufficiently within the range of temperature from 25°C to 80°C, considering the results mentioned above reports.

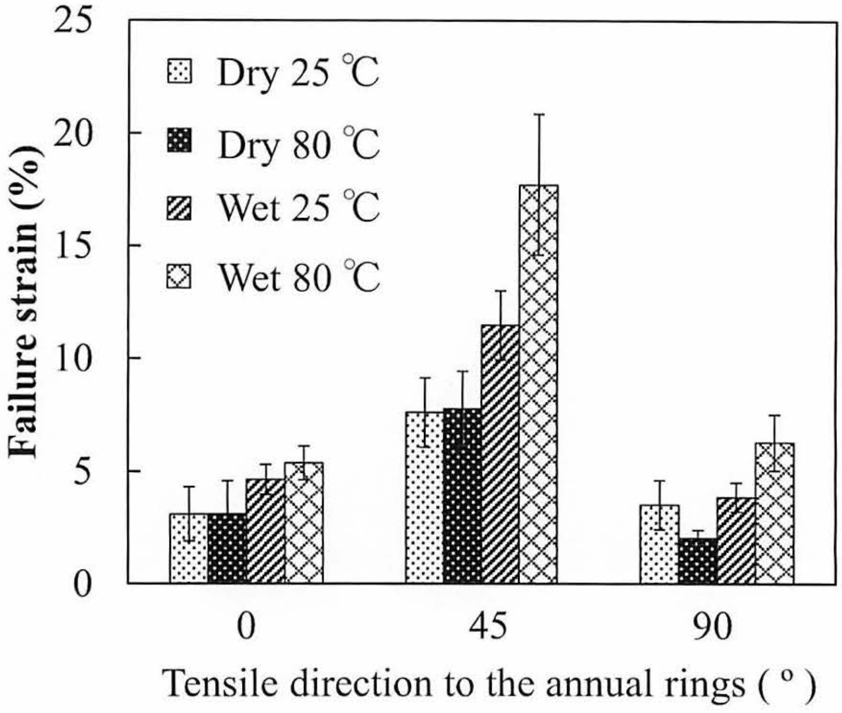

Fig. II-5 Averages of failure strains in tension of lateral wood specimens with different annual ring inclination against the tensile direction. Error bars mean 95% confidence interval.

Whereas, in the specimen with the annual ring inclination of 90° in dry condition, failure strain was at 80°C significantly smaller than those at 20°C. This is possibly caused by fine defects which could be starting point during drying process. However, this discussion is beyond the objective of this study, so we will not make any mention about the cause.

In order to discuss the influence of moisture on tensile failure strain, the increase rate of failure strain is shown in Fig. II-6. Relative failure strain means the value in wet condition relative to that in dry condition. The increase rate from dry to wet condition at 25°C was about 10% in the specimen with the annual ring inclination of 90°, and 50% in the specimen with the annual ring inclination of 0° and 45°C. While, the increase rates at 80°C were 70%, 130% and 200% in order of the specimen with the annual ring inclination of 0° , 45° and 90°, respectively. Incidentally, the large increase rate of failure strain at the annual ring inclination of 90° is considered to be caused by the low failure strain at 80°C in dry condition as shown in Fig. II-5. From the results in Fig. II-6, the reason for increasing of failure strain affected by moisture within the temperature range from 25°C to 80°C can be discussed as follows.

Thermal-softening property differs depending on the moisture content. As previously mentioned, it is known that oven-dried wood has mechanical relaxation caused by micro-Brownian motion of amorphous components above 200°C [51,52]. When the moisture content increases, interaction between molecular chains composed by the wood components weakens, and the temperature of mechanical relaxation, that is, thermal-softening temperature decreases. Therefore, the mechanical relaxation caused by micro-Brownian motion of lignin appears at around 80°C in water-saturated wood [54,55]. In this measurement, molecular motion of wood components was not active because the temperature of 25°C was the lower than the thermal softening temperature. On the other hand, molecular motion of wood components is

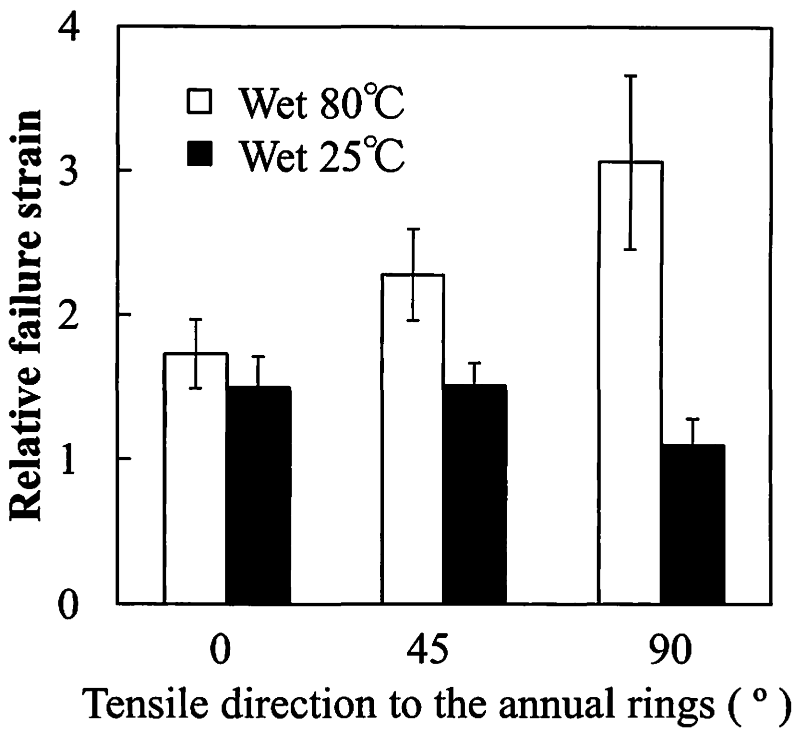

Fig. II-6 Effect of moisture and temperature on failure strain of lateral wood specimens with different annual ring inclination against the tensile direction. Relative failure strain means the value in wet condition relative to that in dry condition. Error bars mean 95% confidence interval.

active in wet condition at 80°C for wood, therefore, it is considered that failure strain increased because of decrease in elastic modulus and increase in fluidity. The failure strain would be also affected by micro-fibril angle or component composition, however, it is confirmed that the thermal-softening property in lateral direction is uniform regardless of the annual ring inclination [56]. Therefore, the change in failure strain affected by moisture and temperature would be explained to a certain extent in terms of thermal-softening. The results mentioned above can be explained as follows. The reason for larger strains at 80°C in wet condition than that of dry condition is attributable to more active molecular motion of wood components in wet than dry condition at 80°C. And in the specimen in wet condition at 25°C, the resistance to deformation reduced because hydrogen bonds created in wood components during dry condition was cut and water molecule exists between molecular chains expend the distance of molecules, as the result, elastic modulus decreases. In general, the strengths are also reduced with increasing moisture content [57-60], however, failure strains in wet condition were larger than those of dry condition at 25°C. This suggests that the effect of reduction in resistance for deformation to the failure strains in lateral tension is larger than that of the strengths.

II-3-2 Effects of anatomical characteristics and thermal-softening on mechanical properties of various wood species in lateral tension

From the discussion of the previous section, the deformation of cell shape and the activation of molecular motion of wood components are important for increasing the deformation further in lateral tension. Then, in this section, to clarify the deformation and fracture mechanisms in detail, mechanical properties concerning to lateral tensile failure strain were measured in various species of woods having different anatomical characteristics. And then, the influences of tissue structure, density and thermal-softening on mechanical properties in lateral tension were discussed.

Stress-strain curves in several wood species

The typical stress-strain curves of each wood species measured in water at 25°C and 80°C are shown in Fig. II-7. In all specimens expect for Arakashi, the slopes of stress-strain curves increased in order of the annual ring inclination of 90°, 0° and 45° both at 25°C and 80°C. The strength of hard wood measured tended to be larger than those of Hinoki at the same temperature. In Hinoki, the failure strain of the specimen with the annual ring inclination of 45° was larger than that of the specimen with the annual ring inclination of 0° and 90°. Similar tendency was found in Yamaguruma from hardwood without vessel and Honoki from diffuse-porous hardwood. However, the failure strains of Arakashi and Keyaki with regularly arrayed vessels were not largest in the specimen with the annual ring inclination of 45°. In Arakashi, the failure strains of the specimen with the annual ring inclination of 45° and 0° were larger than those of the specimen with the annual ring inclination of 90°. In the case of Keyaki, much difference was not found in the failure strains among different annual ring inclinations. In the specimens with the annual ring inclination of 45° in Hinoki and Yamaguruma, the failure strains were about 12% and those of Honoki were about 9% at 25°C. On the other hand, the failure strains of Hinoki at 80°C were about 20%, however, much difference was not found between at 25°C and 80°C in Yamaguruma and Honoki.

Focusing on the behaviors of stress-strain curves, the slopes of these curves for Hinoki with the annual ring inclination of 45° became steeper with increasing the tensile strain. This behavior was caused by increase in share deformation of early-wood cells with increasing tensile stress [50]. This

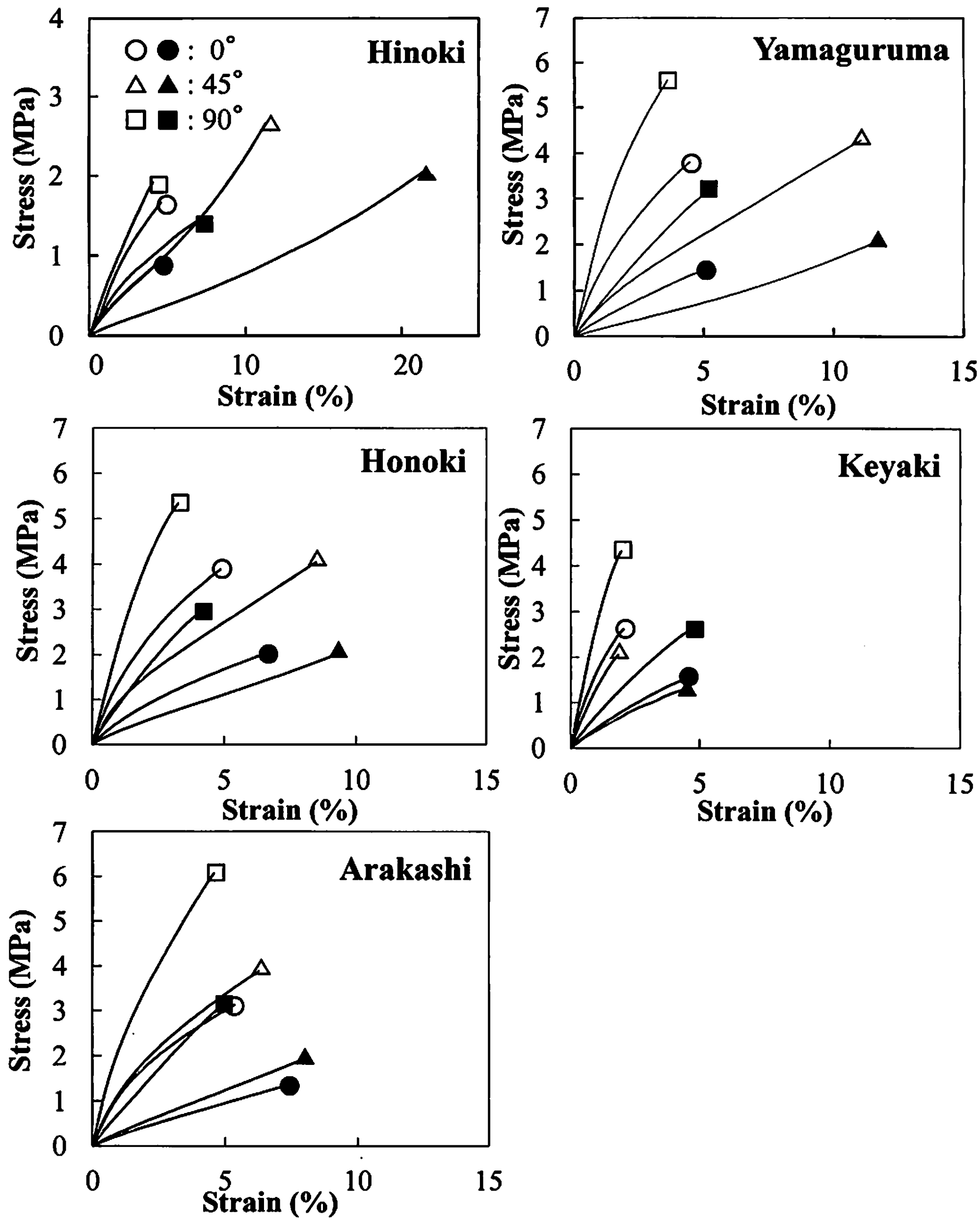

Fig. II-7 Typical stress-strain curves of specimens with the annual ring inclinations of 0°, 45° and 90° against the tensile direction measured in the wet condition at 25°C or 80°C. Open symbols are wet conditions at 25°C, closed symbols are wet conditions at 80°C. Symbols mean failure points.

exceptional behavior was also found in the specimens with the annual ring inclination of 45° at 80°C in Yamaguruma without vessel. However, this exceptional behavior was not found in the other hard wood measured. The slopes of curves in the specimens with the annual ring inclination of 45° in Honoki were linear compared with the other specimens.

The results mentioned above suggest that the behaviors of stress-strain curves are affected by the difference in the deformation mode of tissue. In other words, deformation mode in lateral tension is probably different between the wood species with vessel and without vessel, or between those with diffuse-porous and with regularly arrayed porous.

Lateral tensile properties in several wood species

Next, to discuss the influence of anatomical characteristics on lateral tensile deformation and fracture in detail, tensile failure strain, strength and elastic modulus were shown in Fig. II-8. The failure strains obtained at 25°C were larger in the order of the annual ring inclination of 45°, 0° and 90° for all wood species except for Keyaki. The failure strains at annual ring inclination of 45° were largest for Hinoki, and then decreased in the order of Honoki, Arakashi and Keyaki, and large differences in failure strain at annual ring inclination of 0° and 90° were not found among the wood species. Additionally, as well as the results obtained at 25°C, in the specimen with the annual ring inclination of 45°, failure strains at 80°C were larger than those with the annual ring inclination of 0° and 90° for all of the wood species except for Keyaki. The failure strain at the annual ring inclination of 45° was the largest in Hinoki. In contrast, in the specimen with the annual ring inclination of 0°, the largest failure strain was found in Arakashi. The strengths at the annual ring inclination of 90° were larger than those at the annual ring inclination of 0°, both at 25°C and 80°C for all of the wood species except Hinoki. In the specimen with the annual ring

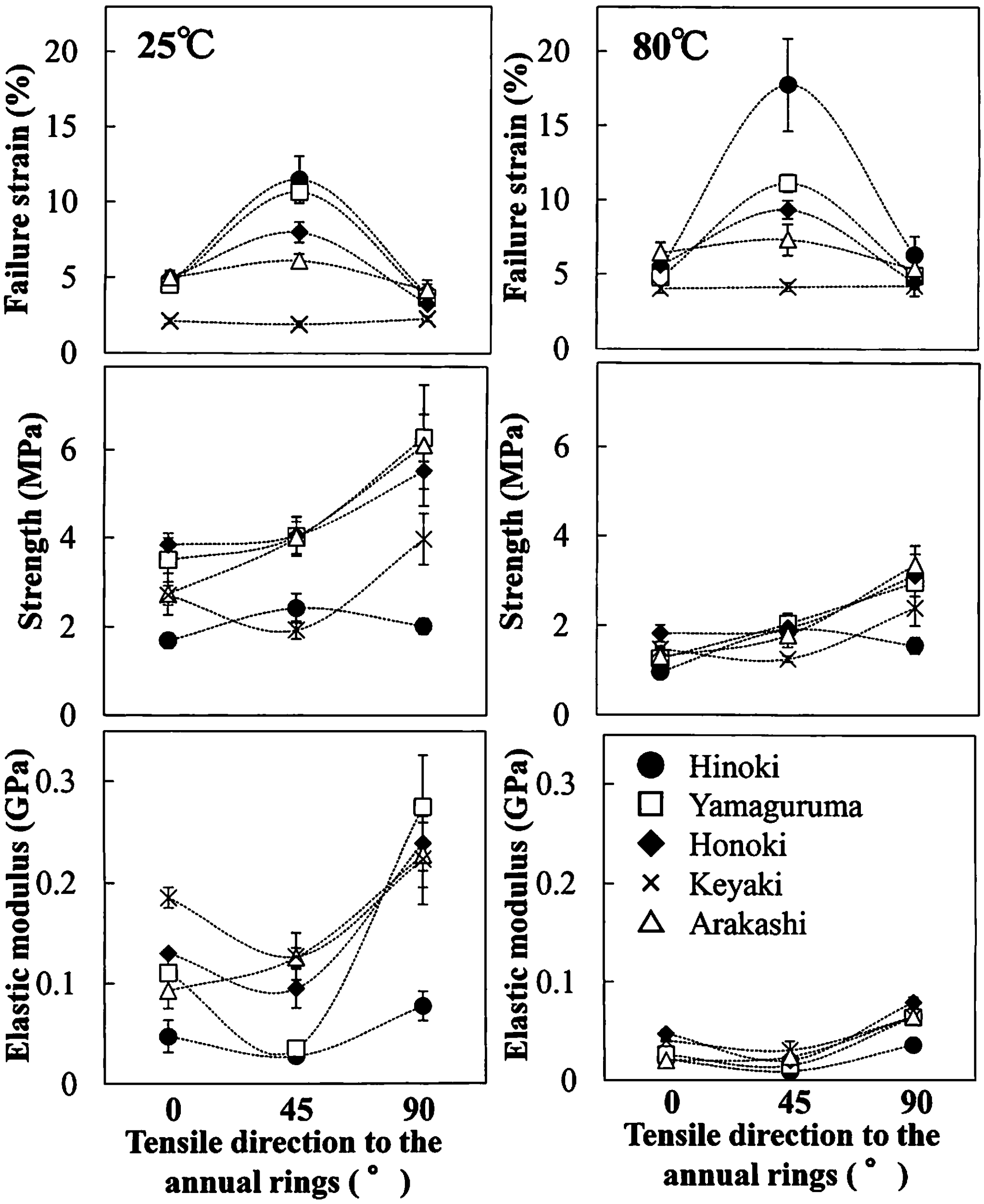

Fig. II-8 Mechanical properties of wood specimens with different annual ring inclination against the tensile direction. Closed circle is Hinoki, open square is Yamaguruma, closed square is Honoki; closs symbol is Keyaki, open triangle is Arakashi. Symbols mean the average values and error bars mean 95% confidence interval.

inclination of 90°, it is considered that the mechanical property of ray tissue is reflected in tensile deformation property, because ray tissue is arranged in parallel to tensile direction. Generally, soft wood has a single-row ray tissue and hard wood has multi-row ray tissue [61], and the percentage of ray tissue contained in hard wood is larger than that in soft wood [62]. The elastic moduli at 25°C and 80°C were smaller in order of the specimen with the annual ring inclination of 45°, 0° and 90° except Arakashi.

Influence of density on tensile failure strain

The wood species used in this examination were selected in consideration of anatomical features of the annual ring. Meanwhile, density and thermal-softening temperature also vary among the wood species. In this part, firstly, the effect of density on failure strain is discussed in Fig. II-9. From the relationship between density and failure strains obtained in wet condition at 25°C, failure strains at the annual ring inclination of 0° were about 3-5% regardless of density and remarkable variations in failure strain were not observed. At the annual ring inclination of 90°, remarkable variations in failure strains to density was also not observed. However, in the specimen with the annual ring inclination of 45°, the tendency of reduction in the failure strains was clearly observed with increasing in density. Also in the measurement condition at 80°C, remarkable reduction in failure strain with increasing density was observed only for the annual ring inclination of 45°. The above results suggest that the failure strain clearly decreases with increasing density only at annual ring inclination of 45°, however, in the specimens with the annual ring inclination of 0° and 90°, the failure strain cannot be explained simply only by a factor of density. The reason of the results mentioned above is considered as follows.

In the specimen with the annual ring inclination of 0°, late wood with thick

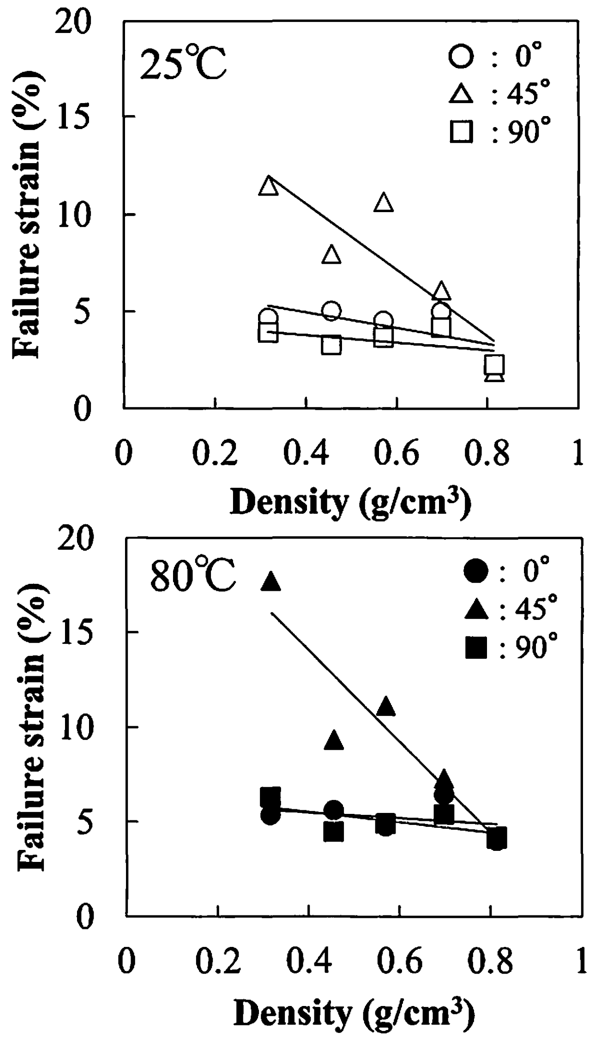

Fig. II-9 Relationship between density and failure strain of specimens in various wood species measured in water at 25°C or 80°C. Open symbols are wet condition at 25°C. Closed symbols are wet condition at 80°C. Correlation coefficient of each sample obtained by the measurement in water at 25°C; open circle: r = -0.42, open triangle: r = -0.75, open square: r = -0.20 and 80 °C; closed circle: r = -0.03, closed triangle: r = -0.83, closed square: r = -0.43.

cell wall and flat shape is arranged in parallel to the tensile direction and ray tissue is arranged in a direction perpendicular to the tensile direction. Therefore, the deformation of specimen with the annual ring inclination of 0° should be restricted by late wood, and strength probably depends on the strength of cell wall of ray tissues or the adhesive strength between the cells in late wood, and thus, failure strains should be small due to those week tissues. In the specimen with the annual ring inclination of 90°, ray tissue is arranged in parallel to tensile direction and ray tissue should restrict the deformation of specimen. On the other hand, in the specimen with the annual ring inclination of 45°, ray tissue and late wood are arranged in the direction inclined at 45° toward the tensile direction. Therefore, these tissues do not restrict the deformation so much and share deformation of cells with increasing tensile stress is occurred [50]. This share deformation of cells is caused by reducing the areas of cell lumens under the observation on cross sectional surface. The cells with thick wall in high density wood were hard to deform, therefore, reduction of the failure strain was shown with increasing density. The results mentioned above suggest that the deformation facility of cell shape affects the increase of lateral failure strain. In addition, it is also suggested that density of wood affects both promotion and restriction of deformation of cell shape for the specimen with the annual ring inclination of 45° in which cells showed share deformation.

Influence of thermal- softening on tensile failure strain

In the previous section, it is clarified that the activation of molecular motion of wood components are important for increasing the deformation further in lateral tension for Hinoki. Therefore, failure strains of hard wood should be also increased in wet condition at 80°C. Then, in this part, to clarify the effect of anatomical characteristics on the deformation facility of cell shape caused by softening of cell walls, the relationship between failure strain and thermal-softening was discussed.

The composition of wood component [63] and thermal-softening temperature of lignin [64] are different among the wood species. Although, the Figure is not shown in this section, dynamic viscoelasticity was measured and the peak temperatures of loss tangent at frequencies of 0.5Hz were as follows; 89°C in Hinoki, 76°C in Yamaguruma, 82°C in Honoki, 82°C in Arakashi and 74°C in Keyaki. While molecular motion of wood components of each wood species is almost active, degree of the activation in lignin molecules should be slightly different among wood species. Then, decrease of the elastic modulus due to the softening of the wood was defined as an index for the degree of activation, the relationship between relative failure strain and relative elastic modulus was shown in Fig. II-10. Each relative value means the value in wet condition at 80°C relative to that in wet condition at 25°C. From the Fig. II-10, temperature increase from 25°C to 80°C causes increase of the failure strain regardless of wood species, because relative failure strains are more than 100% in all of the specimens in wet condition. However, the certain tendency was not observed in the relationship between relative failure strain and relative elastic modulus. Generally, deformation increases, if elastic modulus decreases due to softening of wood. Thus, the cause of that the relationship between relative failure strain and relative elastic modulus was not observed in this measurement was considered as follows.

First, from the viewpoint of the anatomy of the wood, ray tissue in their longitudinal direction and vessel and tracheid in lateral direction receive tensile stress simultaneously in the specimen with the annual ring inclination of 90°. In this case, deformation amount of these tissues should be different because elastic modulus in load direction is different in each tissue. The tissues with higher elastic modulus restrict the deformation in tensile direction of the tissues with lower elastic modulus which is adjacent in the direction of perpendicular to

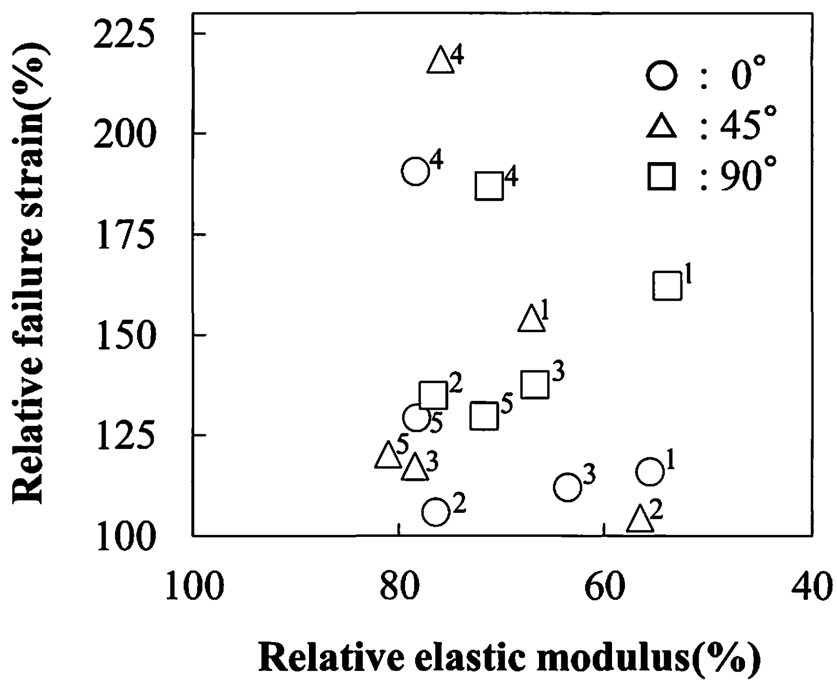

Fig. II-10 Relationship between relative elastic modulus and relative failure strain in various wood species. Each relative value means the value in wet condition at 80°C relative to that in wet condition at 25°C. The number means each wood species, 1: Hinoki, 2: Yamaguruma, 3: Honoki, 4: Keyaki, 5: Arakashi.

tensile direction. As a result, the specimen possibly has a complex deformation behavior. Now, considering the above results from the viewpoint of the thermal-softening of the wood. The chemical composition of wood components is different in each tissue and percentage of lignin contained in intercellular layer is larger than that in secondary wall [65,66]. In addition, in the previous report, reduction of elastic modulus due to softening was observed remarkably in the lateral direction of wood in which the mechanical properties of matrix component is largely reflected [56]. Thus, if the wood species are different, the extent of deformation is different depending on the difference of tissue structure. As the above mentioned, the correlation between increase of failure strain and decrease of elastic modulus was not observed, one of reasons is attributable to complex effects of anisotropy of tissue and inhomogeneity of reduction of elastic modulus in specimen on the lateral tensile deformation.

Observation of fracture surfaces in several wood species

To clarify the effect of anatomical characteristics on fracture in lateral tension, fracture surface of the specimens after the tensile breaking test was observed. The specimens were observed by SEM. The fracture tendency was similar between the wet conditions at 25°C and 80°C. Therefore, typical fracture surface of specimen measured in wet condition at 25°C was shown in Fig. II-11.

In the specimen with the annual ring inclination of 0°, fracture tended to occur along the ray tissue and propagated themselves cracks among ray tissues. From the micrographs in Keyaki, large-diameter vessels were considered as the starting point of fracture, because the vessels were fractured in half. Arakashi also has relatively large-diameter vessels, however, fractured vessels in half were not observed.

In the specimen with the annual ring inclination of 45°, fracture along the

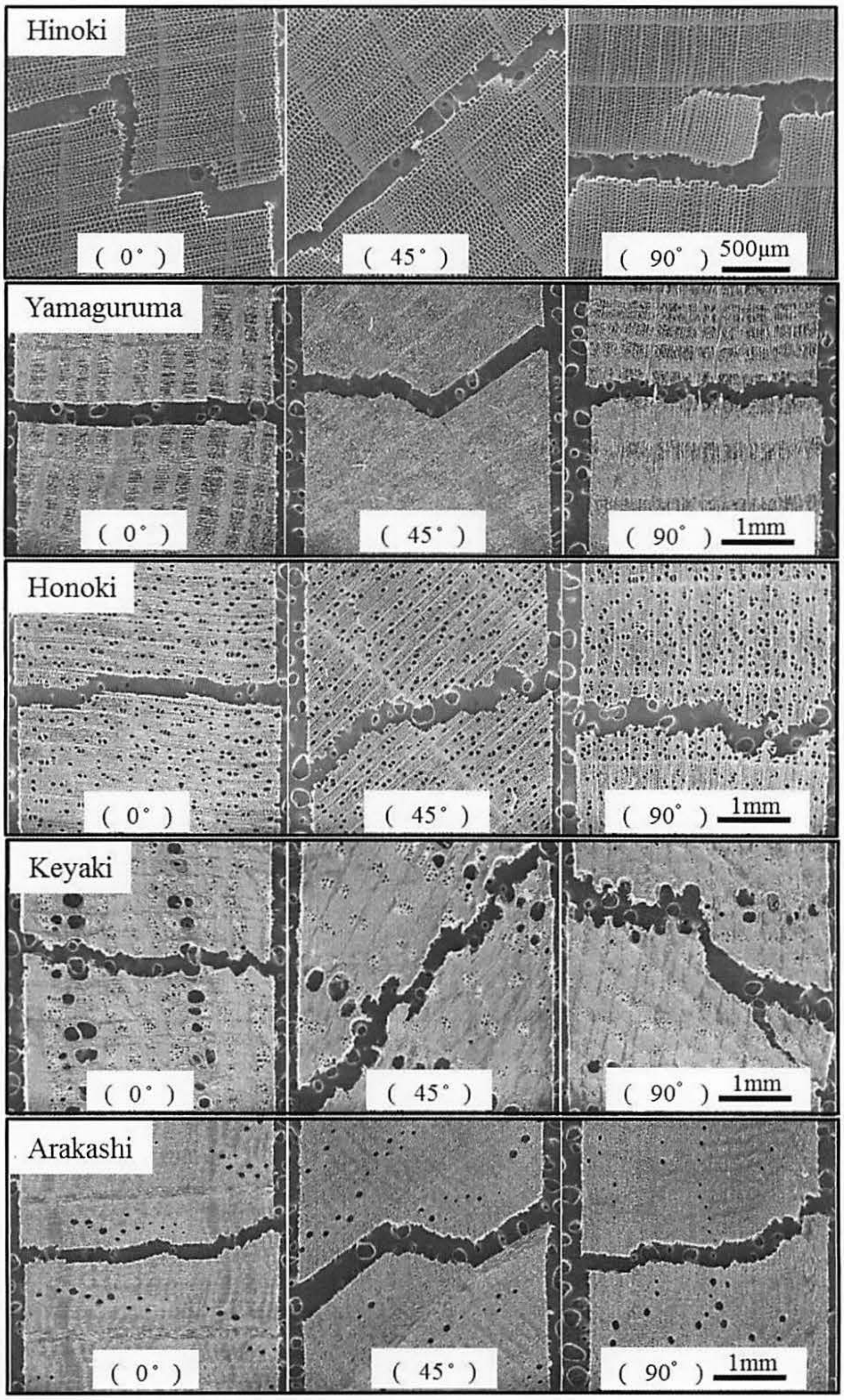

Fig. II-11 Scanning electron micrographs of fractured sections of various wood species in lateral tensile tests in water at 25°C. Micrographs show specimens with annual ring inclinations of 0°, 45° and 90°, respectively. The vertical direction of each photo shows the tensile direction.

ray tissues was clearly observed in Yamaguruma and Arakashi. However, in Honoki and Keyaki, fracture occurs not only along the ray tissue but also vessels with large or small diameter and the annual ring boundary. As the above mentioned, the fracture in the specimen with the annual ring inclination of 45° tend to occur along the ray tissues primarily.

In the specimen with the annual ring inclination of 90°, fractures were occurred parallel to an annual ring in Honoki and Yamaguruma, and fracture tended to occur along the early wood in annual ring boundary in Yamaguruma. The fracture along the early wood was caused by receiving the large stress, because quantity of wood substance is small in the early wood. The fracture along the axial parenchyma cells that continuous like the wave in the tangential direction [67] was shown in micrograph in Arakashi. And the fracture along the vessels arranged in tangential direction was shown in Keyaki.

From the results of observation, it is revealed that fracture occurs easily at the large-diameter vessels or early wood. Additionally, in the specimen with the annual ring inclination of 0° and 45°, mechanical properties of ray tissues conclusively affected the strength and failure strain in this measurement because the fractures of ray tissues were observed frequently.

II-4 Conclusion

For the purpose of establishing the bases of science and technology about tensile deformation processing, maximum tensile deformation and basic data as an index for controlling tensile deformation property was measured. Mechanical properties concerning to lateral tensile failure strain were measured in different inclination to annual ring, moisture and temperature for the similar shape specimens from various wood species having different anatomical characteristics. Then, from the results obtained, the deformation and fracture mechanisms were discussed in terms of thermal-softening and anatomy, and the important factors were discussed to increase the lateral tensile deformation. The results obtained are as follows:

(1) The behavior of stress-strain curves are affected the difference in the deformation mode of tissue, and it is clarified that the activation of molecular motion of wood components are important for increasing the deformation further in lateral tension.

(2) Deformation facility of cell shape affects the increase of lateral failure strain. In addition, it is also suggested that density of wood affects both promotion and restriction of deformation of cell shape for the specimen with the annual ring inclination of 45° in which cells showed share deformation.

(3) The correlation between increase of failure strain and decrease of elastic modulus was not observed, one of reasons is attributable to complex effects of anisotropy of tissue and inhomogeneity of reduction of elastic modulus in specimen on the lateral tensile deformation.

(4) Fracture tends to occur easily at the large-diameter vessels of hardwood or early wood of softwood. Additionally, in the specimen with the annual ring inclination of 0° and 45°, mechanical properties of ray tissues conclusively affected the strength and failure strain in this measurement because the fractures of ray tissues were observed frequently.

CHPTER III

Rheological consideration in fracture of wood in lateral tension

III-1 Introduction

The effects of tensile direction to annual rings, moisture and temperature conditions, tree species and histories of drying or heating on lateral tensile deformation characteristics of the wood were examined for the purpose of elucidating the mechanism of the lateral tensile deformation of wood previously [68-70]. The results of their examination indicated that the failure strain of wood increased under the influence of its softening through moisture and heat effects. Then, it was thought that rheological properties were contributable to fracture.

On the other hand, some previous studies of failure strain of wood have been reported, such as the relationship between structural factors including the lignin content or the crystallinity of wood and failure strains [71-74], the relationship between the strain distribution in the specimen and the fracture surface [75], and the relationship between failure strains and factors such as the moisture content and temperature [76-78]. There is a mathematical modelling study related to the time dependent of creep behavior of wood [79], however, the study of evaluating rheological properties of wood both qualitatively and quantitatively and refer to the relationship between fracture and rheological properties have not been found. If the relationship between rheological properties of wood and its fracture could be clarified, it will be possible to determine a softened state required for the processing and indicate the optimum moisture and temperature conditions. Therefore, it is significant to elucidate the relationship between fractures and rheological properties of wood, which has not been discussed yet, in terms of both studies on wood properties and the development of control technologies for wood.

In this study, therefore, failure strain, elastic modulus and creep at various temperature conditions were measured. And, relationship between the degree of softening or fluidity and lateral tensile failure strain was examined. In order to discuss the fracture mechanism from the results of the failure strains, fracture surface was observed and appearance ratio of fractures was calculated.

III -2 Materials and methods

III -2-1 Materials

The specimens were determined to be of Hinoki (*Chamaecyparis obtusa* Endl.). The block-shaped specimens with the annual ring inclinations of 0° (tangential direction), 45° (angle between tangential and radial direction), and 90° (radial direction) against the tensile direction were collected from the sapwood part. Thin cross sectional specimens with the thickness of about 0.1 mm (longitudinal direction) and with the dimensions of 3.4 mm in width × 20 mm in length were used for the tensile breaking test. This shape of specimen was selected because the most reliable results were obtained within the load range of the test device in pretest and the origin of the fracture could be clearly observed at cellular level. Furthermore, the thin specimens were prepared with extreme care not to making scratches or knife marks which could be the origin of fracture. For the creep test, specimens in the same shape as that for the tensile breaking test were used.

Cross sectional specimens with the thickness of about 1.6 mm (longitudinal direction) and with the dimension of about 3.6 mm in width and 20 mm in length were used for measuring the elastic modulus. This shape was decided because providing repeated load may cause fatigue fracture in the measurement of elastic modulus. Therefore, specimens were made in the maximum shape that could be nipped by chuck, and were cut from the block-shaped specimen using a circular saw. Similarly to the tensile breaking test, specimens were prepared with the annual ring inclinations of 0° (tangential direction), 45° (angle between tangential and radial direction), and 90° (radial direction) against the tensile direction in the measurement of elastic modulus. Furuta et al [56,80, 81] reported that the values of elastic moduli vary sensitively under the influence of drying and heat history given to the specimens before the measurement. In order to unify the histories of specimens, they were cooled down to 1°C at 1°C /min after boiled and measured in water-swollen state.

III -2-2 Tensile breaking test

A thermo-mechanical analyzer (manufactured by Seiko Instruments Co., Ltd.; TMA/SS6100) was used in the measurement. All of the measurements were carried out in water kept at the constant temperature, and the temperatures were 5°C, 20°C, 40°C, 60°C, 80°C, and 95°C. The distance between chucks was set to 10 mm, and the specimens were measured with the load of 1 N/min until they were fractured. The measurement results were adopted only for the specimens fractured roughly at their central portion, and the specimens had exceptional deformation behaviors were excluded. The number of specimens in the results were adopted 5-8 pieces per condition.

III -2-3 Creep test

Creep test was carried out with the same device and in the same temperature conditions in the tensile breaking test. The specimens were attached with the same state as those in the tensile breaking test. The creep load was determined to be 25% of the strength obtained in the tensile breaking test in each temperature condition. At 30 seconds after the load was reached at creep load, the creep amount was calculated as the creep compliance ($J(30)$). The average value of 3 to 5 specimens per condition was used as the results, except extremely large or small results caused by the slip of the specimens at the chuck site or the distortion of their mounting.

III -2-4 Measurement of elastic modulus

Elastic modulus was measured by applying load, half as large as the creep load in each temperature repeatedly. The following load rates were adopted; 1 N/min which was the same loading rate in the tensile breaking test and 9.8 × 106 N/min which was the maximum loading rate controlled by the device. In this text, the elastic modulus obtained by measuring load rate of 1 N/min is defined as the elastic modulus (*E*) and the elastic modulus obtained by measuring load rate of 9.8×10^6 N/min is defined as the instantaneous elastic modulus (*IE*).

For the purpose of unifying the histories of the specimens, as described in "Materials", the specimens were held at about 100°C for about 5 minutes, then cooled down to 1°C at 1°C/min, heated up again to about 100°C at the temperature rising ratio of 2°C/min, and furthermore cooled down to 1°C at 1°C /min and raised again to around 100°C at the temperature rising ratio of 2°C /min prior to the measurements. The measurements were performed during the heating process. The *IE* was measured in the first heating process and *E* was measured in the second heating process, respectively.

III -2-5 Observation of fracture surface

Fracture surfaces of the specimens that were fractured under the tensile breaking test were observed with an electron microscope (TM3030Plus (Bruker AXS Co. Ltd.)). The appearance ratios of fracture surfaces was observed in five specimens showing typical fracture surfaces. The appearance ratios were obtained from the percentage of the projected length of the fracture along the annual ring boundary and the ray tissue in the tensile direction. The definition of the projected length of the fracture is illustratiod in Fig. III-1, using the photograph of the fracture surface of the specimen with the annual ring inclination of 90°. The fractures were observed along the ray tissues arranged parallel to the tensile direction in the specimens with the inclination of 90°. These fracture surfaces could be observed prominently only for the specimens with the inclination of 90°. With regard to the specimens with the inclination of 90°, therefore, breaking length which was judged to be along the ray tissues and was fractured linearly over four cells were also measured. Then, the percentage of the breaking length of ray tissue in horizontal direction was measured when the breaking length measured at 20°C was defined as 100%.

III -3 Results and discussion

Effects of the temperature on the fracture

The relationship between the failure strain and the temperature is shown in Fig. III-2. Significant differences in failure strains between the specimens with the annual ring inclination of 0° and 90° were not found, and failure strains were tended to increase from about 3 % to 6 % with increase in temperature. In contrast, in the specimens with the annual ring inclination of 45°, failure strains were larger than those with the inclination of 0° and 90°. And, failure strain increased with increase in temperature, but the failure strains at 95°C were reduced to the same extent as the result at 5°C. Comparing the relative failure strains in Fig. III-2, in the specimens with the annual ring inclination of 0°, relative failure strains increased largely with increase in temperature. In addition, decrease in failure strain at 95°C was observed in all the specimens regardless of their annual ring inclination, and the decrease in failure strain was

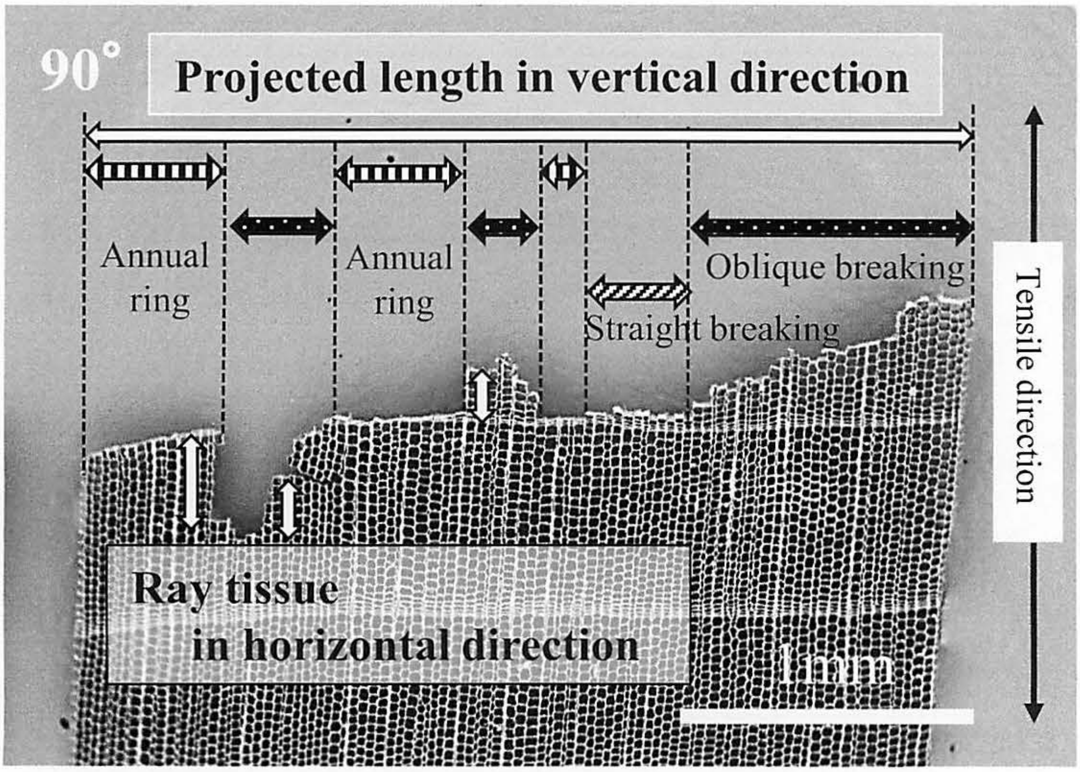

Fig. III-1 An example of photograph used for calculating appearance ratios of fractures along each tissue. From these photographs, projected lengths of fracture surfaces along each tissue and ray tissues in horizontal direction were measured and calculated.

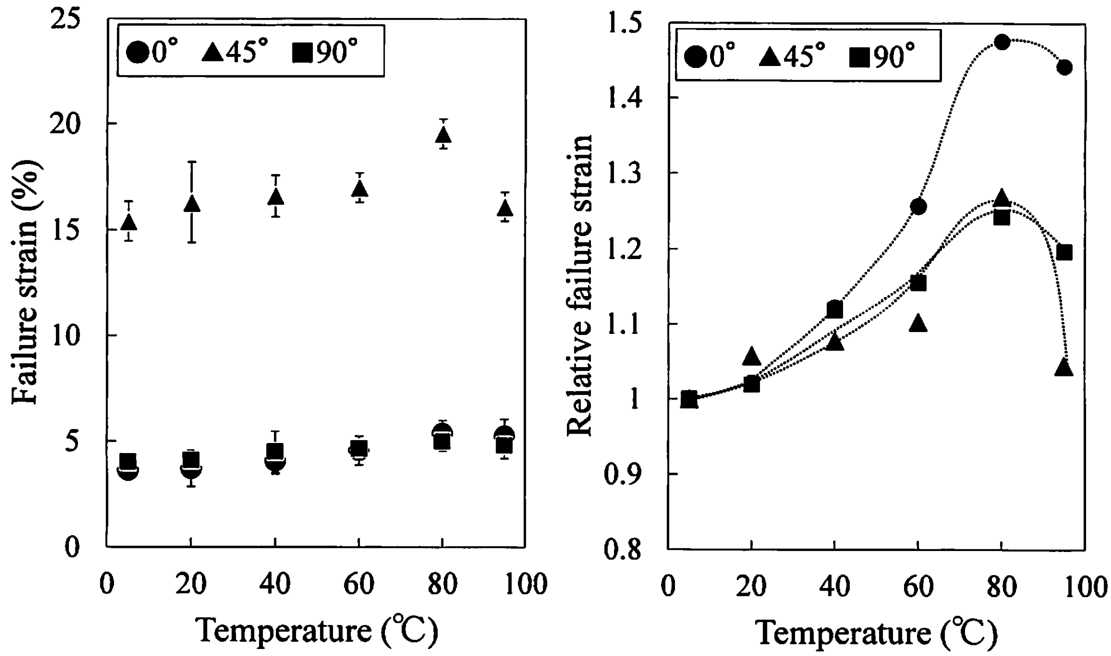

Fig. III-2 Failure strains and relative failure strains measured at each temperature. Relative failure strain is relative to the failure strain measured at 5°C in each annual ring inclination. Symbols mean the average value and error bars mean 95% confidence interval.

the largest for the specimens with the annual ring inclination of 45°.

As a reason for the decreasing of failure strain, the fracture mechanism was considered to be different between 80°C and 95°C. In water-swollen wood, fracture in the intercellular layers is easy to occur at high temperature [82, 83]. Thus, fracture surface was observed to discuss the fracture mechanism.

The appearance ratio of various fractures and photographs of broken specimen with the annual ring inclination of 0° are shown in Fig. III-3. The photographs show that the fractures tended to occur in the intercellular layers along the ray tissues at any temperature. The appearance ratio of fractures along the ray tissues was also as high as about 60-70% at 20°C and 80°C. Such a fracture tendency appeared at 95°C pronouncedly, where the appearance ratio of fractures along the ray tissues was 90%. From these results, fractures in the intercellular layers along the ray tissue tended to occur in the specimens with the annual ring inclination of 0° in the temperature range up to 80°C. While, intercellular layers along the ray tissues became weak in the higher temperature range, therefore, the factures along the ray tissues increased and failure strains decreased as shown in Fig. III-2.

The appearance ratios of various fractures and photographs of broken specimen with the annual ring inclination of 45° are shown in Fig. III-4. The photographs show that fractures tended to occur in the intercellular layers along the ray tissues for the specimens with the inclination of 45°. Focusing on the appearance ratios of fractures, the ratio along the ray tissues was almost equal at 20°C and 80°C. For the specimens at the 95°C, however, the appearance ratio of fractures along the ray tissues decreased and the fractures along the annual rings were observed, and the tendency was not found up to 80°C. From these results, the fractures tend to occur in the intercellular layers along the ray tissues in the temperature range of up to 80°C. Whereas the intercellular layers between early wood and late wood were also considered to become weak part

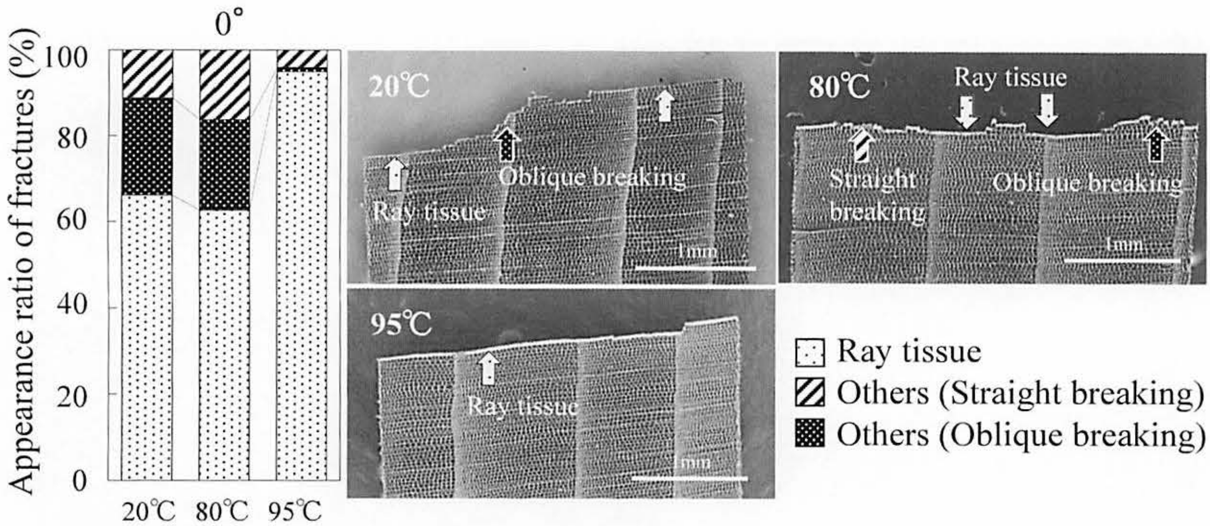

Fig. III-3 Appearance ratio of fractures along each tissue and photographs of broken specimen with the annual ring inclination of 0°.

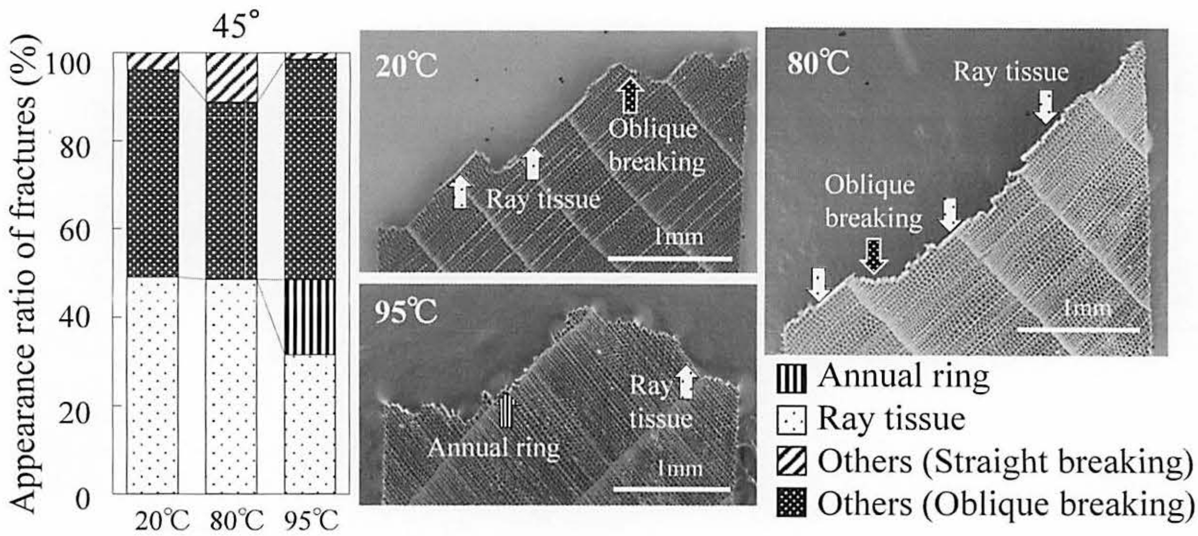

Fig. III-4 Appearance ratio of fractures along each tissue and photographs of broken specimen with the annual ring inclination of 45°.

which was easy to be fractured in addition to the intercellular layers along the ray tissues in the temperature range above 80°C. In other words, the range between 80°C and 95°C is the temperature range where the weak parts as the origin of fracture are changed, and where the fractures tend to occur because weak parts increased in the specimens. Therefore, failure strains decreased largely, as shown in Fig. III-2.

The appearance ratios of various fractures and photographs of broken specimen with the annual ring inclination of 90° are shown in Fig. III-5. The photographs show that fractures tended to occur in the intercellular layers along the annual rings for the specimens with the inclination of 90°. Focusing on the appearance ratios of fractures, the fractures along the annual rings were less frequent in their ratio as low as about 20% at 20°C, whereas the ratios were more or less 50% at 80°C or higher. Thus, the fractures along the annual rings should tend to occur with increase of temperature. Focusing on the percentage of breaking length of the ray tissues in horizontal direction, it decreased at 80°C in comparison with 20°C, and few fractures were found along the ray tissues at 95°C. From the results above, in the specimens with the inclination of 90°, fractures tended to occur in the intercellular layers along the ray tissues at 20°C, whereas the intercellular layers between early wood and late wood also became easier to fracture with increase in the temperature. Therefore, annual ring boundaries was the weakest part in the specimens at 95°C, and failure strains were reduced at 95°C as shown in Fig. III-2.

It was suggested from these results that the weakest parts as origin of fractures changed for all of the specimens in the temperature range between 80°C and 95°C. The weak parts for fractures were increased as the temperature rose. Therefore, the result failure strains decreased.

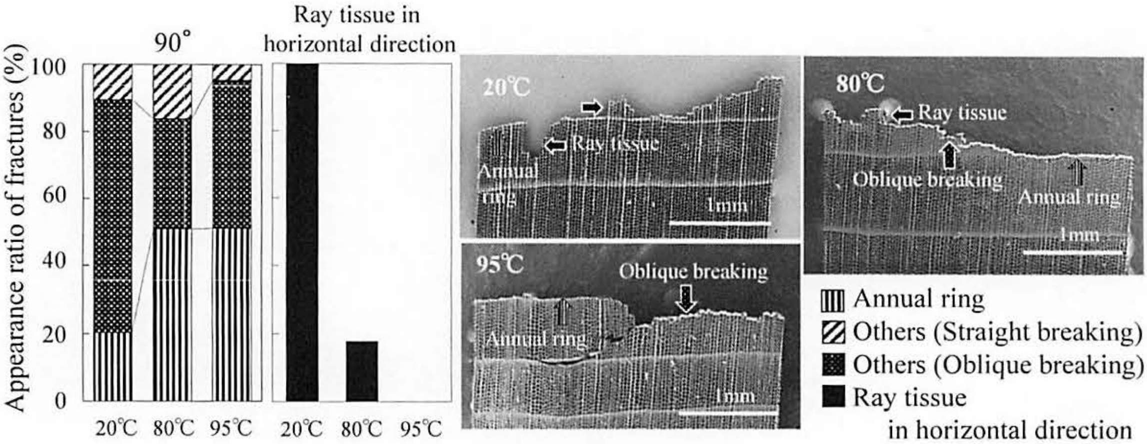

Fig. III-5 Appearance ratio of fractures along each tissue and photographs of broken specimen with the annual ring inclination of 90°.

Changes in the rheological properties of wood with increase in temperature

Then the thermal softening of wood was assessed from the elastic modulus measured in various temperatures to discuss the effect of softening on increase of failure strain. In order to obtain the information about decrease in elastic modulus (*E*) at the tensile breaking in this study, *E* was measured at the same loading rate of the tensile breaking test. The instant elastic modulus (*IE*) was also measured to have the information about the pure elastic modulus that does not include the creep deformation in the loading process as far as possible. The results obtained are shown in Fig. III-6. *E* and *IE* had larger values in the order of the annual ring inclination of 90°, 0°, 45°, and the values tended to decrease with increase in temperature. *IE* had larger value than *E* at any temperature, and the difference between their values was larger in the higher temperature especially for the specimens with the annual ring inclination of 90° and 0°. The reason why *E* is smaller than *IE* at the higher temperatures is considered to be as follows:

The load speed for measuring *E* was much slower than that in *IE*, therefore, larger creep amount of wood is included for *E* than that for *IE*. Because the creep amount during the deformation is greater at the high temperature side where the wood is softened [84], the difference between *E* and *IE* values was shown prominently at higher temperatures.

Elastic compliance which means the ease of elastic deformation was determined from the values for the elastic modulus. Elastic compliance was the largest in the specimens with the annual ring inclination of 45°and followed by those with the annual ring inclination of 0° and 90° as shown in Fig. III-6. In all of the specimens, the *IE* compliance and the *E* compliance increased with increase in temperature, and in which the *E* compliance obviously indicated the larger value than the *IE* compliance at higher temperatures.

From the results above, it was predicted that the tensile failure strains

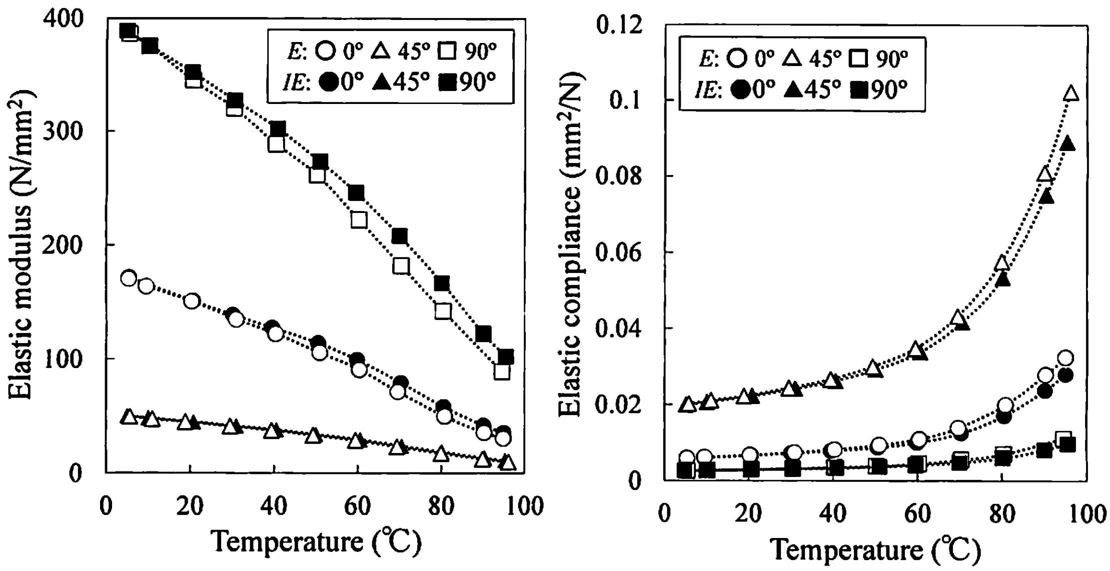

Fig. III-6 Elastic modulus and elastic compliance measured at each temperature. Open symbols are elastic modulus and closed symbols are instantaneous elastic modulus.

measured in this study were affected by creep deformation. Therefore, creep of each specimen was measured at each temperature and calculated the creep compliance (J(30)) as an index of fluidity in order to clarify the effect of fluidity contained at the time of deformation. Fig. III-7 shows the relationship between J(30) and the temperature. As common to all of the specimens, the tendency was observed that J(30) increased with increase from about 40°C where the wood started to be softened [84]. The largest J(30) was shown in the specimens with the annual ring inclination of 45° at any temperature. J(30) of the annual ring inclination of 0° and 90° was comparable up to around 60°C, whereas J(30) of the annual ring inclination of 0° was larger than those of 90° at higher temperatures. The reason why J(30) has different characteristics among annual ring inclinations can be assumed as follows:

In the specimens with the angle of 0°, late wood layers are arranged in parallel against the tensile direction, and creep amount should depend on the amount of fluidity caused in wood substance. Therefore, J(30) increased as the temperature increased because the fluidity of late wood layers aligned in the tensile direction increased the above around 40°C which was the temperature range where Hinoki began to be softened [81].

Whereas, in the specimens with the annual ring inclination of 90°, late wood layer and early wood layer are arranged alternately against the tensile direction. J(30) is determined from the sum of the creep amount of late wood with thick cell wall and early wood with thin cell wall. Therefore, in the specimens with the annual ring inclination of 90°, J(30) was smaller than that of 0° which late wood layers arranged in a row against the tensile direction. In the specimens with the annual ring inclination of 90°, ray tissues are also arranged in parallel to the tensile direction, therefore, the increase in J(30) was also disturbed by those tissues.

In the specimens with the inclination of 45° with the largest J(30), the shape

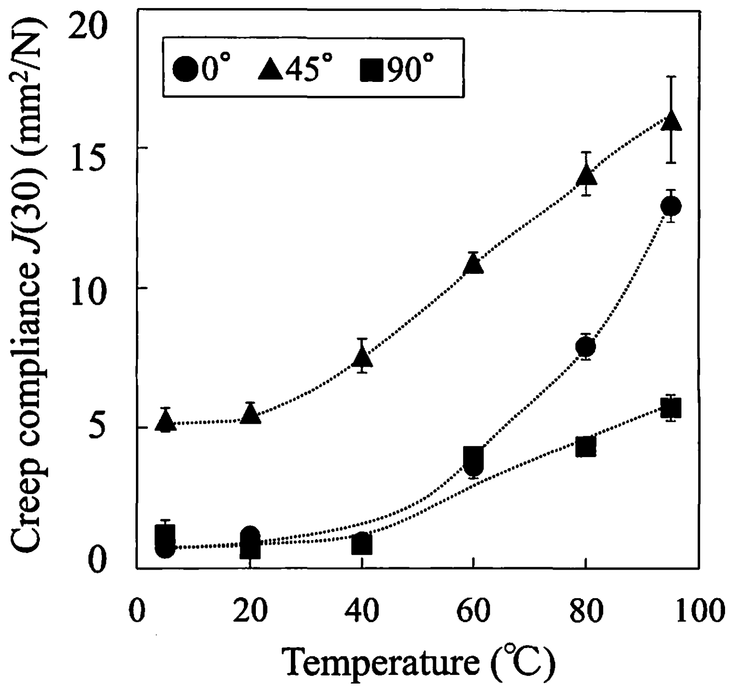

Fig. III-7 Relationship between creep compliance and temperature. Symbols mean the average values and error bars mean 95% confidence interval.

of cell is deformed roughly from a square shape to a diamond shape against the tensile load [59, 68]. Furthermore, ray tissue which disturb the deformation of cells is oriented to the direction with the inclination of 45° to the tensile direction. Thus, these tissues should effect on $J(30)$ complicatedly with increase of the tensile deformation. However, too large deformation did not occur, because the stress applied in this experiment was 25% of the strength and the tensile failure strain was approximately 3%. Therefore, the larger $J(30)$ in the specimens with the inclination of 45° are due to not those tissues but the deformation of cells, and the cell deformation should be accelerated with increase in temperature.

From these results, the effects of creep deformation, that is, fluidity are included in the tensile deformation process of wood, and the fluidity has different characteristics dependent upon the tissue structure and the load direction.

The relationship between the rheological properties of wood and failure strains

In order to examine the effects of changes in rheological properties of wood on failure strains, the relationship between relative values of the E compliance and those of failure strains is shown in Fig. III-8. The elastic modulus measured at the same loading rate was used as the index of the degree of softening with increase in temperature. Except of the temperature range in which failure strains decreased, linear relationship was found between the relative value of the E compliance and that of failure strains. The relationship between the relative failure strains and the relative E compliance was also clearly shown in the specimens with the inclination of 0° which the increase ratio of failure strain was particularly large. The reasons of these results were considered as follows:

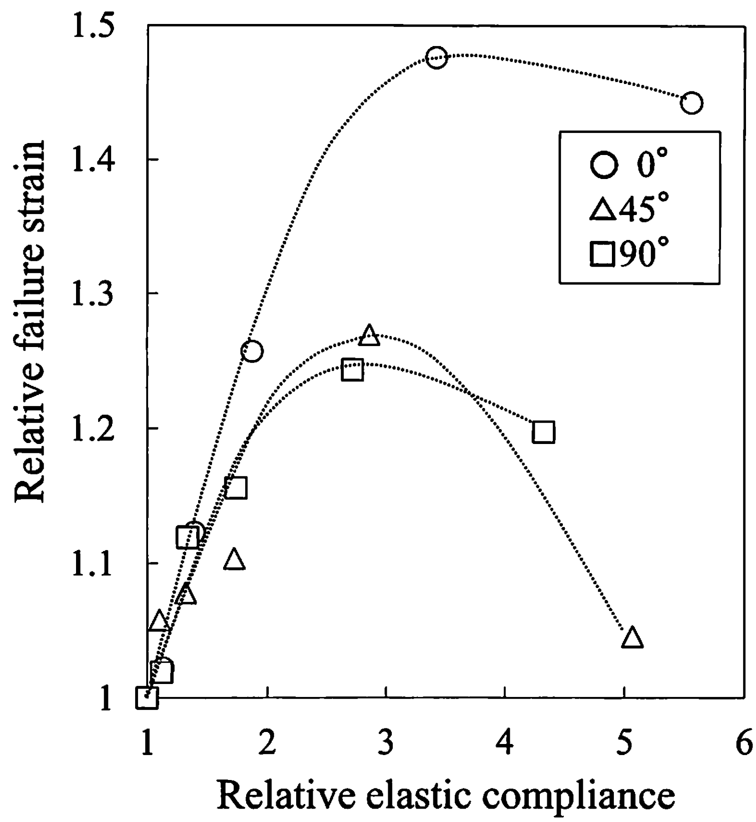

Fig. III-8 Relative failure strain and relative elastic compliance are those measured at 5°C in each annual ring inclination.

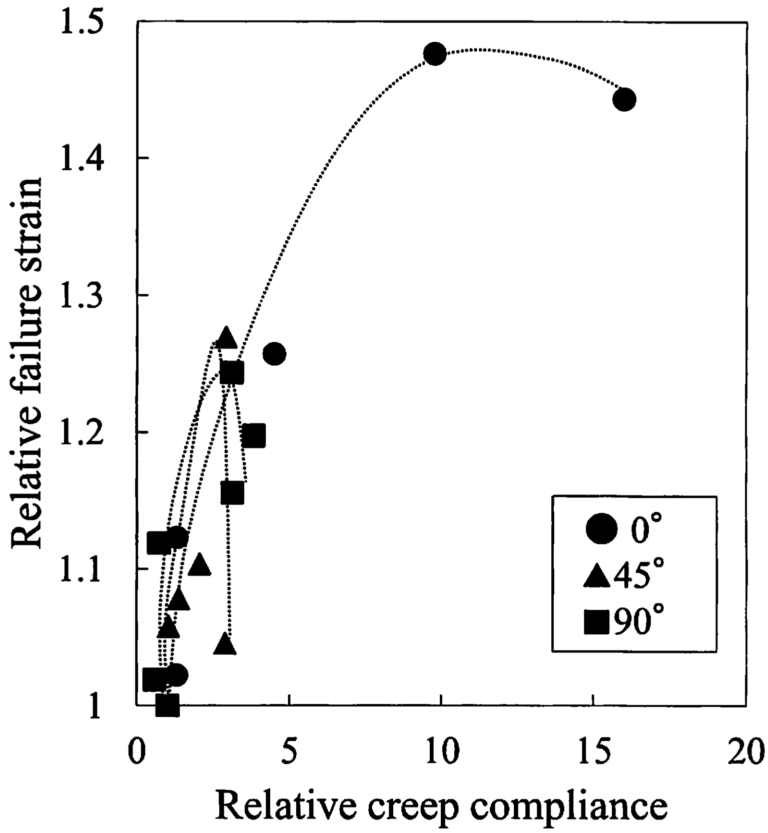

Fig. III-9 Relative failure strain and relative creep compliance are those measured at 5°C in each annual ring inclination.

Deformation mode of the specimen with the inclination of 0° is hardly affected by the ray tissues which disturb the deformation. Therefore, increase in failure strains was affected primarily by the softening, thus the relationship was observed clearly between the relative failure strain and the relative *E* compliance. From the results described above, effects of softening of wood is important for increasing failure strains up to the temperature range in which the fracture mechanism changes. Moreover, effect of softening on failure strains was most clearly found in annual ring inclination of 0° which the ray tissues and deformation of cells did not affect the deformation of the specimens.

On the other hand, the effects of creep deformation of specimens with each annual ring inclination on failure strain were examined, because the creep deformation caused in the loading process is also included in the amount of deformation until the wood fracture. The relationship between the relative values of $J(30)$ and relative failure strains is shown in Fig. III-9. In the specimen with the annual ring inclination of 0° and 45°, roughly linear relationship was found between the increase ratios of $J(30)$ and failure strains up to the temperature range where failure strains decreased. However, that relationship was not clearer than that of the elastic compliance, and the relationship was not found in the specimen with the inclination of 90°. In other words, the creep deformation occurred before fracture did not significantly affect the increase in failure strain.

Ⅲ-4 Conclusion

For the purpose of elucidating the effect of rheological property of wood on lateral tensile fracture, failure strain, elastic modulus, and creep at various temperature conditions were measured. The relationship between the degree of softening or fluidity and lateral tensile failure strain was examined. In order to discuss the fracture mechanism from the results of the failure strains, fracture surfaces was observed and appearance ratio of breaking was calculated. The results obtained are as follows:

(1) The temperature range between 80°C and 95°C is the range where the weak parts as the origin of fracture are changed, and where the fractures tend to occur because weak parts increased in the specimens.

(2) The effects of softening of wood is important for increasing failure strains up to the temperature range in which the fracture mechanism changes. Moreover, effect of softening on failure strains was most clearly found in annual ring inclination of 0° which the ray tissues and deformation of cells did not affect the deformation of the specimens.

(3) Creep deformation, that is, fluidity was included in the wood deformation process, and the fluidity had different characteristics dependent on the anatomical characteristics. It also became clear that the creep deformation did not affect the increase in failure strain significantly.

CHPTER IV

Effectiveness of destabilization of wood to control deformation properties in lateral tension

IV-1 Introduction

Techniques for deforming wood into various shapes are required to improve the processability of wood products. However, wood has a low ductility. When veneers are press molded into a tray or pasted on the curved surface, cracks often occur in large parts of the curvature. It is considered that the reasons for these problems are due to small lateral tensile deformation and strength. Therefore, to improve the processability of wood, first of all, it is necessary to understand the characteristics of the lateral tensile deformation of wood. And then, these studies should be applied to the various deformation processes technology of wood. However, physical properties of lateral tensile deformation have not been made sufficiently clear in previous studies. So, we have studied lateral tensile deformation properties at various moisture content, temperatures, angle to annual rings and species in the same size of specimens in chapter II.

In contrast, it was reported that physical properties of wood are affected by drying and/or quenching histories [80,81,85-100]. In these reports, elastic modulus and strength of wood were reduced, and fluidity of wood was increased in comparison to annealed specimen of water-swollen wood [88,91]. And these physical properties were depended on the soaking time in water after drying and/or quenching. Thus, many physical properties of wood are affected by change of environment such as moisture and temperature. These physical properties changes of wood due to drying and/or quenching histories were observed from the bending test in most cases [88,91]. On the other hand, the lateral tensile deformation property of wood with drying and/or quenching histories is not clear.

For these reasons, lateral tensile deformation properties of water-swollen wood with drying and/or quenching histories were studied. From the results obtained, the influence of these histories on lateral tensile deformation of wood were clarified, and then it was discussed whether giving these histories was effective to increase the lateral tensile deformation of wood.

IV-2 Materials and methods

IV-2-1 Materials of tensile breaking test

Test specimens of block-shaped were taken from the outer region of the log of Hinoki (*Chamaecyparis obtusa* Endl.). The specimens with the annual ring inclinations of 0° (tangential direction), 45° (angle between tangential and radial direction), and 90° (radial direction) against the tensile direction were made. The average ring width of the specimen was about 1.1 mm. Cross section of the block specimen was sliced by a sliding microtome, because fine roughness caused by the circular saw blade can be a starting point of the failure of the specimen in the tensile test. The specimen size was 0.1 mm thickness (longitudinal direction), 3.2 mm width, and 20 mm length.

The results obtained for the specimens with quenching or drying history were prepared each other with that of the control specimens. The specimens were conditioned in the manners shown in Fig. IV-1. Control specimens were boiled for 15 minutes and were cooled slowly to 25°C for 24 hours and then soaked in water at 25°C for 1 week. The specimen with quenching history was boiled for 15 minutes and were put immediately into water at 25°C. The specimens with drying history were obtained as follow. First, the specimens were boiled for 15 minutes and were cooled slowly to 25°C for 24 hours.

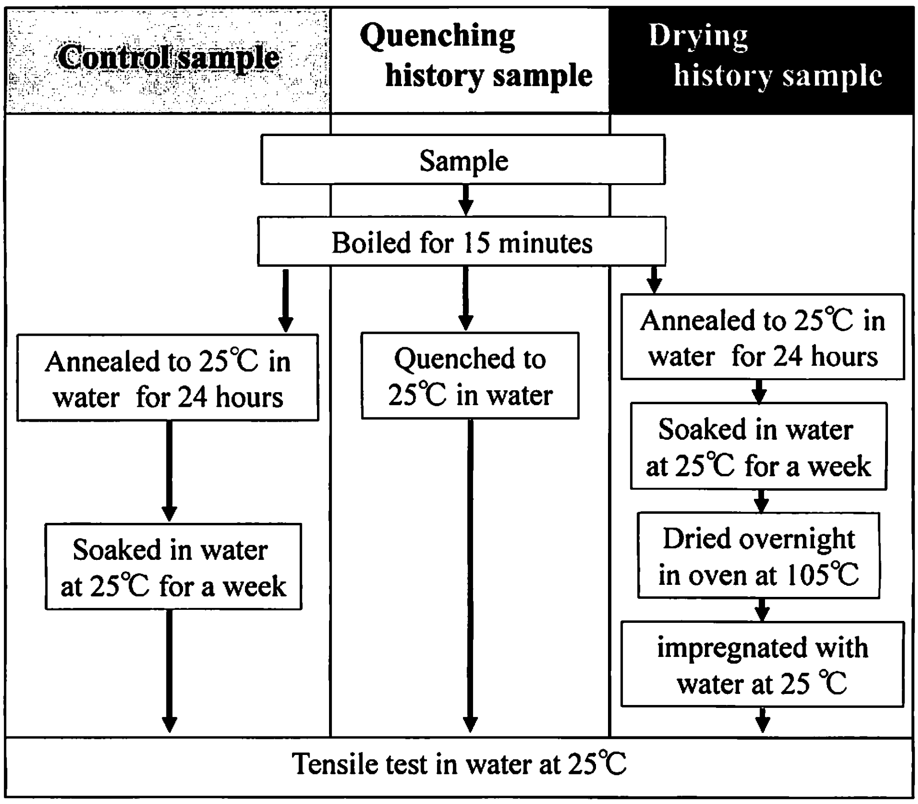

Fig. IV-1 The methods of conditioning.

Next, the specimens were dried at room temperature for 1 day and dried overnight in an oven at 105°C. And then, dried specimens were impregnated with water at 25°C under vacuum.

IV-2-2 Tensile breaking test

The measurement were carried out in water at 25°C by thermo-mechanical analyzer (Seiko Instruments Co., Inc. TMA/SS6100). The measurement span was 10 mm and the rate of load was 1N/min. Fig. IV-2 shows the appearance of the specimens attached to the chuck. The typical stress-strain curve and average values were obtained from 10 specimens which the failure occurred around the center of the span.

IV-3 Results and discussion

Stress-strain curves of wood with drying and/or quenching histories

Fig. IV-3 shows typical stress-strain curves of different angle specimens with different histories. In the specimen with the annual ring inclination of 0°, significant difference in the behavior of stress-strain curves between each specimen was not found. In the specimen with the annual ring inclination of 45° having each history, failure strain and strength were larger than that of control specimen. In the specimens with the annual ring inclination of 45°, failure strains were larger than those with the annual ring inclination of 90° and 0° in all of the measurement conditions. This result suggests as follows: It is reported that cells having an almost square shape relatively easily deform like a diamond shape by tensile force acting to the cells during increasing of tensile stress [50]. Therefore, the deformation of cell for the specimens with the annual ring inclination of 45° were shared larger than other specimens with the

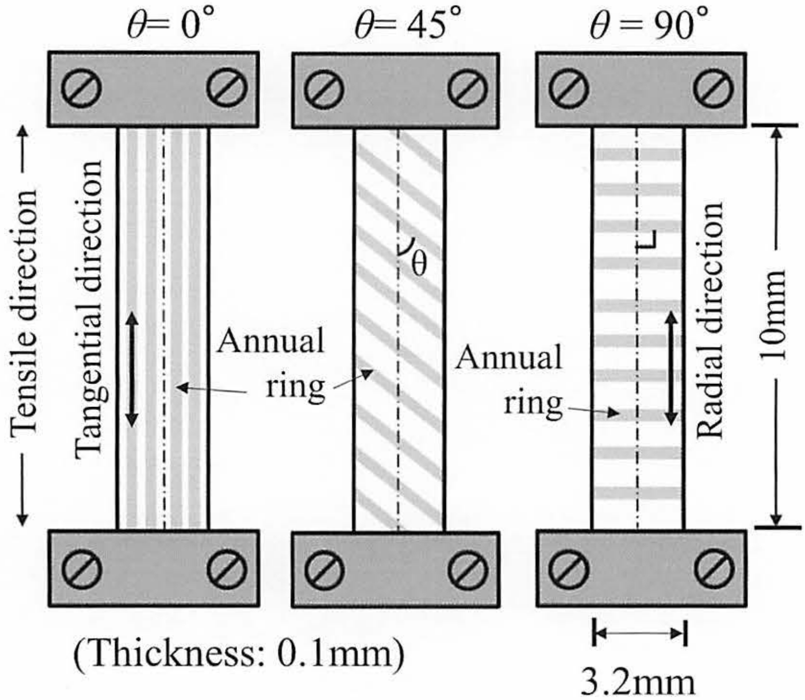

Fig. IV-2 The appearance of the sample attached to the chuck.

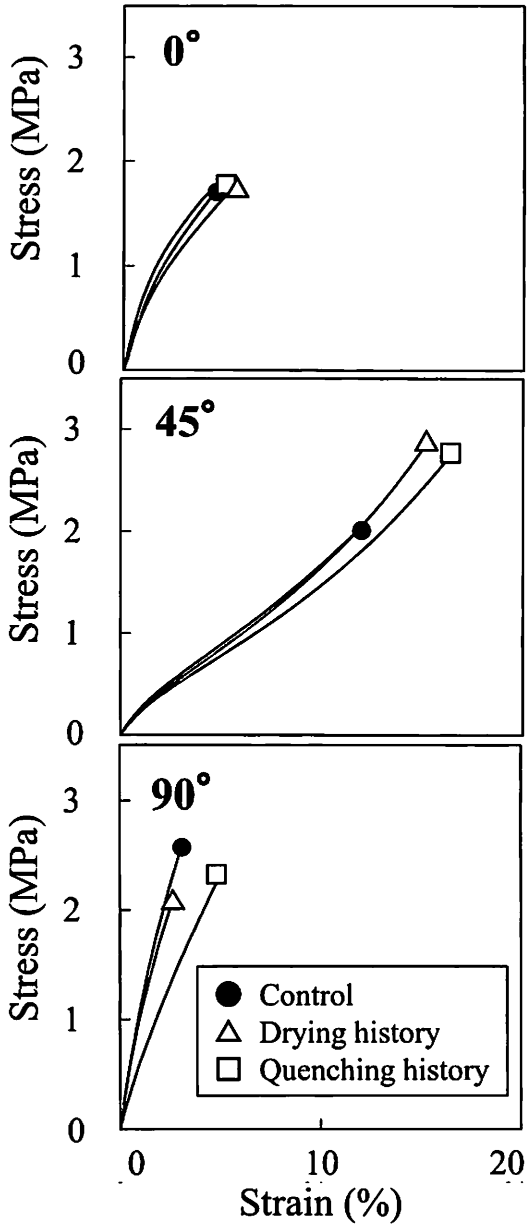

Fig. IV-3 The typical stress–strain curves of specimens with the annual ring inclinations of 0°, 45° and 90° against the tensile direction having different histories. Symbol means each failure point.

annual ring inclination. In the specimen with the annual ring inclination of 90° having quenching history, the gradient of stress-strain curve was smaller and strain was larger than those of control specimens and specimens with drying history. The gradient of the stress-strain curve of the specimens with the annual ring inclination of 90° was steeper and linear than those of other angle specimens. In the specimens with the annual ring inclination of 90°, ray tissue is arranged in the tensile direction. This should be reflected to the above result. Thus, effect of some history on the lateral tensile deformation properties was different in the each angle specimen.

Mechanical properties of wood with drying and/or quenching histories in lateral tension

To investigate the effect of drying and/or quenching histories on lateral tensile mechanical properties in detail, tensile failure strain, strength and elastic modulus of each specimen were obtained. Fig. IV-4 shows relationship between the tensile properties and tensile directions to the annual rings. The average values were obtained from 10 specimens which the failure occurred around the center of the span. Specimens with drying and/or quenching histories tended to have larger failure strain than those of control specimens. The tendency like this was quite similar in the specimen with the annual ring inclination of 90°, 45° and 0°. The specimen with the annual ring inclination of 45° having drying and/or quenching histories showed maximum value of failure strain of about 15%. This result suggests that the tensile deformation of wood was increased by giving drying and/or quenching histories. The reason was considered as follows: According to the present research, the wood quenched has the value of lower modulus of elasticity (MOE) and higher fluidity than those of wood annealed which is kept at a constant temperature for a long time [91].

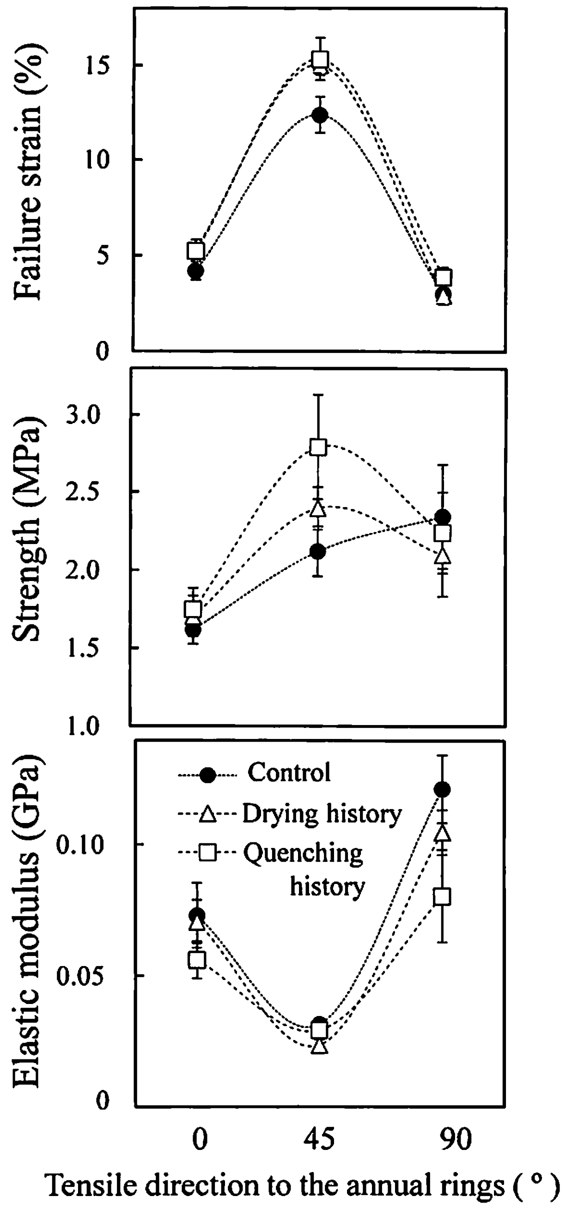

Fig. IV-4 Relationship between the mechanical properties and tensile direction to the annual rings. Symbols mean the average value and error bars mean 95 % confidence interval.

Kudo et al. reported that the fluidity of wood quenched from 80°C to 40°C or below was increased remarkably [88]. However, the fluidity of wood quenched from 80°C to 50°C or above did not change significantly. Whereas, Nakano suggested the increase in relaxation behavior of wood was caused by increase free volume in the amorphous polymer of wood constituents due to quenching treatment [94]. And, he succeeded that theoretical formula which explains the relaxation behavior of amorphous polymer could be adapted to the relaxation behavior of wood quenched. Kojiro et al.[97,98] studied the Pore-size distribution determined by adsorption of CO_2 with specimens dried by outgas at various temperatures from 30°C to 190°C. As a result, it was observed that the micro-spaces were decreased with elevating outgas temperature. Nakatani et al.[101] confirmed some interesting facts by measuring the adsorption of various organic liquids and CO_2 for wood. That is, the most of sub nanometer order of micro-pores of wood was present in lignin. From these mentioned above, it might be considered that the value of the failure strain was increased by increasing of fluidity due to free volume mainly in lignin which was increased by giving drying and/or quenching histories.

Focusing on the strength, the values of control specimens were increased in the order of the annual ring inclination of 0°, 45°, 90°. In the specimens with the annual ring inclination of 45° having drying and/or quenching histories showed maximum strength. The strength the annual ring inclination of 90° was larger than that of the annual ring inclination of 0°. In addition, between the specimens with different histories, the difference of strength was not significant in the specimens with the annual ring inclination of 0° and 90°. However, in the specimens with the annual ring inclination of 45°, the strength of the specimens with drying and/or quenching histories was larger than that of control. From previous studies, bending strength of water-swollen wood was decreased by giving quenching history [91]. Therefore, the results obtained in this study were differed from that of previous study. In the specimens with the annual ring inclination of 45° having each history, the reason for the significant increase in strength was considered as follows. In the specimens with the annual ring inclination of 45°, gradient of stress-strain curve is steeper with increase in strain as shown in Fig. IV-3. In the deforming process, cells of early wood are especially shared like diamond shape. With increasing tensile strain, the angle between the ray tissue or the late wood layer which have higher elastic modulus than that of early wood and tensile direction is decreased. Then, these tissues become bearing larger tensile stress. Therefore, it can be said that strength was also increased because the tensile strain was increased by giving drying and/or quenching histories.

Focusing on the elastic modulus, in the specimens with the annual ring inclination of 45°, elastic modulus was the lowest in any specimens with the annual ring inclination. In the specimens with the annual ring inclination of 0°, the elastic modulus was lower than that of the specimens with the annual ring inclination of 90°. In all of the specimens, the elastic moduli of specimens with some histories were smaller than that of control. Similar tendencies were seen in the previous studies, that is, the bending elastic modulus of water-swollen wood with some histories was smaller than those of green wood or wood annealed [81,88,91]. Additionally, bending elastic modulus which was decreased by giving some histories was increased with the passage of time. The reason of these results was considered as follows: When wood is given drying and/or quenching histories, molecular state of wood constituents is disturbed and microscopic strain is occurred in microstructure of wood. These microscopic strains are eliminated with the passage of time. In the eliminating process of microscopic strain, in other words, in unstable state in which the thermodynamic state of wood component transitions toward stable state, resistance of wood to external force is recovered. Therefore, it might be

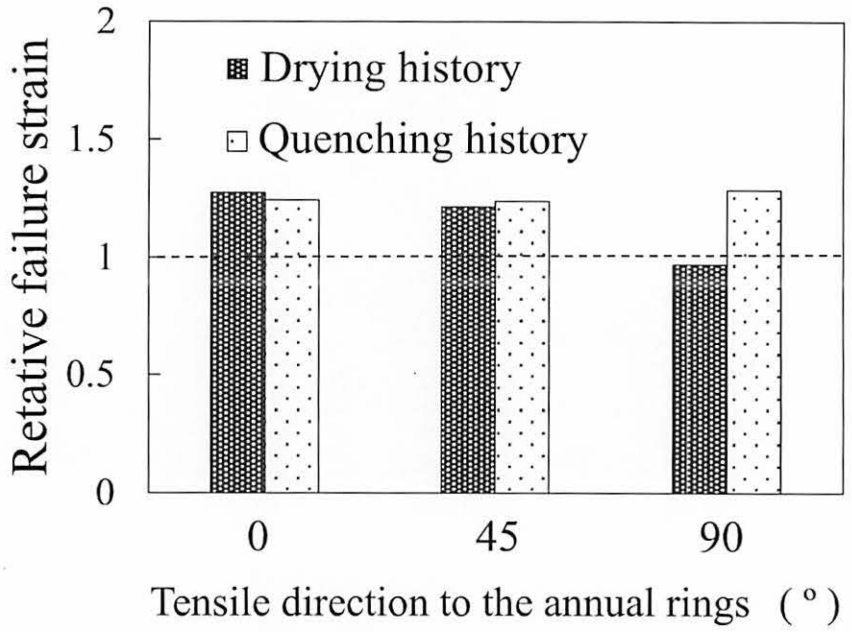

Fig. IV-5 Relative failure strain of specimens with the annual ring inclinations of 0°, 45° and 90° against the tensile direction having different histories. Relative failure strain means the failure strain in specimen with each history relative to that in control specimen.

considered that lateral tensile elastic modulus of wood given some histories was also decreased as well as those of bending.

Influence of drying and/or quenching histories on failure strain

As mentioned above, the lateral tensile deformation were different from the angles of the tensile direction to the annual rings. Therefore, to consider the influence of the drying and/or quenching histories on the failure strain, it is necessary to compare the increasing value of failure strain between specimens. Fig. IV-5 shows relative failure strain of different angle specimens with each history. Relative failure strain means the value in specimen with each history relative to that in the control. In the specimens with the annual ring inclination of 45° and 0° having each history, failure strain was increased about 20% in comparison to that of control specimens. In the specimens with the annual ring inclination of 90° having quenching history, failure strain was increased about 20% as well as the results in the specimens with the annual ring inclination of 0° and 45°. While, in the specimen with the annual ring inclination of 90° having drying history, the failure strain was not increased. The cause of the difference in relative failure strain was considered that fine roughness which was caused during drying process contribute to tensile failure. It is necessary to study in the future, to clarify the cause.

From the results above, except for the specimens with the annual ring inclination of 90° having drying history, failure strains of the water-swollen wood at 25°C were increased about 20% in comparison to the control specimen by giving drying and/or quenching histories.

IV-4 Conclusion

To clarify the influence of drying and/or quenching histories on lateral tensile deformation of wood, we studied lateral tensile deformation properties of water-swollen wood with those histories. The results obtained are as follows:

(1) In the specimens with the annual ring inclination of 45° having drying history, failure strain was about 15%. The value was about 3 to 4 times as compared to the results in the specimens with the annual ring inclination of 0° and 90°.

(2) Further, except for the specimens with the annual ring inclination of 90° having drying history, failure strains were increased about 20% by giving drying and/or quenching histories.

These results show that giving drying and/or quenching histories is the effective method for wood to increase the tensile deformation of the wood even in the water-swollen condition at 25°C because of some unstable states caused by these histories. Therefore, largely unstable states given by various kind of histories should make possible to increase in the lateral tensile deformation of wood.

General conclusion

Several studies were conducted for controlling the property of wood in use. Among the studies, exceptional deformation behavior of wood in lateral tension was shown before fracture. Therefore, focusing on the lateral tensile deformation and fracture, basic study on deformation and fracture properties of wood in lateral tension was discussed since chapter II.

For the purpose of controlling deformation and fracture properties of wood in lateral tension, mechanism of deformation and fracture and factors of increasing tensile deformation were examined. In addition, potential for applying the results obtained to processing technology of wood was discussed. Results obtained were as follows.

In chapter II, mechanical properties concerning to lateral tensile failure strain were measured in different inclination of annual ring, moisture condition and temperature for the similar shape specimens from various wood species having different anatomical features. It was clarified that deformation of cells affected the whole deformation of wood specimens, and thermal-softening property, anatomical features and density were closely related to the deformation of cells. Especially, cell deformation was promoted by thermal-softening in all of specimens and restricted by increase of density in the specimen with annual ring inclination of 45°.

In chapter III, the relationship between the degree of softening or fluidity and lateral tensile failure strain was examined. In order to discuss the fracture mechanism from the results of the failure strains, broken specimens were observed and each appearance ratio of fractures in different tissues was calculated. The weak parts as the origin of fracture changed in the temperature range of from 80°C to 95°C, and it was considered that weak parts increased within the specimens in the temperature range.

The effects of softening of wood is important for increasing failure strains up to certain temperature range in which the fracture mechanism changes. Creep deformation, that is, fluidity was included in the wood deformation process, and the fluidity had different characteristics dependent on the anatomical features. It also became clear that the creep deformation did not affect the increase in failure strain significantly.

In chapter IV, to clarify the influence of drying and/or quenching histories on lateral tensile deformation of wood, we studied lateral tensile deformation properties of water-swollen wood with those histories. From the results obtained, giving drying and/or quenching histories is the effective method for wood to increase the tensile deformation of the wood even in the water-swollen condition at 25℃ because of some unstable states caused by these histories. Therefore, largely unstable states given by various kind of histories should make possible to increase in the lateral tensile deformation of wood.

From the results and discussion in this study, new information regarding the deformation and fracture mechanism of wood in lateral tension were obtained, and important factors for increasing the deformation were elucidated. In the future, it will be possible to establish new processing technologies utilizing the tensile deformation positively if these information were applied to wood processing technologies practically.

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Acknowledgments

The author wishes to express her sincere thanks to Professor Yuzo Furuta, Graduate School of Life and Environmental Sciences, Kyoto Prefectural University, for his valuable discussions and suggestions and kind encouragement for this work. The author learned many things from his enthusiastic attitude and interest to wood research.

The author would also like to thank Professor Kazuki Matsumura, Professor Takefumi Ikeda, Professor Toshinari Kawada and Associate Professor Hisashi Miyafuji, Graduate School of Life and Environmental Sciences, Kyoto Prefectural University, for their valuable suggestion for this manuscript.

The author wishes to express her gratitude to Emeritus Professor Shuichi Kawai, Graduate School of Advanced Integrated Studies in Human Survivability, Kyoto University, for his supervision, valuable discussion and suggestions for study of slit material.

The author wishes to express her deepest gratitude to Emeritus Professor Hiromu Kajita, Emeritus Professor Yutaka Ishimaru and Emeritus Professor Makoto Ohkoshi, Kyoto Prefectural University, for there warmhearted guidance and valuable suggestion for this study.

The author wishes to express her gratitude to Assistant Professor Keisuke Kojiro, Kyoto Prefectural University, for his helpful advice and encouragement for doctoral thesis.

The author express her thanks to Lecturer Koji Murata, Graduate School of Agriculture, Kyoto University, Professor Kozo Kanayama and former Associate Professor Toshiro Morooka, Research Institute for Sustainable Humanosphere, Kyoto University, and Assistant Professor Yuki Fuchigami, Mie University forests for their useful suggestions.

Acknowledgement is also to Dr. Tsunehisa Miki and Dr. Masako Seki, National Institute of Advanced Industrial Science and Technology, for their encouragement and discussion on the research.

The author express her special thanks to Professor Hiroyuki Yano, Research Institute for Sustainable Humanosphere, Kyoto University, Dr. Masaki Sugiyama, Forestry and Forest Products Research Institute, and Dr. Chika Ishihara, Gifu Prefectural Research Institute for Human Life Technology for their kind advice and support.

The author gives her special thanks to Mr. Koji Uenae and Mr. Toshiharu Tokuda, technicians of Kyoto Prefectural University forests, for their assistance with the preparation of the wood specimens. The author greatly appreciates to Ms. Yuki Tashiro, Mr. Junichiro Ida, Ms. Fumi Sasaki, Mr. Tomoaki Kiryu, Ms. Mariko Furutani and other laboratory members for having meaningful time.

Finally, the author also expresses her thanks to her family concerned with Daiwa sliced veneer Industry Co., Ltd., for their understanding and support.

List of publications

The present study has been reported in the following articles.

1. Miyoshi Y, Furutani M, Ishihara M, Tai S, Furuta Y, Kawai S. Technological development for the control of humidity conditioning performance of slit materials made from Japanese cedar. Journal of Wood Science 61:641-646 (2015)
2. Miyoshi Y, Furutani M, Furuta Y. Swelling Behavior of Cells in Compressed Wood. Journal of the Society of Materials Science, Japan (Accept: November 12, 2015)
3. Miyoshi Y, Kojiro K, Furuta Y. Deformation Properties of Wood in Lateral Tension. Effect of tensile direction to the annual rings, moisture, and temperature on lateral tensile deformation of hinoki (*Chamaecyparis obtusa*) (in Japanese). Mokuzai Gakkaishi 60:241-248 (2014)
4. Miyoshi Y, Kojiro K, Furuta Y. Effect of Anatomy and Thermal-Softening Properties on Lateral Tensile Deformation Properties of Various Wood Species (in Japanese). Journal of the Society of Materials Science, Japan 64:356-361 (2015)
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