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## Changes in the mechanical properties of wood during a period of moisture conditioning

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**Abstract** Changes in the modulus of elasticity (MOE), modulus of rupture (MOR), and stress relaxation in the radial direction of wood (hinoki: *Chamaecyparis obtusa*) moisture-conditioned by the adsorption process from a dry state and by the desorption process from a moisture content slightly below the fiber saturation point were investigated. The MOE and MOR of wood conditioned by the adsorption process showed significant increases during the later stages of conditioning when the moisture content scarcely changed. However, with the desorption process they did not increase as much during later stages of conditioning, though they increased during early stages of conditioning when the moisture content greatly decreased. The stress relaxation of wood decreased with an increase in the conditioning period with both the adsorption and desorption processes. These results suggest that wood in an unstable state, caused by the existing state of moisture differed from that in a true equilibrium state shows lower elasticity and strength and higher fluidity than wood in a true equilibrium state. Furthermore, the present study demonstrates that the unstable states of wood induced during the course of drying, desorption, and possibly adsorption of moisture are slowly modified as wood approaches a true equilibrium state.

**Key words** Moisture-conditioned wood · Modulus of elasticity · Modulus of rupture · Stress relaxation

### Introduction

Recently, it has been observed that the dynamic modulus of elasticity of water-saturated wood with a drying history increased with increased time left in water for more than a month.<sup>1</sup> In addition, the wood swollen in some organic liq-

uids showed higher elasticity and strength as the wood approached the swelling equilibrium, and it showed lower fluidity than wood swollen in water to the same degree. However, if the wood was far from the swelling equilibrium, it showed lower elasticity and strength and a higher fluidity than wood swollen in water to the same degree.<sup>2</sup>

These results are interpreted to suggest that the mechanical properties of wood in an unstable state during the process of approaching a true equilibrium state differ from those of wood in an equilibrium state; in other words, such wood shows lower elasticity and strength and higher fluidity than that in a true swelling equilibrium. Furthermore, it was previously found that the moisture content of wood conditioned by adsorption and desorption changed continuously during a moisture-conditioning period of over 50 weeks.<sup>3</sup> In light of this result, the sorption hysteresis of wood is thought to be a phenomenon that occurs during a transitional process as wood approaches the true sorption equilibrium.

There seem to be no systematic studies in which the mechanical properties of wood were investigated in terms of an unstable state of wood. Hence, this study aimed to clarify some of the mechanical properties of wood in unstable states approaching the sorption equilibrium. It was achieved by examining the elastic, strength, and viscoelastic properties of wood during the moisture-conditioning process of adsorption and desorption of moisture.

### Materials and methods

#### Wood samples

Wood samples were obtained from a similar portion of the outer region of heartwood with a straight grain in a log of Japanese cypress (hinoki: *Chamaecyparis obtusa*). Three wood sticks with cross sections of 100 mm in the radial (R) direction, 15 mm in the tangential (T) direction, and a longitudinal (L) length of about 60 cm were prepared. Each wood stick was marked along an annual ring near the cen-

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**Table 1.** Saturated salt solutions used for moisture conditioning

Salt	Relative humidity <sup>3</sup> (%)	Usage for conditioning		Moisture content after 9 weeks of conditioning (%)
		Adsorption	Desorption	
CH <sub>3</sub> COOH	22.8		+	6.2
MgCl <sub>2</sub> ·6aq	33.1	+		5.6
K <sub>2</sub> CO <sub>3</sub> ·2aq	42.6		+	9.5
Mg(NO <sub>3</sub> ) <sub>2</sub> ·6aq	54.3	+		9.0
NH <sub>4</sub> NO <sub>3</sub>	64.9		+	13.5
NaCl	75.4	+		13.0
KCl	84.9		+	19.5
KNO <sub>3</sub>	93.8	+		20.7

aq, water

tral part in the radial direction, and then a loading point was placed on the marked position for measuring the mechanical properties of the samples during bending. Approximately 100 cross-sectional wood samples with a thickness of 4mm in the longitudinal direction were successively cut from each wood stick. There were 280 wood samples in all obtained from each of the three wood sticks, and they were divided into 40 groups (moisture conditioning at eight relative humidities and conditioning for five periods) of seven pieces each (Table 1).

#### Pretreatment of the wood samples

It has been observed that the mechanical properties of wood are affected by previous drying.<sup>4</sup> In this study, all wood samples were pretreated in the following manner to modify the effects of the previous drying. First, the samples were vacuum-dried for at least 1 week; they then were soaked in water under a vacuum and left for 1 week in water. After this period, the samples were dried for several days in a room at a constant temperature of 20°C and relative humidity (RH) of 60%; they were then placed in a desiccator containing a saturated aqueous solution of calcium sulfate dihydrate (98% RH<sup>5</sup>). They were left in the desiccator for 1 week to modify the effect of liquid tension, which originates during the desorption of moisture from the water-saturated state<sup>3</sup> and which affects the mechanical properties of wood. Then the samples were vacuum-dried again in a desiccator containing diphosphorus pentoxide for at least 1 week, followed by oven-drying at 105°C for 15h. After these processes, each wood sample was placed in a polyethylene bag of known weight, and the bags were sealed with zippers. After cooling the samples in a desiccator containing dry calcium chloride, the samples were weighed with the bags.

#### Moisture conditioning

For moisture conditioning during the adsorption process from the dry state, each dried sample was placed separately in a different desiccator containing a saturated aqueous solution of one of the salts listed in Table 1. In regard to moisture conditioning during the desorption process, it was

observed that the wood samples that began in a water-saturated state showed lower elasticity and diminished strength due to liquid tension compared with those that began with a moisture content below the fiber saturation state, as described above. Therefore, in the present study moisture conditioning during the desorption process from a moisture content below the fiber saturation point was adopted. That is, the samples were conditioned once their moisture content was about 25% in a desiccator containing a saturated aqueous solution of calcium sulfate dihydrate. Each sample was then placed separately in a different desiccator containing a saturated aqueous solution of one of the salts listed in Table 1. The desiccators were then placed in a room stabilized at 20°C for predetermined periods.

#### Grouping of the wood samples

Various properties of wood are judged to be most alike along the longitudinal direction, depending on the arrangement of wood tissues. The present study aimed to obtain some information about the dependence of mechanical properties of wood during moisture-conditioning periods and about the difference between wood moisture conditioned by the adsorption and desorption processes. Accordingly, the successive wood samples were divided into moisture-conditioning groups as shown in Table 2. For example, seven pieces were selected, one by one, for 10 turns. Five samples from each moisture-conditioning group were used to measure the modulus of elasticity (MOE) and the modulus of rupture (MOR), and two samples were used to measure stress relaxation.

#### Measurement of mechanical properties

The test samples moisture-conditioned for 1, 2, 3, 4, or 9 weeks were sealed with a polyethylene film to avoid changes in moisture content during the test, and then the samples sealed in the film were weighed prior to the test. A testing machine (Tensilon UTM-4L; Toyo Sokki Co., Japan) was used for both the static bending test and the stress relaxation test in the radial direction under the following conditions: span 80mm of central loading; down-speed of a cross-head, 4mm/min.

**Table 2.** Example of the division of successive wood samples for testing

Test	Adsorption in 33.1% RH at 1 to 9 weeks					Desorption in 22.8% RH at 1 to 9 weeks				
	1	2	3	4	9	1	2	3	4	9
Static bending	102	103	104	105	106	107	108	109	110	111
	112	113	114	115	116	117	118	119	120	121
	122	123	124	125	126	127	128	129	130	131
	132	133	134	135	136	137	138	139	140	141
	142	143	144	145	146	147	148	149	150	151
Stress relaxation										
	2 hours	152	153	154	155	156	157	158	159	160
15 hours	162	163	164	165	166	167	168	169	170	171

See text for explanation of the table

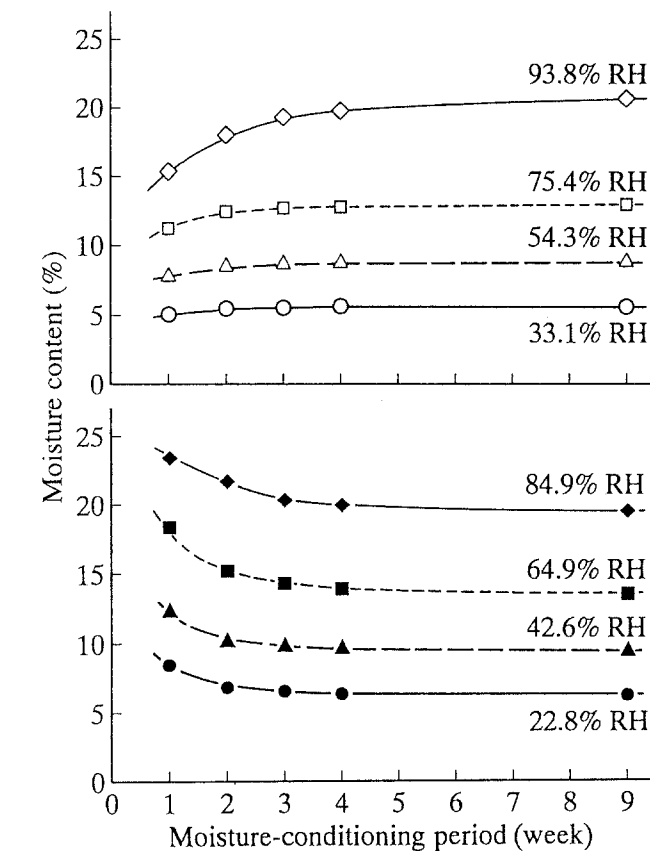
The tests were discontinued when the samples had broken down completely. Immediately after the test the wood samples were unsealed, and their width and thickness and the weight of the polyethylene films were measured. The MOE and MOR were calculated using the conventional method, which employs the obtained stress-strain diagrams and dimensional data. The moisture contents of the samples were also determined from the weights of the samples in a dried state and after moisture conditioning.

In the case of the stress relaxation test, movement of the cross-head stopped when the samples were deflected by 1.2 mm. This deflection corresponded to one-third of the breaking load of the samples conditioned in the highest relative humidity. Stress relaxations were then measured for 2 or 15 h. After the test, the samples conditioned for 3 and 4 weeks were again placed in the same desiccator and left for 5–6 weeks in a sealed state to avoid changes in moisture content. After these periods, the samples were subjected to a second measurement of stress relaxation. After this stress relaxation test, all the wood samples were unsealed, and their widths and thicknesses and weights of the polyethylene films were measured. The relaxation moduli during various relaxation periods were calculated by the load recorded on a chart and the dimensional data. The moisture contents of the samples were also determined as described above.

## Results and discussion

### Modulus of elasticity and modulus of rupture

The changes in moisture content of the samples moisture conditioned by the adsorption and desorption processes during the conditioning period are shown in Fig. 1. The moisture content increased the longer the conditioning period lasted during the adsorption process, and it decreased during the desorption process. These moisture content changes were greater in the samples conditioned at a higher relative humidity during both processes for the conditioning period examined. Changes in the moisture content of the samples from 4 to 9 weeks of the conditioning period was 0.9% in the maximum case conditioned during the adsorption process at 93.8% RH, 0.5% during the desorption process at 84.9% RH, and less than 0.2% for the other cases. As these results show, changes in the moisture content from 4 to 9 weeks were small.



**Fig. 1.** Changes in the moisture content of wood moisture-conditioned at various relative humidities (RH) during the adsorption (**top**) and desorption (**bottom**) processes with conditioning

cess at 84.9% RH, and less than 0.2% for the other cases. As these results show, changes in the moisture content from 4 to 9 weeks were small.

Figure 2 shows changes in MOE and MOR throughout the conditioning periods for the wood moisture-conditioned by the adsorption process. Wood conditioned at 33.1% RH showed an increase in MOE from 4 weeks to 9 weeks of conditioning. The MOE of the wood conditioned at higher RH reached a minimum and then increased according to the duration of the conditioning period. A similar trend was observed more clearly for the changes in the MOR.

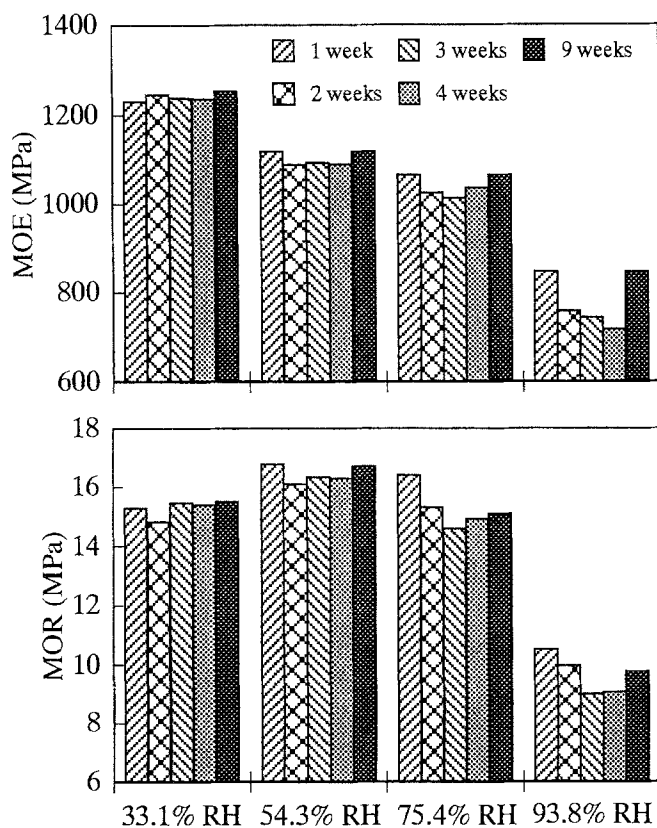


Fig. 2. Change in the modulus of elasticity (MOE) and modulus of rupture (MOR) of the wood moisture-conditioned at various relative humidities during the adsorption process with conditioning

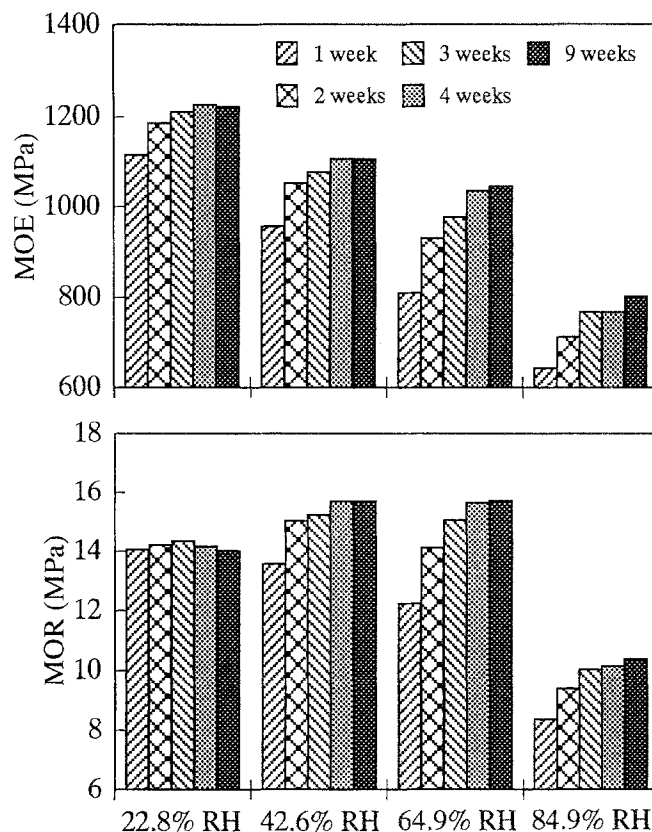


Fig. 3. Change in MOE and MOR of the wood moisture-conditioned at various relative humidities during the desorption process with conditioning

It has previously been found that the MOE of wood moisture-conditioned by adsorption reached a plateau with a relatively low moisture content and then decreased almost linearly with an increase in moisture content; furthermore, the MOR of wood similarly conditioned reached a maximum at around 7%–8% of the moisture content and then also decreased with increasing moisture content.<sup>6</sup> Similar trends are shown in Fig. 2; that is, the MOE decreased with an increase in RH, and the MOR reached a maximum at 54.3% RH (8%–9% moisture content) and then decreased with an increase in RH. Therefore, considering the changes in moisture content during the conditioning period as shown in Fig. 1, the decreases in MOE and MOR during the early stages of moisture conditioning in relatively high RH can be mainly attributed to increases in the moisture content. However, for the MOR of wood conditioned at 33.1% RH (5%–6% moisture content) and 54.3% RH, this explanation cannot apply because the MOR would be expected to increase at 33.1% RH and not to change so much at 54.3% RH during conditioning, considering the dependence of the MOR on moisture content as described above. Therefore, the fact that the MOR reached a minimum during the early stages of conditioning at relatively low RH suggest that the wood distant from a true adsorption equilibrium results in a lower MOR; this result is similar to the well-known phenomenon that wood in a state of nonequilibrium with respect to moisture shows a higher

degree of fluidity than wood in the equilibrium state.<sup>7</sup> In any event, it is notable that the MOE and MOR increased despite slight increases in the moisture content during the later stage of conditioning.

Figure 3 shows the changes in the MOE and MOR during the conditioning period for wood moisture-conditioned during the desorption process. Both MOE and MOR showed extensive increases during the early stages of moisture-conditioning, and then the increases diminished throughout the duration of the conditioning period, except in the case of wood conditioned in 22.8% RH, where the MOR reached a maximum and then decreased. However, both the MOE and MOR of wood conditioned at 84.9% RH showed significant increases between 4 and 9 weeks.

Relative to these results, Furuta et al.<sup>1</sup> found that the MOE of water-saturated wood with a drying history and heating history increased continuously with increased time left in water and with the time after cooling, respectively. They attributed these results to a long-term release of strains induced by drying and cooling. However, it is possible to think that wood with such strains exists in an unstable state, different from a true equilibrium or stable state. The authors found that wood swollen in certain organic liquids showed greater elasticity and strength and less fluidity as it approached the swelling equilibrium than wood swollen in water to the same degree. However, if the wood was far from the swelling equilibrium, it showed less elastic-

ity and strength and more fluidity than wood swollen in water to the same degree. These findings suggest that wood in an unstable state approaching a true equilibrium state has less elasticity and strength and greater fluidity than wood in a swelling equilibrium state.<sup>2</sup>

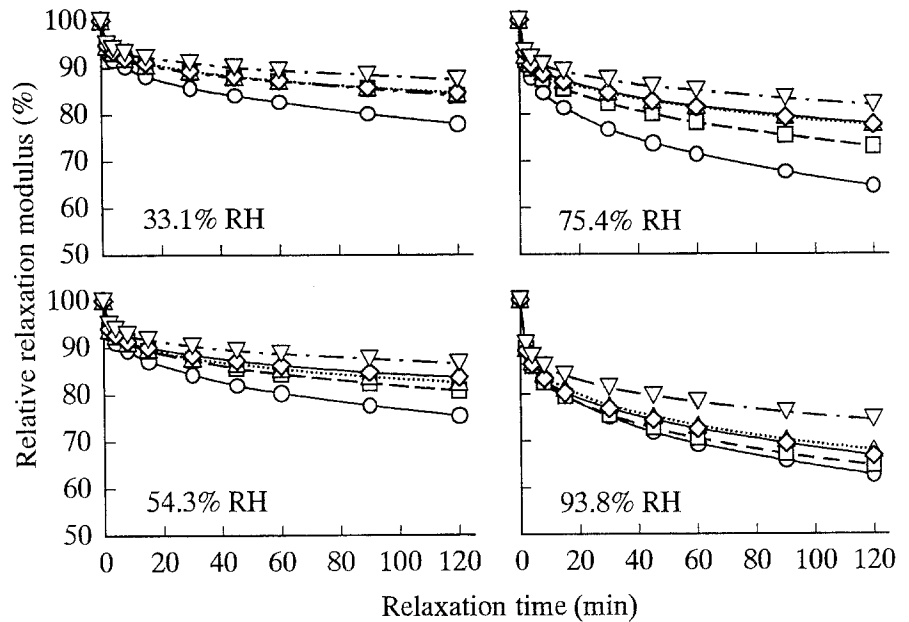
From the above-mentioned viewpoint, the results shown in Fig. 2 (that is, the significant increases in MOE and MOR during the later stage of the adsorption process where the moisture content scarcely increased) can be interpreted as a phenomenon that occurs during a stabilizing process, such as the reorientation of molecular chains of wood constituents toward a true swelling equilibrium by adsorption of moisture. On the other hand, the results of the desorption process shown in Fig. 3 (that is, only a slight increase or even a slight decrease in MOE and MOR during the later

stage of conditioning, except conditioning at the highest RH) is notable, because they suggest that the stabilization caused by reorientation of molecular chains, for instance, is more difficult during the desorption process than during the adsorption process or that in an unstable state such disorder of the molecular chains is also induced during the latest stage of the desorption of moisture.

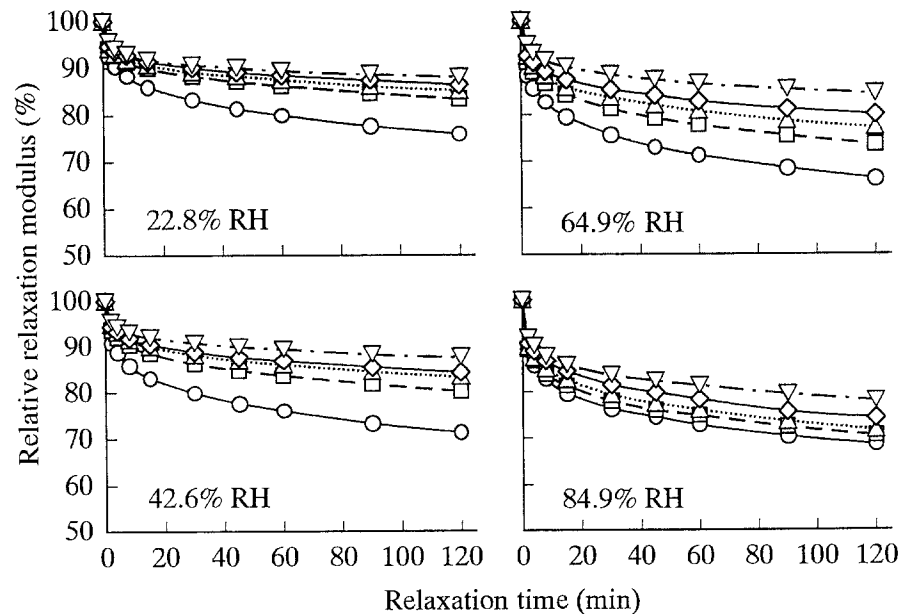
Changes in stress relaxation during the moisture-conditioning period

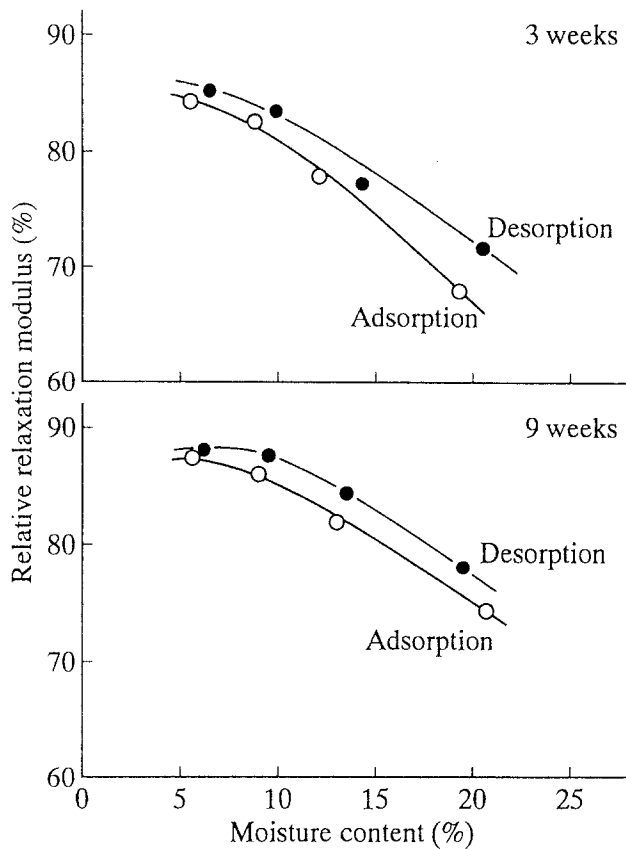
Figures 4 and 5 show changes in the relative relaxation modulus (relative to the modulus at the beginning of the test) as a function of the relaxation period of the wood

**Fig. 4.** Stress relaxation behavior of wood moisture-conditioned at various relative humidities for various periods during the adsorption process. *Circles*, conditioned 1 week; *squares*, 2 weeks; *triangles*, 3 weeks; *diamonds*, 4 weeks; *inverted triangles*, 9 weeks



**Fig. 5.** Stress relaxation behavior of wood moisture-conditioned at various relative humidities for various periods during the desorption process. See Fig. 4 for explanation of symbols





**Fig. 6.** Relation between the relaxation modulus 120 min after beginning the test and the moisture content. Wood samples were moisture-conditioned for 3 weeks (**top**) and 9 weeks (**bottom**)

moisture-conditioned by adsorption and desorption. The relaxation moduli of wood in both processes at all RHs tended to increase throughout the conditioning period despite increases in the moisture content of the wood during the adsorption process and decreases during the desorption process. This means that the fluidity of moisture-conditioned wood decreases throughout the conditioning period of both the adsorption and desorption processes.

Incidentally, it has previously been observed that the creep compliance of wood reaches a minimum at 10%–14% of the moisture content and then decreases with increasing moisture content.<sup>8</sup> However, that study reported that, at most, a 3% change in moisture content occurred; therefore, this result cannot be used when discussing the present results.

The moisture content dependence of the relative relaxation modulus at 120 min after the beginning of the test of wood conditioned for 3 and 9 weeks by the adsorption and desorption processes is shown in Fig. 6. At any point during the moisture conditioning, decreases in the relative relaxation moduli were observed with an increase in moisture content, though the increases were smaller in the lower moisture content range. Accordingly, it is clear that the fluidity of wood increases with an increase in the moisture content within the limits of the moisture contents examined. This result accounts for the decreases in the fluidity of wood

during the early stages of conditioning at any RH in the desorption process shown in Fig. 5 because the moisture contents decreased as the conditioning period progressed in these cases. It is not clear why the relative relaxation modulus increased between 4 and 9 weeks in the desorption process despite minimal decreases in moisture contents, or why the relaxation modulus during the adsorption process also increased despite increases in moisture content.

Comparison between stress relaxation for first and second measurements in the same wood sample

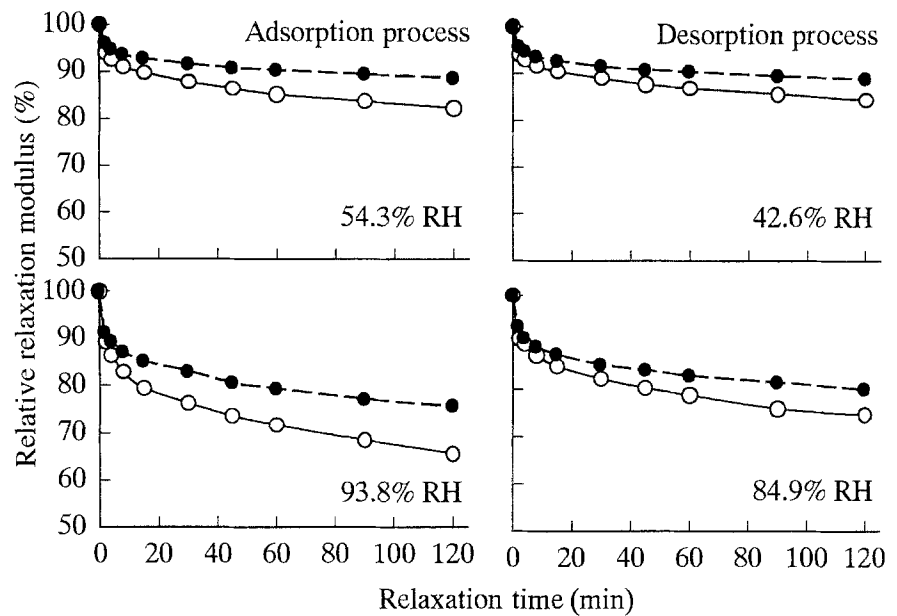
The results shown in Figs. 4 and 5 were obtained as averages from two wood samples conditioned separately; therefore, they may have been affected by variations among samples caused by their sampling positions. Moreover, because the results obtained from different samples conditioned for different periods were compared, they were also affected by the difference in moisture content based on the difference in conditioning periods. Hence, the stress relaxations were again measured 5–6 weeks after the first measurement using the same samples that had been conditioned for 3 and 4 weeks at the time of the first measurement. The moisture contents of these samples did not change, except in the case of the experimental error, because the samples were maintained in a sealed state and placed in the same desiccator in which the samples had been conditioned before the first measurement.

Figure 7 shows a comparison between the first and second measurements of the relative relaxation modulus of the samples conditioned for 4 weeks in 54.3% RH and 93.8% RH by the adsorption process and in 42.6% RH and 84.9% RH by the desorption process. The samples were loaded on the same side for the first and second measurements. The relaxation moduli obtained at the second measurement were distinctly larger than those for the first measurement in all cases. This means that fluidity of the wood conditioned by both adsorption and desorption decreases with the passage of time, even if the moisture content does not change.

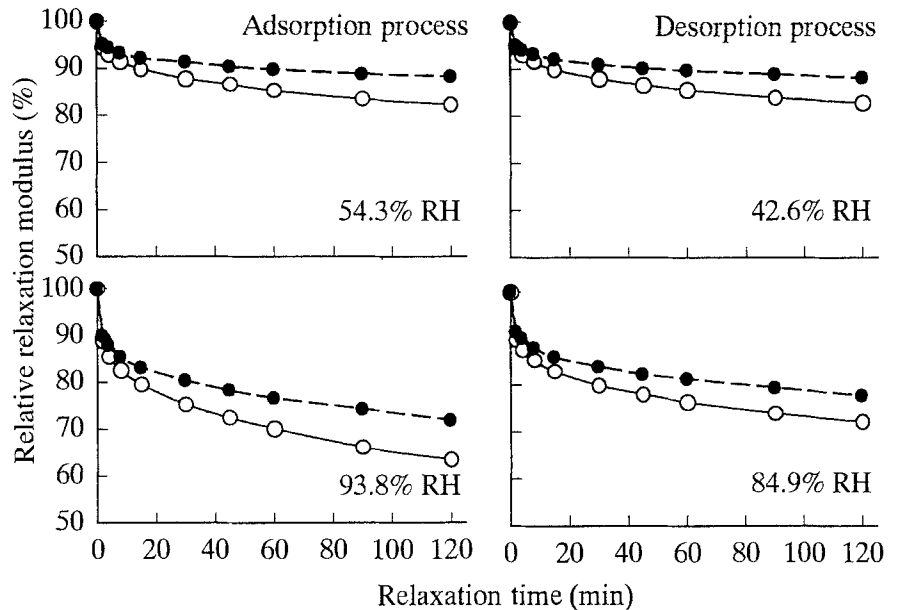
This result may have been affected by loading history or memory effect<sup>9</sup> based on the first measurement, though the samples were left in the desiccator 5–6 weeks after the first measurement. To examine this effect, the relaxation moduli of the samples that had been measured after 3 weeks of moisture conditioning were measured again by loading in the side opposite to that used for the first measurement. The results are shown in Fig. 8. The relaxations at the second measurements were also distinctly less than those at the first measurements. These results indicate that the memory effects can be disregarded when the samples are left for 5–6 weeks after the first measurement. Accordingly, these results confirm that the fluidity of wood decreases with time, even if the moisture content does not change.

It has been observed that wood requires a long time to reach equilibrium in terms of the adsorption or desorption of moisture (or both). Accordingly, wood in the process of adsorption and desorption has been thought to exist in a

**Fig. 7.** Comparison of the first and second measurements of stress relaxation of wood moisture-conditioned by adsorption (left) and desorption (right). The first measurement was of a wood sample conditioned for 4 weeks, and the second measurement was of the same sample about 5 weeks after the first measurement. The sample was loaded in the same direction for the first and second measurements. *Open symbols*, first measurement; *solid symbols*, second measurement



**Fig. 8.** Comparison of the first and second measurements of stress relaxation of wood moisture-conditioned by adsorption (left) and desorption (right). The first measurement was of a wood sample conditioned for 3 weeks, and the second measurement was of the same sample about 6 weeks after the first measurement. The sample was loaded in opposite directions for the first and second measurements. *Open symbols*, first measurement; *solid symbols*, second measurement



transitional state approaching true equilibrium.<sup>3</sup> Furthermore, as described above, it has also been found that the elasticity of wood in a water-saturated state increases with increased time left in water.<sup>1</sup> The authors interpreted these results as a phenomenon that occurs during the course of modifications of an unstable state distant from a true equilibrium state.<sup>2</sup>

From this point of view, decreases in fluidity with increases in the moisture-conditioning period during the adsorption and desorption processes observed in the present study can also be interpreted as a phenomenon that occurs during modification of an unstable state caused by the existing state of moisture, differing from that in true equilibrium. The results obtained in the present study suggest that such an unstable state affects fluidity more extensively than the

elasticity and strength of wood, considering the MOE, MOR, and stress relaxation results.

Incidentally, it has long been established that fluidity of wood increases extensively in a nonequilibrium state of moisture, and that such increases in the fluidity occur during the course of changes in moisture content or swelling.<sup>10</sup> The present study clearly indicates that a similar increase in fluidity occurs during moisture-conditioning stages, even if the moisture content of the wood does not change. The nonequilibrium state of moisture can be regarded as a highly unstable state for wood. Accordingly, the results in the present study should be considered for better understanding the abnormal mechanical behavior of wood in a nonequilibrium state of moisture.

## Difference in fluidity of wood moisture-conditioned by adsorption and desorption processes

The stress relaxations of wood moisture-conditioned by the adsorption and desorption processes can be compared in Fig. 6. Wood conditioned by adsorption showed greater relaxation (i.e., greater fluidity) than wood conditioned by desorption regardless of the conditioning period and RH. Considering the characteristics of the unstable state as described above, this result can be interpreted to mean that wood moisture-conditioned by adsorption is more unstable than that conditioned by desorption. This interpretation is reasonable and can be explained as follows: because trees live in a state swollen by water, the swollen state is the most stable state for wood. During the process of drying, a highly unstable state is induced. Such an unstable state is not easily modified by adsorption of moisture. Accordingly, wood moisture-conditioned by the adsorption process remains in a more unstable state than wood conditioned by the desorption process to the same moisture content.

## Conclusions

The elasticity and strength of wood moisture-conditioned by the adsorption process increase significantly during the later stages of conditioning, though the moisture content of the wood scarcely changes. During the desorption process these properties did not increase as much in the later stages of conditioning, though they increased during the early stages of conditioning when the moisture content markedly decreased. The stress relaxation of wood decreased with an increase in the conditioning period, regardless of the adsorption and desorption processes. The stress relaxations of wood with a similar moisture content were greater with the adsorption process than with the desorption process. These

behaviors were interpreted to be the result of an unstable state caused by the existing state of moisture different from that in a true equilibrium state or by modifications of such an unstable state. The present study demonstrates that the unstable states of wood induced during the course of drying, desorption, and possibly adsorption of moisture are slowly modified as wood approaches a true equilibrium state, and that such an unstable state affects the fluidity more extensively than the elasticity and strength of wood.

## References

1. Furuta Y, Norimoto M, Yano H (1998) Thermal-softening properties of water-swollen wood. V. The effect of drying and heating histories (in Japanese). *Mokuzai Gakkaishi* 44:82–88
2. Ishimaru Y, Narimoto S, Iida I (2001) Mechanical properties of wood swollen in organic liquids with two or more functional groups for hydrogen bonding in a molecule. *J Wood Sci* 47:171–177
3. Ishimaru Y, Arai K, Mizutani M, Oshima K, Iida I (2001) Physical and mechanical properties of wood after moisture conditioning. *J Wood Sci* 47:185–191
4. Furuta Y, Yano H, Kajita H (1995) Thermal-softening properties of water-swollen wood. I. The effect of drying history (in Japanese). *Mokuzai Gakkaishi* 41:718–721
5. Miyabe H (ed) (1968) In: Japan Society of Polymer Science: hand book of material and moisture [Zairyo to Mizu Handbook] (in Japanese). Kyoritu Shuppan, Tokyo, pp 240–262
6. Ishimaru Y, Minase T (1992) Mechanical properties of wood in various stages of swelling. I. Mechanical and swelling behavior of wood swollen in various organic liquids (in Japanese). *Mokuzai Gakkaishi* 38:550–555
7. Tokumoto M (1994) Creep and set of wood in the non-equilibrium states of moisture (in Japanese). *Mokuzai Gakkaishi* 40:1157–1164
8. Suzuki M, Nakato K, Aikawa K (1965) Frequency dependence of dynamic Young's modulus of wood and its relation to creep (in Japanese). *Mokuzai Gakkaishi* 11:76–82
9. Onogi S (1973) Principles of rheology (in Japanese), 3rd edn. Maki Shoten, Tokyo, pp 97–102
10. Grossman PUA (1976) Requirement for a model that exhibits mechano-sorptive behavior. *Wood Sci Technol* 10:163–168