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Differences in wood material responses for radial and tangential direction as measured by dynamic mechanical thermal analysis

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Wood is a complex cellular structure with different properties in the radial and tangential direction. Many researchers have measured dynamic properties in the longitudinal direction and a few in the radial direction but very little data can be found in the literature on dynamic mechanical properties in the tangential direction. The purpose of the work presented in this paper was to investigate the dynamic mechanical behaviour in the radial and tangential directions of wood (Pinus sylvestris). Testing was done in tension at 1 Hz with a Dynamic Mechanical Thermal Analyser. Properties in radial and tangential direction were different. The radial direction showed a higher elastic modulus and lower loss factor levels at temperatures between −120°C and 80°C. The tangential direction had on average a higher peak temperature than the radial direction for a loss factor peak around −80°C. It is the opposite of synthetic composites where the stiffer direction has a higher peak temperature. A loss factor peak at around 0°C was seen, most significantly in the tangential direction. This peak has scarcely been reported in the literature before. The distance between annual rings did not significantly affect the dynamic behaviour in the tangential direction. © *2001 Kluwer Academic Publishers*

1. Introduction

The interactions between polymers in polymer blends and in copolymers have been widely investigated using various dynamic measurement techniques. However, wood, which consists of a natural polymer blend of the three principal polymers cellulose, hemicellulose and lignin, has not been investigated to the same degree.

Dynamic mechanical analysis can give important information about the compatibility of wood and other polymers, information needed when considering the application of glue and paint to wood. The dynamic mechanical and dielectric properties of wood and wood products have mainly been investigated using dielectric measurement and torsional pendulum.

Wood is an anisotropic material with three principal axes; longitudinal, radial and tangential, the latter two sometimes being called transverse directions. Only a few references to the difference between the radial and tangential direction measured with dielectric methods have been found [1–5]. The longitudinal direction was found to exhibit higher dielectric constant (ε') and dielectric loss factor (ε'') than the radial and tangential directions for oven-dried wood. The radial direction often has slightly higher ε' than the tangential direction. The anisotropy of the dielectric constant in the transverse directions is mostly influenced by latewood fraction and cell arrangement [5].

Only one reference was found for differences in radial and tangential direction measured with dynamic mechanical measurements [6]. It was on water-swollen softwood (Japanese cypress) with temperature varied between 10°C and 95°C. A loss factor ($\tan \partial$) peak in transverse direction at 80°C was attributed to micro-Brownian motions of matrix substances. The peak was above 95°C for the longitudinal direction.

Handa [7] measured the dielectric properties of beech in the longitudinal direction and the dynamic mechanical properties in tangential direction at different moisture contents (m.c.) below 0°C. Dry wood was shown to have a dielectric loss peak at about −105°C (30 Hz). This peak decreases in height but does not move when the m.c. is increased, and is not visible above 1.7% m.c. At low m.c. a new peak appears at −40°C (0.7%) and then moves to lower temperatures with increasing m.c. At 5.6% the peak is at −92°C and at 8.7% at −108°C. Peak height increases with increasing m.c. Maeda [8] measured the dynamic mechanical properties of bamboo at 10 Hz and obtained a loss modulus (E'') peak at −87°C for 3% m.c. and at −97°C for 6%. The same experiments with Japanese cedar gave a loss modulus peak at −93°C for 7% m.c. The loss modulus peak is normally a few degrees lower than the $\tan\partial$ peak. Kelley [9] measured the longitudinal properties of spruce in dual cantilever and at 1 Hz at different m.c. with a Dynamic Mechanical Thermal Analyser (DMTA). At a m.c. of 10% a tan ∂ peak at about −85°C with a tan∂ height of about 0.025 was observed. For the same m.c. a peak at about 20°C was also seen which was thought to be associated with the T_g of hemicellulose and at another peak at about 80°C, the T_g of lignin. At a m.c. of 5% these peaks move to approximately −75°C, 70°C and 120°C. Salmén [10] measured dynamic mechanical properties of water-saturated Norway spruce and determined the T_g for lignin to be about 100°C, measured both longitudinally and transversely at 10 Hz.

When measuring dynamic mechanical properties in tension the results are presented as elastic modulus (E'), loss modulus (E'') and loss factor (tan $\partial = E''/E'$). A sinusoidally varying strain is applied to the specimen with the measurement being made at constant frequency with varying temperature, or the opposite. In a temperature scan, a peak of tan ∂ or E'' in the graph indicates a certain molecular movement having the same relaxation time as the applied frequency. In this paper a frequency of 1 Hz was used and the temperature was increased from −120°C to 80°C by 2°C/min. The tan ∂ peak measured in a DMTA at 1 Hz can be 15–20°C higher than the true glass transition temperature [11], but for simplicity tan ∂ peaks are often taken as being representative of T_g peaks. Other transition peaks than T_g are also dependent on the frequency. When analysing dynamic mechanical properties, it is the changes in peaks observed in one analyser that should be studied. Comparison with absolute values of peak temperatures obtained with other machines should be treated with caution since the same material measured in several different machines, even those from the same manufacturer, will give different responses [12]. This is due to temperature measurement errors from the thermocouples, differences in heat transfer to the specimen and the way the machines carry out measurements. Variation due to the way in which the operator handles the specimens and equipment also has a contributory effect on the results.

Wood is also a highly complex cellular composite. Softwood (earlywood) consists mainly of tracheids, which are closed tubes with tapered ends with approximately 40 × 40 μm cross section and 2–4 mm length. The tracheids are oriented in the direction of the stem, the longitudinal direction, Fig. 1. Latewood tracheids have thicker cell walls than earlywood tracheids. Radially oriented cells are known as ray cells, and are much shorter than tracheids and have thin cell walls. In the wood cell walls, cellulose molecules are arranged in the form of microfibrils with the polymers hemicellulose and lignin which act as matrix material. Dry hemicellulose and dry lignin have glass transition temperatures (T_g) of 150–220°C and 130–205°C respectively, while cellulose glass transition varies between 200°C and 250°C [13].

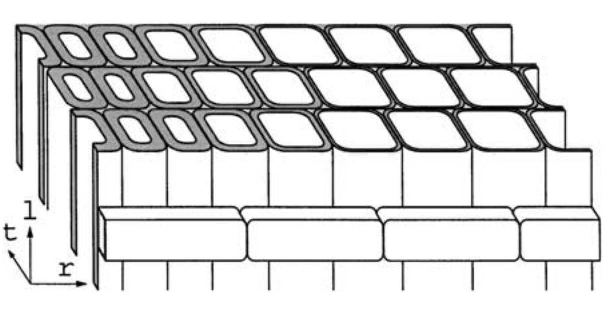

Figure 1 Schematic drawing of the structure of softwoods. The direction of growth is to the left. On the right can cross-sections of earlywood tracheids be seen, and to the left latewood tracheids. On the side of the tracheids a single row of ray cells has been drawn.

Many researchers have measured dynamic properties in the longitudinal direction or in the longitudinal and one of the transverse directions. Very little data can be found in the literature on dynamic mechanical properties in the tangential direction. Unfortunately, the directions in which measurements were made are poorly defined in some papers on dynamic mechanical measurements [10, 14–17]. This indicates a lack of understanding in regard to the important differences in wood anisotropic behaviour due to its complex cellular structure. The purpose of the work presented in this paper was to investigate the dynamic mechanical behaviour in the radial and tangential directions of wood. The effect of different levels of moisture content was also considered. The reason for this work was the need for understanding of the properties of wood for future studies of interaction with synthetic polymers such as glue and lacquer. The dynamic mechanical properties of wood were tested in tension using a DMTA.

2. Method and material

The wood used was clear sapwood from Scots pine (Pinus sylvestris). Slices 1.5 mm thick were sawn perpendicular to the longitudinal direction. From these slices, smaller specimens (3 × 1.3 × length (mm)) were cut using a razor blade and finally smoothed using 80-grade sandpaper. The wood had been dried without external heating and was stored in a laboratory climate. Only one radial specimen (R1) had been exposed to temperatures exceeding 80°C. Tangential (T) and radial (R) specimens were paired from two adjacent slices. Before testing the specimens they were conditioned at room temperature in a sealed container over a saturated solution of Magnesium Chloride ($MgCl_2$) or Lithium Chloride (LiCl) which gave 33% or 11% relative humidity (RH) respectively at 20–30°C [18]. This corresponds to a moisture content of 6–7% for 33% RH and 3–3.5% for 11% RH [19].

After removing a specimen from the conditioning container, each specimen was then mounted in a Dynamic Mechanical Thermal Analyser MkIII (DMTA) from Rheometric Scientific at room temperature. Clamps were attached at both ends at room temperature with a torque force of 0.1 Nm. The DMTA chamber closed two minutes after the specimen was removed from the conditioning container. Cooling down to the starting temperature of −120°C took 20 minutes. Two types of specimen were used, radial and tangential, Fig. 2. In both cases the clamps were applied against the transverse plane (RT-plane). A Sartorius scale with an accuracy of 0.1 mg was used to check the weight of the specimens, which were in the range 40–60 mg. The

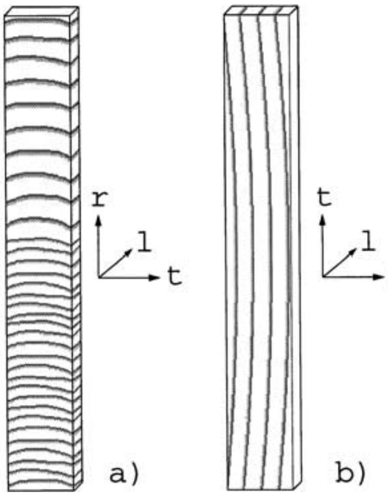

Figure 2 DMTA samples for tensile testing: (a) for the radial and (b) for the tangential direction.

DMTA chamber had no climate control, but the weight loss during measurement up to 0°C was deemed negligible. It was measured by weighing specimens before measurement and after measurement to 0°C.

The DMTA was used in tension mode at a frequency of 1 Hz. Runs were made from −120°C to 80°C at 2°C/min and data was collected at intervals of about 1°C. The length and dynamic strain varied between different specimens; however, all strains were considered to be within the linear viscoelastic range. Specimens varied in length between 12 mm and 24 mm, and the dynamic average strain varied between 0.02% and 0.05%. When measuring in tension, a static force equivalent to about twice the force necessary for the dynamic amplitude was applied to prevent buckling.

The tan ∂ values obtained during measurements were low and only slightly above background noise. The data was smoothed using an average of seven consecutive values of tan ∂. To further reduce the effects of noise, the curves were an average of two or more DMTA measurements on the same specimen. The temperature of the tan ∂ peak was taken as the location of the maximum value.

Specimen density was measured at RT by immersion in mercury. The specimens had been conditioned in 33% RH. Specimen weight was between 40 and 60 mg and was measured on a scale with 0.1-mg resolution. The specimen was then pushed down by the use of two needles into a tared jar filled with mercury placed on a scale with 1-mg resolution. The measured mass is the volume of the mercury and the mass of the specimen. Specimen volume was then calculated as the mercury mass subtracted by specimen mass and then divided by the density of mercury (13.55 kg/dm^3).

3. Results and discussion

There was a clear difference in the elastic modulus (E') in the radial and tangential directions, the elastic modulus being much lower in the tangential than in the radial direction, Fig. 3. At 25°C the radial value averaged 1.26 GPa and the tangential value 0.66 GPa, the radial being some 90% higher. This is consistent with the difference in static Young's modulus for Pinus sylvestris [20], which at room temperature is 93% higher in the radial direction (1.14 GPa) than in the tangential direction (0.59 GPa). The elastic modulus was only a few percent higher than the static Young's modulus. The measurement of elastic modulus with DMTA was very reproducible. No significant difference in E' between 3% and 6% moisture content was observed.

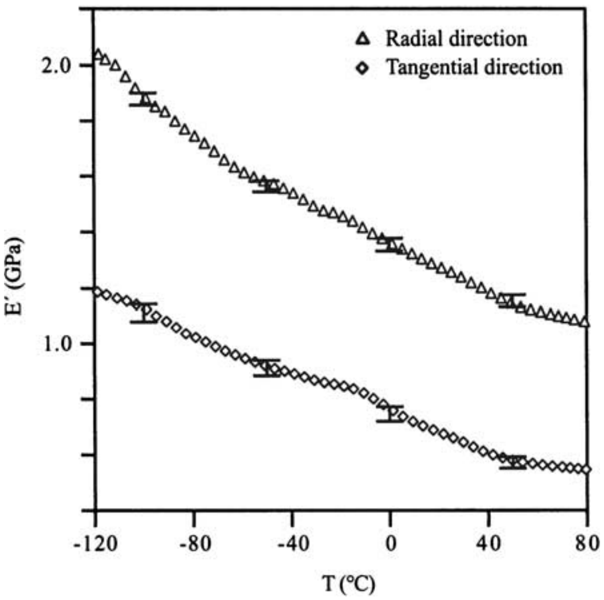

Figure 3 Elastic modulus (E') for radial and tangential directions. Error bars indicate 95% significance in a one-sample t-test.

The reason for the large difference in modulus in the radial and tangential directions remains unclear. Since latewood has much higher density and therefore a higher modulus than earlywood, it was surprising to find that the radial direction, with its serial early- and latewood cells, had a higher modulus. Several explanations for this behaviour have been proposed. One theory is that ray cells oriented in the radial direction act as stiffening ribs. However, the ray cells in Scots pine have thin cell walls so their stiffening effect cannot be significant [21].

The radial cell walls have pits, and therefore the cellulose microfibrils are distorted from the longitudinal direction, giving the radial cell wall higher transverse stiffness, although this is not sufficient to cause the large differences observed.

The irregular hexagonal cell structure in Scots pine and many other softwoods gives a higher stiffness in the radial direction. According to [22] the stiffness for wood is about 50% higher in the radial than in the tangential direction. The Young's modulus for regular hexagonal cellular structures in the transverse direction is proportional to the square root of the density [22].

If the latewood is assumed to have a density of 1.2 g/cm^3 and the earlywood 0.25 g/cm^3, the Young's modulus would be 23 times higher for the latewood. Compression experiments have shown that earlywood has a much lower stiffness in the tangential than the radial direction whilst latewood was observed to have comparable stiffness in the two directions [21].

When using the rule of mixtures and the inverse rule of mixtures, reasonable data of the modulus in radial

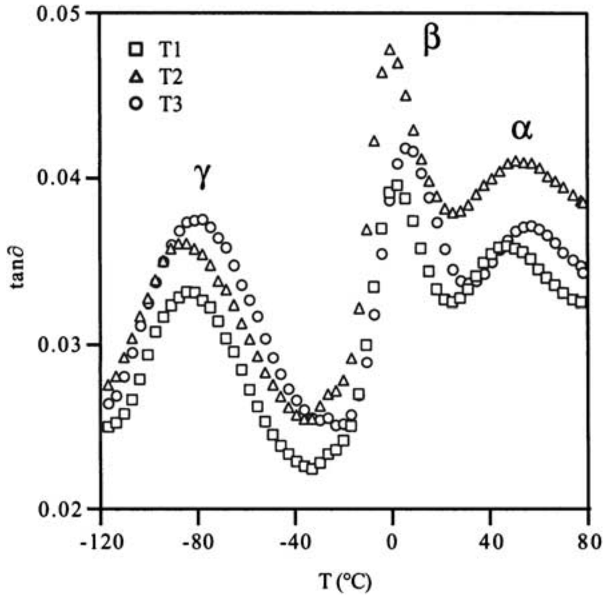

Figure 4 Three different tangential specimens (T1, T2, T3) conditioned in 33% RH prior to testing. Specimens T1 and T3 had narrower annual rings than specimen T2.

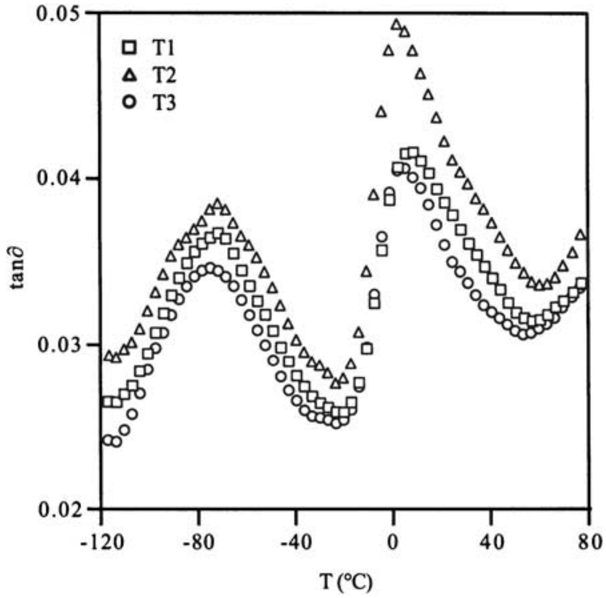

Figure 5 Three different tangential specimens (T1, T2, T3) conditioned in 11% RH prior to testing. Specimens T1 and T3 had narrower annual rings than specimen T2.

and tangential directions can be obtained from measurements on separate early- and latewood specimens [23].

This low tangential modulus in the earlywood is one plausible explanation for the difference in modulus between the two directions, though probably not the last explanation to be presented. A tentative theory proposed by the authors of the present paper is that the latewood could be considered as this stiff ribs which are already buckled at a small angle, with very soft earlywood lying between the ribs, thus resulting in the low tangential modulus that is observed.

TABLE I Loss factor peak temperatures for tangential (T) and radial (R) specimens conditioned in 11 and 33% RH

11% RH	R1*	R2	R3	R4	T1	T2	T3
α	—	—	—	—	—	—	—
β	16	9	—	34	8	3	4
γ	−82	−80	−82	−88	−72	−71	−75
33% RH	**R1***	**R2**	**R3**	**R4**	**T1**	**T2**	**T3**
α	40	42	—	57	46	50	56
β	2	−7	—	−7	2	−1	7
γ	−76	−84	−95	−89	−83	−87	−79

*Thermally treated above 80°C.

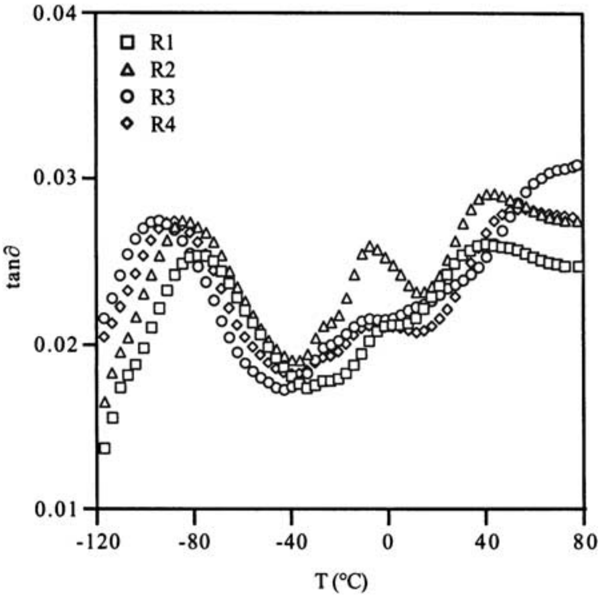

Figure 6 Four different radial specimens (R1, R2, R3, R4) conditioned in 33% RH prior to testing. All specimens were paired, and specimen R1 had been subjected to thermal treatment.

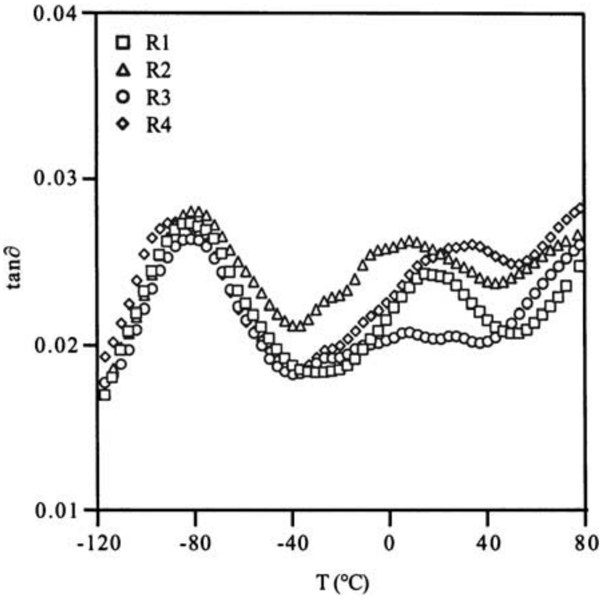

Figure 7 Four different radial specimens (R1, R2, R3, R4) conditioned in 11% RH prior to testing. All specimens were paired, and specimen R1 had been subjected to thermal treatment.

The radial and tangential directions also differed in their response as far as loss factor levels and peak temperatures are concerned, Figs 4–7. The peak temperatures are listed in Table I, peaks being labelled α, β and γ in order of decreasing temperature, Fig. 4. Several consecutive measurements on the same specimen were made, as mentioned earlier. Repetitive measurements on specimens initially conditioned in 33% RH were almost identical over many measurements, but the reproducibility was lower for specimens conditioned at 11% RH. The differences in γ-peak temperature between paired specimens were also reproducible. Since the specimens were conditioned in the same way and the measurements carried out using identical methods, the only possible reason for the differences are differences in initial moisture content.

Our results show that the specimens cut in the tangential direction had higher γ-peak temperature when conditioned in 11% RH than in 33% RH. The γ-peak temperatures found in the radial direction did not show any difference between 11% and 33% RH. The scatter in measurements was too large.

For absolutely dry wood a peak is situated at about −105°C, and with the slightest moisture contents (0.7%) another peak appears at about −40°C (30 Hz, dielectric) [7]. With increasing moisture content the −40°C peak moves towards lower temperatures whilst the peak at −105°C decreases in height and eventually disappears at about 2% m.c. The peak at about −105°C for dry wood is due to the movement of methylol side groups [7, 8]. The peak dependent on moisture content is assumed by many to be associated with movement of a methylol side group bonded to water molecules [7, 8]. The methylol group is present in all the three main polymers found in wood, although hemicellulose is assumed to adsorb most of the moisture.

The γ-peak which initially appears at −40°C could be due to adsorption of water on the methylol groups [7, 8]. However, if water molecules are adsorbed onto the methylol groups, the complex should become larger and stiffer with increasing moisture content and the peak shift towards increasing temperatures, not decreasing, which is the case. It is possible that water molecules are only adsorbed as a monolayer on the methylol groups, thus shifting the peak from −105°C to −40°C due to increase of the complex size. Further adsorption of water acts as a plasticizer making movement of the complex easier and lowering the peak temperature, which would better explain the movement of the γ-peak.

The γ-peak could also be due to site to site exchange of water [9], since one of the reasons proposed against a side-group movement was the absence of a drop in elastic modulus. In the present work a tendency for the modulus to fall was noted, Fig. 3. In Polyamide 6 several secondary relaxations are present, which all move with changes in moisture content. One of these, a peak which moves from about −40 to −80°C increasing from zero to 8% m.c., is argued to be due to the motion of chain segments and polymer-water complex [24].

Specimen R1 was the only specimen thermally treated above 80°C, being dried at 103°C for 48 hours and then used in two runs in the DMTA up to 150°C. The thermal history of specimen R1 was clearly visible as a change in the position of the γ-peak for 33% RH. The increase in temperature by at least 10°C for R1 compared to the untreated radial specimens indicates lower moisture content in the specimen. In [25] wood dried at 115°C for 120 hours showed a two percent points lower moisture content than wood dried at 82°C for 120 hours. Why the R1 specimen in the present work seems to have a higher moisture content following conditioning in 11% than in 33% RH is not clear.

The loss factor β-peak is seldom referred to in the literature. The tangential specimens showed this peak at 3°C to 8°C for 11% RH and −1°C to 7°C for 33% RH. The radial specimens had a much lower tan ∂ level for this peak. For one of the specimens the peak was missing entirely, Fig. 7. The peak temperatures for the radial specimens varied between −7°C and 34°C. The scatter was very large. No correlation between the peak temperatures and the moisture content or between peak temperatures and direction was apparent, Table I.

Only a few references to a peak similar to the β-peak were found. In [26] measurement in radial direction on hardwood with a torsion pendulum with damped oscillation revealed a small peak at 10°C. This was attributed to the local mode of wood components related to water. Also in [27] a peak at 10°C was seen in low-frequency torsional pendulum measurements on beech in the longitudinal direction with approximately 10% moisture content. In [8] a peak at 15°C was seen for longitudinal direction of Japan cedar with 7% moisture content. The peak was not discussed in the reference. It is possible that these references show the same peak as the β-peak in the present work.

In the tangential samples, Fig. 3, a clear drop in the modulus was seen at the transition temperature. This means that the peak was a true relaxation process and not merely related to movement of water molecules. The β-peak could therefore be a hemicellulose glass transition peak. The hemicellulose molecules consist of 150–200 sugar monomers in a heavily branched structure. Different compositions of hemicellulose have been found, and more than one type of hemicellulose is present in Scots pine. In low-molecular-weight amorphous polymers T_g is dependent on the molecular weight, a lower molecular weight giving a lower T_g [28]. If different types of hemicellulose with different molecular weights were phase separated this could lead to regions where the hemicellulose glass transition was lower, and thus produce the β-peak which was observed. Low-molecular-weight regions might also adsorb more water and are one possible explanation for the differences in the observed specimen moisture content.

The inconsistent appearance of the β-peak in the radial samples is not related to the presence, or otherwise, of certain molecules when testing. Radial specimens tested in the present work could sometimes show this peak in one measurement and not the following one. The peak at about −90°C is consistent, and the behaviour of the peaks is probably related to the measurement method used. It would seem that the method used to make the measurements on the radial samples does not produce consistent results. At low temperatures the modulus was high and the material apparently stable. However, at around 0°C the modulus of the material dropped and the DMTA tests ceased to give consistent results.

The specimens conditioned in 33% RH show an α-peak at 40°C to 60°C. According to Kelley [9] this is the glass transition peak of the hemicellulose. The specimens conditioned in 11% RH do not show the α-peak since they are drier and the α-peak therefore moved to temperatures above 80°C. Since the experiments were performed in a non climate-controlled chamber, the specimens could only be expected to maintain a relatively constant m.c. up to about 0°C. The scattering seen in the α-peak temperatures was therefore not surprising.

The radial specimens had lower tan ∂ values than the tangential specimens, Figs 4–7. A lower tan ∂ value indicates that less material is being moved. A tendency for the tangential direction to have higher peak temperatures for the γ-peak was observed. The longitudinal direction in wood and polymeric composites has a higher tan ∂-peak temperature than the transverse directions [29–31]. The difference in T_g between different structural directions is influenced by the underlying structure [31]. For wood, the stiffer radial direction had lower peak temperature. One possible hypothesis for this discrepancy may be that latewood was strained more in the tangential direction than in the radial direction, whilst most of the strain occurred in the earlywood in the radial direction. Most of the signals for E', E'' and tan ∂ then came from latewood when measured in the tangential direction and from earlywood in the radial direction.

Tangential specimens are shown in Figs 4 and 5. T2 is a specimen with wide annual rings whilst specimens T1 and T3 have narrower annual rings. The density of specimen T2 was higher than T1 and T3. T2 was between 0.53 and 0.54 kg/m^3 and T1 and T3 were between 0.48 and 0.49 kg/m^3. The radial samples had a density between 0.46 and 0.49 kg/m^3. The elastic modulus did not differ significantly, but the tan ∂ values at the α- and β-peaks are higher for the specimen with wide annual rings. The higher values were probably related to the higher density. The experiments were reproducible for the two similar tangential specimens, and the distance between annual rings did not have any marked effect on the dynamic behaviour.

4. Conclusions

The dynamical mechanical properties in radial and tangential directions of wood were different.

It is possible to measure the stiffness of wood by using DMTA and get good reproducibility and a relevant level of stiffness for radial and tangential directions.

The stiffness could easily be measured as a function of temperature up to room temperature. The measured radial stiffness was about two times the tangential stiffness, as expected.

Secondary loss peaks had lower tan δ levels measured in the radial direction than in the tangential direction.

The secondary transition peaks found testing wood in the tangential direction had higher temperatures than those measured in the radial direction did. This is in conflict with synthetic composites where the higher peak temperatures are found in the stiffer direction.

A loss factor peak at around 0°C was seen in the tangential direction. This peak has scarcely been reported in the literature before.

Measurements in the tangential direction gave reproducible values of loss factor, whilst measurements in the radial direction did not give enough signals to allow evaluation of the peaks from 0°C and above. All measurements were reproducible on the same specimen if temperatures did not exceed 80°C.

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