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AXIAL STIFFNESS AND SELECTED STRUCTURAL PROPERTIES OF YEW AND SPRUCE MICROTENSILE SPECIMENS

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ABSTRACT

Compared to other gymnosperms, the longitudinal modulus of elasticity (MOE_L) of yew wood is relatively low in spite of its high raw density. This interesting relationship, however, has only been established by means of standardized macro specimens so far. Therefore, the goal of this study was to find out if the low MOE_L can be confirmed for a lower hierarchical level. For this purpose, microtensile tests have been performed on Common yew specimens and, so as to serve as a reference, on Norway spruce specimens as well. Furthermore, the microfibril angles (MFA) of the specimens were determined by the pit aperture method in order to shed more light on the structure-function relationship.

The results reveal a lower MOE_L for yew than for spruce and there are indications that the reason for it must be present at both the macro and micro level. A significantly greater latewood MFA was found for yew compared to spruce. This is discussed further and the compliant behaviour of yew wood is attributed to this fact.

KEY WORDS: Common yew (*Taxus baccata* L.), Norway spruce (*Picea abies* [L.] Karst.), modulus of elasticity, longitudinal strain, micro tensile test, microfibril angle, Taxus baccata instead of Taxus baccate

INTRODUCTION

In literature, it is widely accepted that mechanical properties (such as stiffness) of clear softwood are largely influenced by density (e.g. Lohmann et al. 2003), often with a linear proportional relationship. This relationship may be true for stems grown at the same site under similar conditions, within a species. A species-spanning comparison, however, reveals at least one distinctive exception to this "rule": common yew.

In view of its superior raw density (~ 620–720 kg m^{-3} at 11-12% equilibrium moisture content), the remarkable tensile and compressive strength of yew and its hardness in all three orthotropic directions are not surprising. But interestingly, its axial stiffness is relatively low compared to other gymnosperms. At least one might come to this conclusion when evaluating the few published studies dealing with the longitudinal MOE (MOE_L) of yew, mostly determined in 3-point bending on standard specimens. Only Sell's (1997) data are roughly in the order of magnitude (15700 MPa) one would expect, in contrast to predominantly much lower values (6300 to 12000 MPa) published by other authors (Sekhar and Sharma 1959, Jakubczyk 1966, Wagenführ 2000). Key factors contributing to this exceptional mechanical behaviour of yew, however, are as yet unknown.

Knowing the density of wood as a measure of mass per volume unit provides no information about the qualitative composition of the cell wall. However, particularly its cellulose content and its orientation in tracheid cell walls are involved in determining the stiffness of wood. In recent years, several authors (e.g. Cave and Walker 1994, Reiterer et al. 1999) emphasized that the microfibril angle (MFA) of the dominant cell wall layer S_2 is capable of effecting large changes in the longitudinal stiffness of wood. A large MFA usually results in a low MOE_L (Reiterer et al. 1999). Therefore, a mean MFA of yew being larger than that of other gymnosperms is a conceivable explanation for the low MOE_L in spite of its high density.

In the majority of cases, MOE_L is determined by means of 3- or 4-point bending tests. In our own 3-point bending tests (Keunecke et al. 2007a) applied on yew and spruce members, we took a closer look at the axial deformation. Other than the slightly lower MOE_L compared to spruce, the high axial strain of yew in the linear-elastic and plastic pre-peak regime was the most conspicuous result. To be precise, the peripheral axial strain was about 50 percent higher for yew than for spruce. These findings might also indicate a large MFA, even though larger extensibility for specimens with larger MFA has only been established for single fibers (Page et al. 1971, Page and El-Hosseiny 1983) and thin wood foils of 200 μm thickness (Reiterer et al. 1999).

The objectives of this study were

- to ascertain, by means of small-scaled specimens loaded in longitudinal tension, if the low MOE_L of yew can be confirmed for a lower hierarchical level. Since wood is a multi-scale material, the results may indicate if the cause for the low stiffness of yew is present on both hierarchical levels.
- to find out if there are differences between yew and spruce concerning the intra-annual MFA distribution. The results may provide a basis for conclusions regarding the structure-function relationship.

For this purpose, micro tensile tests parallel to the grain have been carried out in this study in order to determine MOE_L. The size of the small-scaled "dog-bone" specimens allowed documenting selected micro structural features such as MFA. It has to be considered that MOE_L is a material parameter which also depends on the boundary conditions of the experiment. For comparison reasons and in order to evade the "size effect", the tensile tests have additionally been applied on Norway spruce under identical circumstances.

In earlier micro tensile tests on thin wood samples (Ifju and Kennedy 1962, Biblis 1970, Bariska and Bosshard 1974, Michon et al. 1994), mechanical properties (i.e. mostly microtensile strength) of individual tissues such as earlywood and latewood have been determined, primarily by means of tangential sections. Volkmer and Wagenführ (2005) determined material properties in the radial-tangential plane. Frühmann et al. (2003) used radial sections to observe crack propagation *in situ* in an environmental scanning electron microscope. Even though not interested in the fracture behaviour, we chose radial sections as well, mainly for one reason: We wanted to measure the orientation of cross field pit apertures as an approximation for the MFA of our specimens. This method is restricted to radial walls.

Numerous researchers used this method to measure MFAs (Pillow et al. 1953, Dadswell and

Nicholls 1960, Hiller 1964, Cockrell 1974). In contrast to averaging techniques such as X-ray diffraction (where large numbers of tracheids, mostly by means of tangential sections, are sampled simultaneously), the pit aperture method allows the MFA measurement of individual tracheids.

MATERIAL AND METHODS

Micro tensile tests were performed on common yew (*Taxus baccata* L.) and Norway spruce (*Picea abies* [L.] Karst.) specimens. The axial elastic modulus MOE_L of the specimens was determined and selected structural properties were documented. The investigations were divided into preliminary tensile tests and main investigations; an overview of the experimental set-up is given in Tab. 1.

Tab. 1: Overview of experimental setup and methodology

	number of specimens	determined properties	method / equipment
preliminary tensile tests	20 x yew 20 x spruce	• tensile strength	• micro tensile stage • digital micrometer
		• strain concentrations	• CCD-camera • strain-mapping software
		• sliding between specimen and clamps	• micro tensile stage • stereo microscope
main investigations	41 x yew 40 x spruce	• MOE_L	• micro tensile stage • video extensometer • SEM-images • image analysis software
		• cell wall area (%), density	• SEM-images • image analysis software
		• MFA, radial cell diameter	• light microscope • image analysis software

Specimens

As basic raw material, small wood cuboids (Fig. 1a) of 45 mm (L) x 9 mm (R) x 10 mm (T) were cut from the adult heartwood (at breast height) of two yew and two spruce stems from stands close to Zurich, Switzerland (at an altitude of about 500 m above sea level). Compression wood was carefully avoided. The samples were taken at a sufficient distance from the pith where the curvature of the growth rings can be neglected and the material can be treated as simply orthotropic. The "dog-bone" shaped specimen outline with moderate curvature for cross-sectional reduction (Fig. 1 and 2) was punched into the cuboids (Fig. 1b) by means of a stamping template with razor blades. One to four tree ring boundaries were located in the narrow section. After saturating the cuboids with water, a 220 µm thick radial slice with the following geometry was cut out of each cuboid using a sledge microtome (Fig. 1c): length over-all 45 mm (longitudinal direction), width of the narrow section 3.6 mm (radial direction), and thickness ~220 (± 20) µm (tangential direction). The specimen thickness was controlled with a digital external micrometer.

For the purpose of light-microscopical investigations, we cut a second slice out of each cuboid (Fig. 1f), this time 20 µm thick. Consequently, this second slice was originally located directly adjacent to the tensile specimens in the small cuboids. After cutting, we separated the central part of the slice (Fig. 1g) and mounted it on a slide for transmission light microscopy (Fig. 1h).

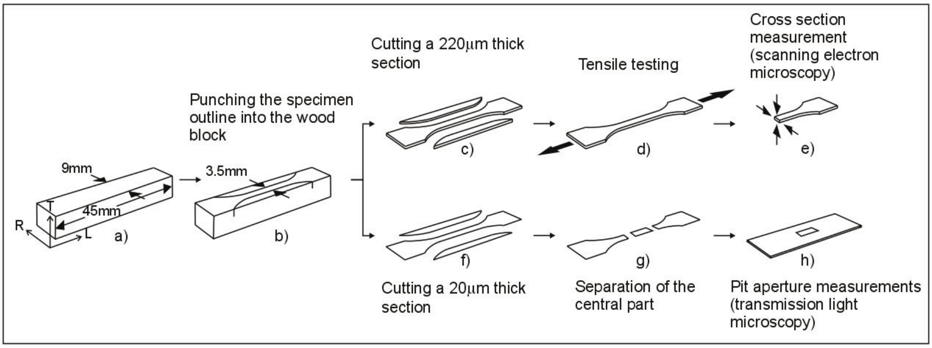

Fig. 1: Procedure of sample preparation for preliminary / main tensile tests and for wood anatomical measurements

Boundary conditions of the tensile test

Displacement-controlled tensile tests were performed under standard climatic conditions (20°C, 65% RH) using a micro tensile stage (Deben, Fig. 2) with fixed clamps and a load cell of 300 N maximum capacity. The specimens were accurately aligned in order to avoid load-eccentricities causing shear stresses. The free length of the specimens between the clamps was 34 mm. Precise strain measurement was assured by video extensometry. The contrast marks were fixed within the narrow section of the specimens (Fig. 2) with a distance of 12 mm between the marks, which is the gauge length that the percent elongation calculations are based on.

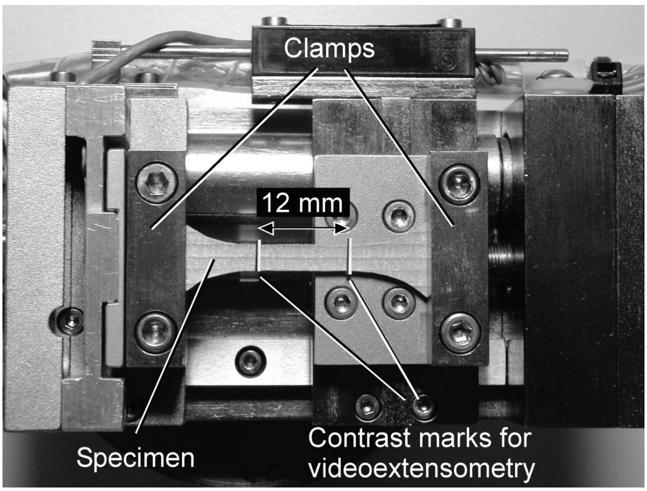

Fig. 2: Top view of the micro stage with a specimen readily prepared for a tensile test

Preliminary tensile tests

Preliminary tensile tests were performed on 20 yew and 20 spruce specimens with the same geometry as used in the main tests. This was necessary for two essential reasons:

- The main tests were performed only within the linear-elastic range in order to avoid damage to the specimens that were subsequently subjected to anatomical evaluation. Therefore, complete stress-strain curves up to specimen failure had to be recorded in preliminary tests. By this means, the stress limit where the specimens had to be unloaded again, could be defined for the main tests.
- The main boundary conditions of the test procedure had to be checked. Firstly, we had to clarify at a sufficiently high magnification under a stereo microscope if the friction between clamps and specimen was large enough to avoid sliding of the specimen. Secondly, the qualitative two-dimensional strain distribution during the tensile test had to be analysed. For this purpose, a thin paint layer of white and black pigments serving as speckle pattern (Fig. 3a) was sprayed on the specimens by means of an airbrush gun. A CCD camera filmed the specimen surface during the tensile test and a high-resolution strain-mapping software (VIC 2D, Correlated Solutions) calculated displacement fields of speckles.

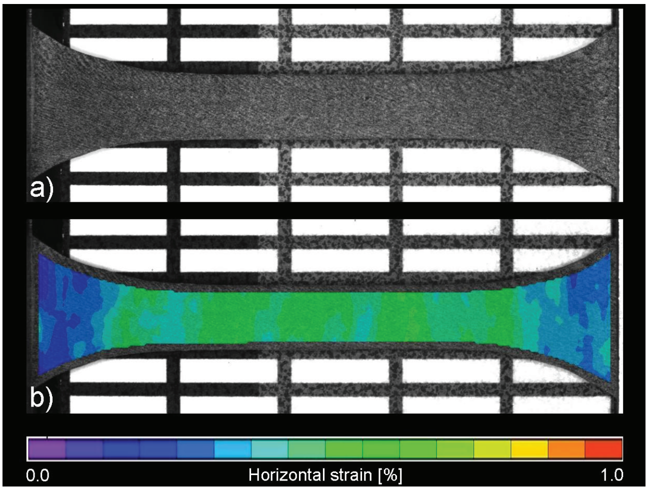

Fig. 3: a) Tensile specimen provided with a speckle pattern; b) axial strain distribution of the same specimen at 30 percent of the maximum load

Main tensile tests

A loading rate of 0.1 mm min^{-1} for yew specimens and of 0.2 mm min^{-1} for spruce specimens was derived from the results of the preliminary tests (i.e. the chosen loading rates resulted in the same total test time for both species). The main tensile tests were carried out within the linear-elastic regime, i.e. after reaching 30 percent of the expected peak stress determined in the preliminary tests, the still intact specimens were unloaded again.

The 41 yew and 40 spruce specimens, that were eventually used for data interpretation, were compiled as follows: 27 specimens from yew stem 1, 14 specimens from yew stem 2, 21 specimens from spruce stem 1 and 19 specimens from spruce stem 2. The number of specimens is non-uniform since the specimen quality (particularly a plane upper and lower surface) was examined subsequent to tensile testing by means of scanning electron microscopy (SEM) images. Specimens not meeting the requirements were rejected afterwards.

Calculation of MOE_L

After the main tensile tests, the exact cross sectional area of the specimens had to be determined as a basis for the calculation of stress-strain curves. For this purpose, a first rough cut through the specimens centre (perpendicular to the grain, Fig. 1e) was made using a razor blade. Then the cut end of one specimen half was embedded in polyethylene glycol (PEG 2000) and thus stabilised for the subsequent precise cutting with a rotary microtome. PEG was dissolved and washed out again and the specimens were dried in an oven. After these essential steps, the exact cross-sectional area was measured in the SEM (Fig. 4) and used as the reference value for stress calculation. MOE_L now could be determined by calculating the slope of the stress-strain curves within the linear-elastic regime.

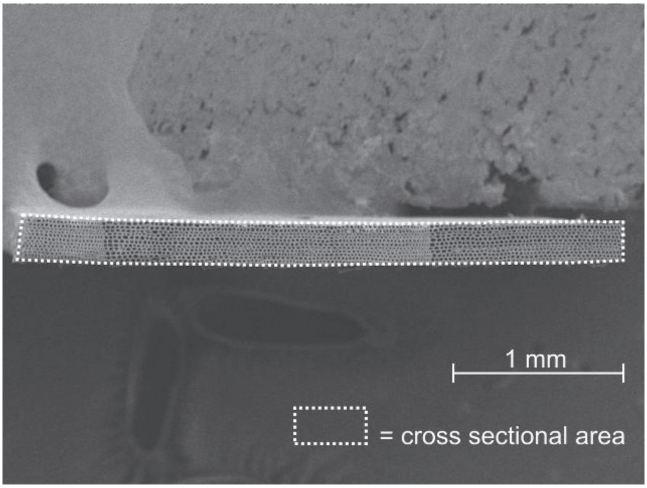

Fig. 4: SEM image: measurement of the cross sectional area of a tensile specimen fixed upright on the sample holder. See also Fig. 1e

Measurement of selected wood-anatomical characteristics

Cell wall area percentage of the cross section: Cell walls appear as bright areas in SEM images, lumens as dark ones. The total area of bright objects within the cross-sectional area of the specimens was measured using image analysis software (Image Pro Plus 4.0, Media Cybernetics). This approach allows for calculating MOE_L on basis of the pure cell wall area, i.e. omitting the lumen area. The cell wall area percentage is the ratio between the total area of bright objects and the cross-sectional area of the specimen.

Density in the cross section: Density was calculated as cell wall area percentage multiplied by the theoretical cell wall density. For the latter we used a value of 1.5 g cm^{-3} which is generally accepted to be more or less in the same range for all wood species.

MFA and tracheid diameter: The microfibril angles of the specimens were measured by means of ray / tracheid cross field pit apertures (Fig. 5). According to the theory, the angle between pit aperture orientation and longitudinal tracheid axis corresponds to the MFA of the secondary cell wall layer S_2. Such measurements are usually done by means of transmission light microscopy. Therefore, we relied upon thin tissue sections. The tensile specimens themselves were too thick for this purpose. Moreover, it was also impossible to cut a thin section out of these specimens since they could not be fixed in a microtome. Therefore, a second slice (20 µm thick) has been cut out of each cuboid (Fig. 1f) and prepared, as described above, for transmission light microscopy. It can be assumed that the anatomical properties of these slices do not differ considerably from those of the tensile specimens.

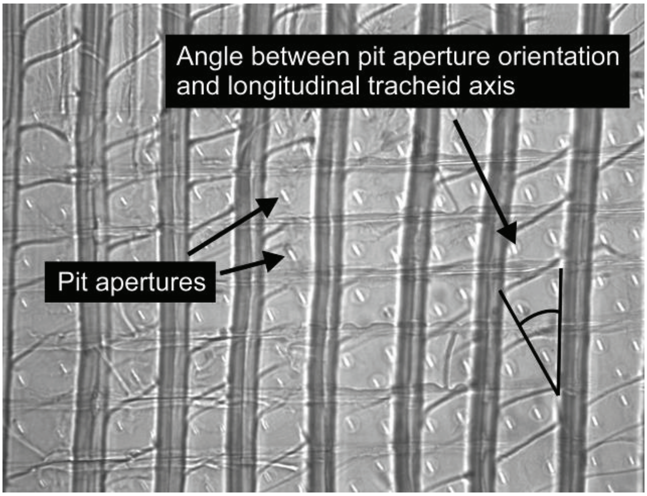

Fig. 5: Radial microtome section of yew wood: principle of measuring pit aperture orientations in the ray / tracheid crossing field

We measured the microfibril angle but also the radial cell diameter of each individual tracheid over the whole radial width of the samples. Our samples complied with the following requirements for the measurement of ray tracheid cross field pit apertures: 1) the slices are radial microtome sections and 2) the crossing field pits are piceoid (spruce) and cupressoid (yew).

RESULTS AND DISCUSSION

Preliminary experimentation

In preliminary tests performed on 20 yew and 20 spruce micro specimens, a tensile strength of about 84 MPa for yew and about 74 MPa for spruce was determined. This is slightly lower than the literature references for solid wood (108 MPa for yew and 80 to 90 MPa for spruce (Sell 1997)). The mean strain at peak load was nearly twice as high for yew specimens (~1.3 percent) compared to spruce (~0.7 percent). In the main tests, tensile loading was stopped at 30 percent of the determined strength values, i.e. at ~25 MPa for yew and at ~22 MPa for spruce. Up to this stress point, linear-elastic response was observed.

High magnification observations with a stereo microscope during tensile tests revealed that no sliding occurred between specimen and clamps. Furthermore, the strain deformation analyses at 30 percent of the peak load revealed that the maximum strain was, as intended, present in the narrow section of the specimens (Fig. 3b). In the area where the contrast marks for videoextensometry would be fixed, a homogeneous strain distribution was observed, which confirmed that the specimen shape was appropriate to determine the MOE.

MOE_L

In the literature, a low MOE_L in relation to its high density is reported for yew at the macro level, usually determined by means of standard test procedures such as 3-point bending. The results of our tensile test reveal a similar relationship for yew at the micro level (Tab. 2):

Tab. 2: Results of tensile tests and structural analyses: Mean values and coefficients of variation. MOE_{CSA} = MOE based on the total cross sectional area, MOE_{CW} = MOE calculated on basis of cell wall cross sectional areas.*

samples	n	cell wall area [%]		density [g cm^{-3}]		MOE_{CSA} [MPa]		MOE_{CW} [MPa]	
yew 1	27	44.3	(5.3)	0.66	(5.3)	7700	(25.6)	17400	(26.6)
yew 2	14	45.7	(5.8)	0.69	(5.8)	6200	(22.2)	13700	(27.1)
spruce 1	21	33.3	(11.3)	0.50	(11.3)	9100	(24.3)	27200	(20.2)
spruce 2	19	34.0	(7.6)	0.51	(7.6)	10700	(18.6)	31600	(17.0)

*Coefficient of variation (%) is given in parentheses.

- the MOE_{CSA} (= MOE based on the total **c**ross **s**ectional **a**rea) of yew is about one third lower than for spruce;
- the MOE_{CW} (= MOE calculated on basis of **c**ell **w**all cross sectional areas) of yew is about half as high as for spruce due to the higher density of yew. Thus, including the density into the calculation of MOE demonstrates even clearer that a fundamental difference must exist between yew and spruce regarding the structure-function relationship.

The calculated yew density (0.66 and 0.69 g cm^{-3}, respectively) is in the range well known from literature whereas spruce density (0.50 and 0.51 g cm^{-3}, respectively) seems slightly increased. Since only few growth rings were covered in the radial width of the specimens, it is possible that the latewood percentage in spruce specimens was higher-than-average.

Our findings indicate that the reason for the low MOE_L of yew must be present at both, the macro and micro level. In a recent study (Keunecke et al. 2007a), we determined the static bending MOE (MOE_B) of yew and spruce by means of a standard 3-point bending test. The MOE_B was higher than the MOE_{CSA} measured in this study. In detail: The MOE_B was 9700 MPa for yew and 12100 MPa for spruce. It has to be taken into account that tensile and bending stiffness generally slightly deviate from each other. A comparison with the MOE_{CSA} (7700 and 6200 MPa for yew; 9100 and 10700 MPa for spruce) suggests a stiffening effect for the bend specimens.

At first view, the stiffening effect seems to be greater for yew specimens. However, this can also be due to the spruce density which was higher for the specimens in this study than for the bend specimens. Furthermore, the boundary conditions were completely different in both test procedures. For example in case of the 3-point bending test, shear stresses are present in addition to uniaxial stresses and may contribute to the stiffening. Since the shear modulus of yew is extremely high in general (G_{LT} of yew is nearly three times as high as for spruce (Keunecke et al. 2007b)), a slightly higher stiffening effect for yew specimens must be expected. The high ray percentage but also the high density contribute to the high shear modulus of yew. In particular, yew earlywood tracheids are characterised by a cell wall / lumen aspect ratio being more than twice as high as for spruce earlywood tracheids (Wagenführ 2000).

One problem inherent to investigations at the micro level is related to the discussion about the representative specimen volume. In standard tests at the macro level, a sufficient homogeneity of the specimens is, for example, ensured by prescribed minimum numbers of annual rings in the loaded cross section. This regulation is not simply transferable to the micro level. As a result, specimens with different numbers of annual ring boundaries (resulting in different latewood percentages), which are therefore not absolutely comparable, have been tested in this study. As a compromise, we calculated the MOE on the basis of the cell wall areas (as described above) in order to consider density variations anyhow. Further legitimate criticism is related to the cellular dimensions: Compared to spruce, yew tracheids are slightly smaller in length and diameter. This means that more yew than spruce tracheids fit into the specimens' cross section. Consequently, both mentioned methodological weaknesses might influence the informative value of the results.

Microfibril angles

In Fig. 6, the measurements of all samples (41 yew and 40 spruce samples) are summarised and illustrated as intra-annual trend. By means of the pit aperture method, we found a clear trend of decreasing microfibril angles from the first earlywood cell to the last latewood cell in all growth rings of both species. The mean spruce MFA decreases from ~43° (earlywood) to ~9° (latewood), the mean yew MFA decreases from ~42° (earlywood) to ~32° (latewood). In other words, the large MFA in yew latewood and the small MFA in spruce latewood turned out to be the most conspicuous difference between both species.

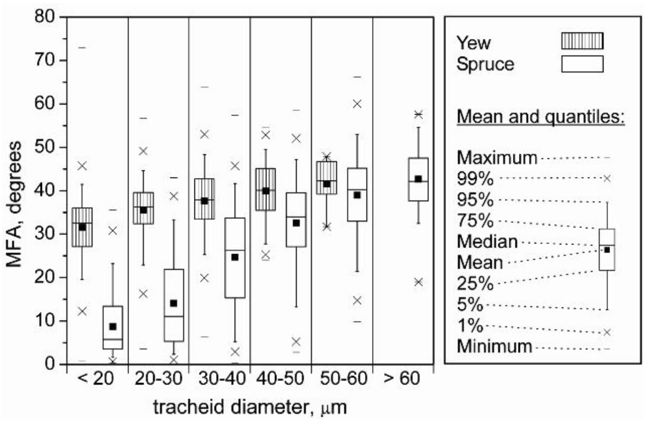

Fig. 6: Relationship between MFA and tracheid diameter based on 41 yew and 40 spruce samples, i.e. on ~4000 tracheids per species. Small tracheids diameters correspond to latewood regions

The spruce results are in good accordance with values measured by Kyrkjeeide (1990) and Herman et al. (1999) who used the pit aperture method as well. However, microfibril angles reported in the literature for spruce earlywood and latewood tracheids are inconsistent. Values often vary depending on the measurement methodology used. Even when a technique such as X-ray diffraction was applied, earlywood MFAs were larger in some studies and latewood MFAs in others. An overview is given by Brändström (2001).

Nowadays, MFA measurement techniques such as X-ray diffraction are widely accepted but only allow for averaging a large number of tracheids sampled at the same time. Thus, it was not an alternative in our case since we were interested in the MFAs of individual cells. In the literature it is still intensely debated what measurement method comes closest to the "true" MFA. Huang et al. (1997) compared commonly used techniques by means of southern pine samples. For latewood tracheids, they found high correlation coefficients between the pit aperture method and other techniques such as polarised light, iodine staining, ultrasonic checking and X-ray diffraction whereas earlywood MFAs were overestimated. This means that - irrespective of the earlywood tracheids - our study at least revealed a clearly larger latewood MFA for yew than for spruce.

Several studies document the great influence of the MFA on axial stiffness. Thus, it is conceivable that the large latewood MFA of yew is responsible for the relatively low MOE_L at the macro and micro level. Keckes et al. (2003) demonstrated that the MFA decreases while applying a tensile force parallel to the cell axis. As a result, a higher microfibril angle raises the strain to fracture (Reiterer et al. 1999). This relationship probably caused the high strain we observed for yew in the preliminary tests of this study and in the case of bend specimens (Keunecke et al. 2007a). Since even the MFA of latewood tracheids seems to be large in the case of yew, the whole tissue is much more stretchable under longitudinal tensile load.

Suitability of specimen preparation and experimental setup

As usual in the case of experimentation at the micro level, no standardized procedure is prescribed. Therefore, in the majority of cases one has to venture into uncharted terrain. In retrospect, the methodology chosen in this study revealed advantages as well as disadvantages, which are listed as the following.

Advantages:

- Due to the specimen size, potential flaws such as knots, grain deviation and resin channels can be avoided.
- Boundary conditions such as specimen geometry and specimen / clamps interaction proved to be suitable to perform tensile tests.
- The small specimens are suitable for strain measurement by means of videoextensometry.
- Precise measurement of the cross sectional area is possible by means of SEM images.
- The calculation of density by means of SEM images and image analysis software seems to work reliably.
- The MFA of individual tracheids can be measured with the pit aperture method.

Disadvantages:

- Without exception, the individual work steps are very time-consuming (specimen preparation, embedding, SEM-images, MFA measurement). Thus, only a relatively small number of specimens can be evaluated which often is too small to draw final conclusions.
- The specimens are not as homogeneous as larger specimens due to a stronger influence of parameters such as cell wall thickness, cell diameter, earlywood-to-latewood ratio, cellulose content, fiber length and grain deviation. This leads to results with a high coefficient of variation.

- The tensile specimens themselves can not be used for MFA measurements. Instead, samples located directly adjacent to the tensile specimens (in the cuboid, Fig. 1) have to be used. Neighbouring tissues may be similar, but they are never identical.
- According to Huang et al. (1997), only the MFA of latewood tracheids measured with the pit aperture method is in good accordance with other commonly accepted methods but earlywood MFAs tend to be overestimated.
- No direct correlation between MFA and MOE_L is possible: Calculating a mean MFA for each specimen would make no sense since earlywood and latewood MFAs would be averaged. The contribution of latewood tracheids to wood stiffness, however, is stronger than that of earlywood tracheids. Moreover, reliable values for the MFA can only be determined for latewood tracheids.

CONCLUSIONS

The present study demonstrates that the low axial stiffness of yew is also present at the micro level. It also shows that density alone cannot be used as an indicator for the longitudinal stiffness of wood. The determined MOE_L, however, only provides a rough approximation since a limited number of specimens were tested. Compared to spruce, a relatively large latewood MFA was determined for yew, which could explain the low MOE_L and the large strain at peak load. No other softwood species with comparable structural design and mechanical behaviour is known to us. The biomechanical function of the low stiffness in the living yew tree, however, remains unclear. A high MFA usually contributes to absorbing mechanical stresses, e.g. in compression wood or in young slender trees where bending loads are caused by wind. In contrast to the latter, the shape of yew trees is rather compact.

The chosen experimental setup demonstrates possibilities of micromechanics but also reveals several difficulties and ultimately is too time-consuming for standardised application. Plenty of specimens had to be sorted out since the specimen quality did not meet the requirements.

In future investigations, the following questions are of particular interest:

- Can the large latewood MFA of yew tracheids be verified by means of further measurement methods and with yew samples from different regional provenances?
- How do wood rays affect the mechanical behaviour under longitudinal load? Do they reinforce the tissue, do they support shear failure at ray / tracheid interfaces or do they potentially have a damping effect?
- How do yew and spruce differ regarding the longitudinal elasticity at the next hierarchical level, namely at the level of single tracheids?

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