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The elastic modulus of spruce wood cell wall material measured by an in situ bending technique

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Abstract Using a novel in situ testing technique, the elastic modulus of wood cell wall material can be determined with great accuracy. The method relies on a focussed ion beam system (FIB) to prepare samples from individual structural components at a length scale which otherwise is hardly, if at all, accessible for testing. To determine the elastic modulus of cell wall material, cantilevers are cut with the FIB from wood cells for beam bending experiments inside the FIB or a scanning electron microscope (SEM). This type of sample preparation is site-specific and, at the same time, minimises the usual sample mounting problems. Once cut, the cantilever is tested by applying a known force with a piezoresistive AFM tip that is mounted on a micromanipulator. The resulting displacement is determined from SEM micrographs taken during the test. The cross-sectional area of the cantilever is determined for a number of positions along its length using the FIB as a cutting tool. Applying this method, we measured the elastic modulus of spruce wood cell wall material to be ~28 GPa.

Introduction

The mechanical properties of single wood cell walls are of interest to materials scientists for two reasons: they determine, to a large extent, the mechanical performance of the hierarchically structured wood and that of wood-based composites, which are increasingly made from individual hard- and softwood fibres. Despite the long and sophisticated use of wood in applications ranging from construction to musical instruments and sports equipment, the correlation of structure and mechanical properties at the scale of individual cells is not yet fully understood and currently a very active area of research.

Softwoods, such as spruce (*Picea* sp.), are composed of cells, termed tracheids or fibres. The tracheids have two functions: they transport nutrients and provide structural support for the tree. Tracheids are long hollow cells composed of up to 50% largely crystalline cellulose fibrils wound in a spiral fashion around the longitudinal cell axis and embedded in a matrix of hemicellulose and lignin. Typically, their length ranges from 2 mm to 4 mm, their diameter from 20 μm to 40 μm and their thickness from 2 μm to 10 μm. The cell wall consists of four layers: the primary wall and the three secondary cell wall layers S1, S2 and S3. The S2 layer is the thickest and forms up to 80% of the entire cell wall. This and the small angle between the cellulose fibrils and the long axis of the cell make the S2 layer the mechanically most important. The tracheids are joined by a middle lamella and give softwoods their characteristic cellular structure [1].

So far, the elastic modulus of wood cell wall material has been determined from tensile tests of whole wood fibres, which were isolated either chemically—using hydrogen peroxide and glacial acetic acid, for example—or mechanically—using fine tweezers for peeling [2–5]. Both routes of sample preparations have disadvantages, since the chemical treatment degrades both cell wall constituents and structure, and the mechanical

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method leads to a separation of cell wall layers [6]. Alternative measurements of the elastic modulus of wood fibres were made by nanoindentation [7, 8]. However, this technique yields average property values for this orthotropic material rather than properties in well defined directions.

Our new in situ bending technique overcomes the above problems and the additional very critical one, that of the accurate measurement of the cross-sectional area of the sample. Another advantage of the method is that samples can be prepared site-specifically and with great precision inside the FIB microscope, as we will demonstrate below.

Sample preparation

The samples for in situ bend tests were prepared by hand and using the FIB (FEI 200 xP, FEI Company [9]). First a small wedge was cut from a larger piece of spruce wood parallel to the fibre direction, with a razor blade, to expose individual tracheids (Fig. 1a). Then the wedge was coated with a thin layer of carbon to avoid charging effects during sample preparation in the FIB and testing in the SEM. To ease sample handling, the carbon coated sample was clamped into a custom-made mini-vice, which has stubs on neighbouring sides so that it can be mounted on FIB and SEM sample trays in two positions perpendicular to one another. This not only eases the preparation of samples with a rectangular cross-section but also means that the sample can be tilted from a 0° position in the FIB to a 90° position for testing in the SEM without removing it from the holder.

At the tip of the wedge, where individual tracheids were exposed, cantilever-shaped samples with a near rectangular cross-section were micromachined using the FIB as illustrated in Figs. 1b–d. The depicted cantilever is 353.0 μm long, 17.39 μm wide and 3.47 μm thick. The sample stiffness was always more that two magnitudes smaller than that of the AFM cantilever, to ensure that the deflection of the AFM cantilever during loading is negligible and linearity in the force measurements is preserved.

Sample testing

The bend tests were carried out using the newly developed device shown in Fig. 2a inside an SEM (Leo 1530 VP [10]) for high resolution and elimination of ion beam damage. It consists of a piezoresistive AFM tip (Nascatec GmbH [11]) mounted on a three-axis micromanipulator MM3A (Kleindiek Nanotechnik GmbH [12]) and is described in detail elsewhere [13]. The design of this force measuring device, especially its small size and weight (~65 g) and the fact that it has neither drift nor backlash on reversal, makes it an

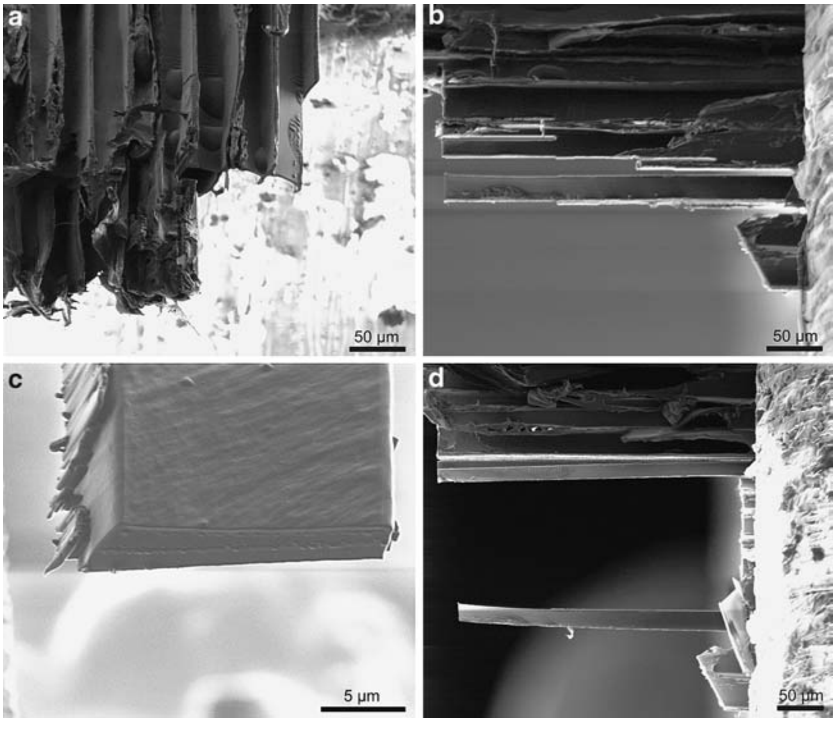

Fig. 1 (**a**) Spruce wood wedge cut with a razor blade. (**b**) FIB machining of a spruce wood cell wall cantilever. (**c**) A FIB cross-section of a wood cell wall cantilever. (**d**) A wood cell wall cantilever ready for testing

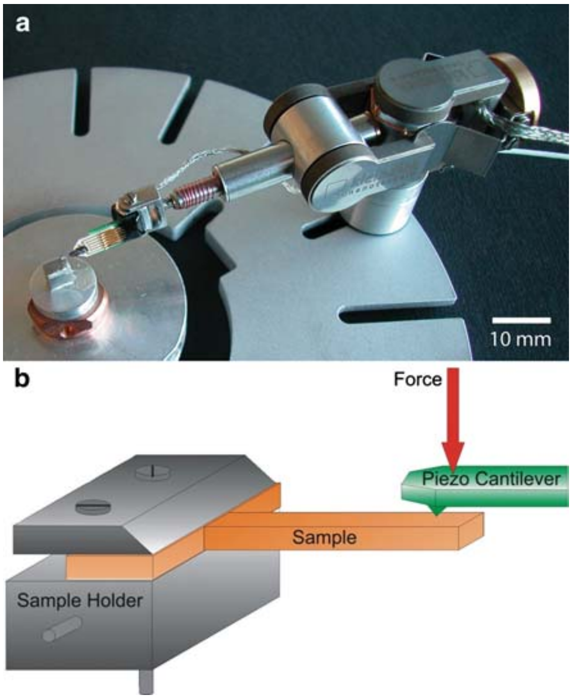

Fig. 2 (**a**) The testing device. (**b**) Schematic of the bending test method

ideal tool for use inside SEM and FIB microscopes. The tip used for the experiments described below had a nominal stiffness of 270 N/m and its force resolution was limited by noise to 15 μN to 20 μN. The micromanipulator is driven by piezoelectric motors and has one longitudinal and two rotational axes, which can be operated in five gears. In the smallest gear, the smallest step size of the rotational axes is 5 nm at a fully retracted and 6 nm at a fully extended longitudinal axis, and the maximum displacement is 4,096 steps. The micromanipulator is used to bring the AFM tip into contact with the sample and to apply a defined load. See Fig. 2b for a schematic of the bending test method.

The elastic modulus of the spruce wood cell wall material was determined for several positions along the length of the cantilever with the micromanipulator driven in its smallest gear and positioned so that the micromanipulator could be moved through the whole range of its displacement. The load was applied and increased by moving the AFM tip incrementally and a defined number of fine steps (ranging from 1 to 128) towards the sample. Once a new load was applied, the mean force, its standard deviation, as well as the minimum and the maximum forces were recorded (sampling rate: 500/s) with a BIOPAC data acquisition and analysis system (Model MP100A-CE, BIOPAC Systems, Inc. [14]. For each load, an SEM micrograph was recorded and stored to determine the displacement. Unloading curves were determined with the same number of measurements. This procedure is automated and controlled by AutoIt software (v.3.0.102) [15] macros.

While direct force measurements were possible with the AFM cantilever, the displacement could not be measured with sufficient accuracy by this device due to a hysteresis in both the piezoelectric motors of the micromanipulator and the piezoelectric AFM cantilever. The displacement was therefore determined directly by automated image analysis of the micrographs taken for each load during the test, using Coral DRAW10 [16]. After the test, the width and the height of the sample were determined from FIB cross-sections for a number of positions along the cantilever. The beam in Fig. 1c is 17.39 ± 0.35 μm wide and 3.47 ± 0.07 μm thick.

Results and discussion

Two typical force-displacement curves for bending tests on spruce cell wall cantilevers are shown in Fig. 3. They reveal initial linear-elastic behaviour for loading and unloading. The small hysteresis in the measurements is due to mechanical losses in the sample material.

The elastic modulus of the material was determined using standard beam theory, assuming a uniform rectangular cross-section

$$E = 4 \cdot \frac{F}{\delta} \cdot \frac{l^3}{wh^3} \tag{1}$$

where F is the applied load, δ the deflection, l the length of the cantilever, w its width and h its thickness, and F/δ is the slope of the load-displacement curve in the linear elastic region of both the loading and unloading curve. Table 1 lists the elastic moduli determined from the loading and unloading curves for five different positions along a spruce cell wall cantilever. Their mean values are 29.9 ± 2.3 GPa and 26.0 ± 2.3 GPa for the loading and unloading curves respectively (Table 2).

To be able to compare these values with those from the literature, we have to take the moisture content of the samples into account which influences the mechanical properties of the wood cell wall material significantly. According to Skaar [17], the Young's modulus at a moisture content of 12% and that at a moisture content of M% are correlated as:

$$E_M = E_{12\%} \exp[B(12 - M)] \tag{2}$$

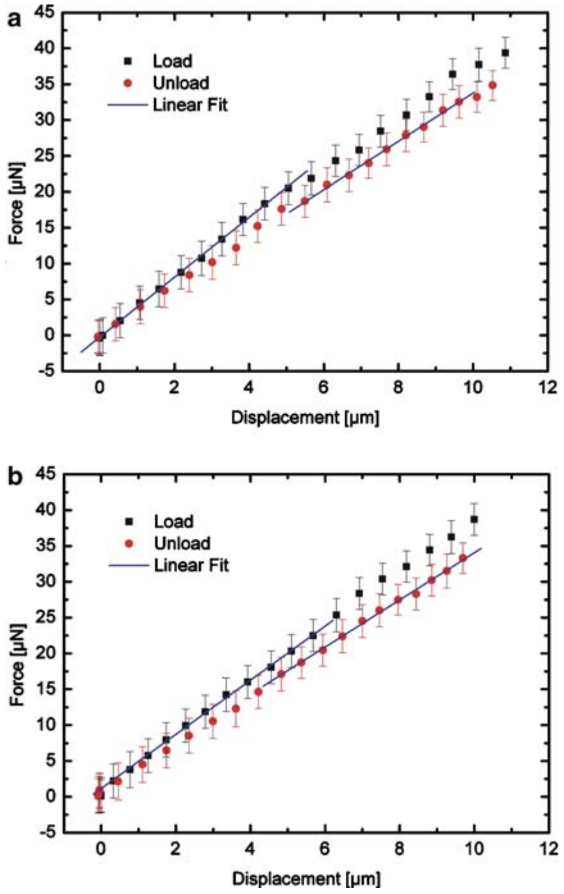

Fig. 3 Force versus displacement curves for two bending tests on spruce wood cell wall cantilevers. The black lines are linear regressions to determine the elastic modulus

Table 1 Elastic modulus measured during loading and unloading at five positions along a spruce cell wall cantilever

Cantilever length [μm]	Young's modulus Loading [GPa]	Young's modulus Unloading [GPa]
134.1 ± 2.7	31.7 ± 5.4	31.4 ± 9.1
122.8 ± 2.5	29.0 ± 3.9	24.3 ± 5.1
114.0 ± 2.3	32.0 ± 5.7	26.8 ± 4.9
109.8 ± 2.2	30.5 ± 3.9	24.6 ± 4.6
108.1 ± 2.2	26.4 ± 3.7	23.0 ± 3.7
Mean	29.9 ± 2.3	26.0 ± 3.3

With $B = 0.02$ and $\mathrm{M} = 0$, the elastic modulus for a moisture content of 12% can be calculated from the tests in vacuum at a moisture content of 0% with following equation:

$$E_{12\%} = \frac{E_M}{\exp(0.24)} = \frac{E_M}{1.27} \qquad (3)$$

to be 23.5 GPa and 20.5 GPa for loading and unloading, respectively. These values agree well with those reported by [4, 5], measured on mechanically isolated spruce fibres tested in tension.

An interesting observation made during sample preparation was that the wood cell wall cantilever started to twist once it was cut free in the FIB. Burgert et al. [18] report similar behaviour. This twisting of the cantilever is probably due to an anisotropic shrinkage of the cell wall material and reflects the helical arrangement of the cellulose microfibrils in the S2 layer.

Conclusions

Applying a novel in situ testing technique for use in SEM and FIB microscopes, the elastic modulus of vacuum dry spruce (*Picea* sp.) wood cell wall material was determined to be ~28 GPa. One considerable advantage of the testing of samples in vacuum is that it provides a well-defined reference state that can quickly and, more importantly, reproducibly be achieved, a fact which is critical for biological materials, for which our new technique was developed. By contrast, the frequently reported 'natural' or 'wet' state is less well, if at all, defined and hence not always very suitable for material comparisons. A further advantage of this technique is that samples can be custom-made in size, as illustrated here on the manufacture and testing of microscopic wood cell wall cantilevers. Finally, the technique is remarkably versatile and neither restricted to bend testing nor to the use inside a FIB. We have applied it with similar success to the tension and compression of samples inside SEM and FIB microscopes [13] and have found it well suited for use in an environmental scanning electron microscope (ESEM) and ex situ in a light microscope.

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Table 2 Measured mechanical properties of the spruce wood cell wall sample and literature values

Sample	Mode of testing	Young's modulus [GPa] Air-dry	Reference
Wood fibre (Spruce, *Picea abies*)	Tension (Air-dry)	12–31	[4, 5]
S2 layer of wood fibre (Spruce, *Picea* sp.)	Nanoindentation (Air-dry)	13.5–21.3	[7, 8]
Cell wall material cantilever (Spruce, *Picea* sp.)	In situ bending (Vacuum-dry)	29.9 ± 2.3 (Loading) 26.0 ± 3.3 (Unloading)	This study

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