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Contribution of cellulose to the moisture-dependent elastic behaviour of wood

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ABSTRACT

Wood has a hierarchical structure involving several levels of organisation. The stiffness of wood relies on its capacity to transfer mechanical stress to its stiffest element at the lowest scale, namely crystalline cellulose. This study aims at quantifying to what extend crystalline cellulose contributes to wood stiffness depending on its moisture content. The crystal strains of cellulose were measured using X-ray diffraction on wet and dry specimens of spruce, based on a previously published methodology. The comparison between crystal strain and macroscopic strain shows that, during elastic loading, cellulose strain is lower than macroscopic strain. The means ratio of crystal/macroscopic strain amounts 0.85 for dry specimens and 0.64 for wet specimens. This strain ratio cannot be explained just by the projection effect due to the difference in orientation between cellulose microfibrils and cell wall, but results from deformation mechanisms in series with cellulose. Analysis shows that this series contribution represents a non-negligible contribution to wood compliance and is strongly moisture-dependent. This contribution amounts 9% for dry specimens and 33% for wet specimens, corresponding to a 4-fold increase in compliance for the series contribution. The origin of these strains is ascribed to mechanisms involving bending or shear strain at different scales, due to the fact that reinforcing element are neither perfectly straight nor infinitely long.

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1. Introduction

Wood has a hierarchical structure, with several levels of organisation. Wood tissue at the sub-millimetric scale is made of different kinds of cells, namely fibres (or tracheids) and rays, and sometimes vessels and axial parenchyma. Among these cells, fibres are from far the most abundant and stiff. Fibres are long hollow cells, with cell walls glued to adjacent cell walls, that form a honeycomb structure. The cell wall is made of several concentric layers, and forms a multilayer structure. Each wall layer is a fibre composite layout where the matrix phase is a mixture of lignin and hemicellulose, and the fibres (called microfibrils) are made of cellulose. The cellulose microfibrils are arranged helically in the cell, with a characteristic angle for each layer, termed microfibril angle. Cellulose is present in a partly crystalline form, where amorphous cellulose is organised in series and in parallel with cellulose crystallites. Crystalline cellulose is from far the stiffest constituent of wood. Its extraordinary large stiffness (approx. 135 GPa [1–3]) provides the good properties of wood in the axial direction.

The mechanical behaviour of wood depends on the organisation of the material at all levels. The link between structure and properties is therefore the key to predict the variability of the wood material and understand its origin. Structure and properties can be linked through multiscale modelling. The principle of this modelling is using hierarchical models, where each model predicts the behaviour at one scale, based on the structure and behaviour at lower scales. Such schemes have been developed for wood to model different kinds of behaviour, such as drying shrinkage, elastic and viscoelastic behaviours [4,5]. Another key element of the models is the assumption used for the kinematics of elements, i.e. how they deform relatively to each other. Most of the time, these assumptions are based on an idealization of wood structure [5] and are particularly difficult to assess.

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In material science, different methods are used to evaluate the stress transfer between the fibre and the matrix phase in fibre-reinforced composites. These methods are based on the measurement of strain at the molecular scale, using Raman spectroscopy, infrared spectroscopy or X-ray diffraction. They have been used to assess the performance and characterize the kinematics of composite materials [3,6].

X-ray diffraction methods have been used for wood to evaluate how the cellulose crystal strains in response to different loading, such as elastic [7–12], viscoelastic [13,14] or mechanosorptive [13] strains, hygro-thermal treatments [15–17], chemical treatments [17], drying [17,18], stress release [17,18] or stress induced by biological transformations [19]. Montero et al. [12] probed the behaviour of crystalline cellulose during elastic deformations of poplar wood. The results showed that the deformation of crystalline cellulose is lower than the macroscopic wood strain, and that this difference could not be explained by the projection effect of microfibril angle alone, but originated in one or more elements associated in series with crystalline cellulose. Hypotheses for the localization of these contributions are related to the "non-ideal" nature of wood structures, where elements at different scales may not be completely straight and have a finite length, leading to bending or shear-lag effects for which strain happen without straining the cellulose crystal. Understanding the origin and behaviour of these localized strain is essential for understanding how the wood structure ensures a good transmission of mechanical stress from the macroscopic scale to its stiffest constituent at a molecular scale. Here we aim at analysing the moisture-dependant behaviour of wood. The ratio between crystal and macroscopic strain is measured during elastic loading using an improved methodology based on Montero et al. [12], and this ratio is compared between wood in wet and dry states, to probe the contribution of cellulose to wood strain and its change with moisture content.

2. Material and methods

2.1. Wood material and macroscopic measurements

Experiments were performed on wood specimens taken in the same area of a board of European spruce (*Picea abies*). Wood was first air-dried, then earlywood specimens of 50 × 3.2 × 1.8 mm (L × T × R) were cut far enough of the pith to neglect the annual ring curvature, and consider the R and T directions as uniform over the specimen. Specimens were polished with a precision motorized sander to make sure that their dimensions were uniform and that surfaces were parallel. The basic density of the specimens was 364 kg/m^3 (±16 S.D.). The mean microfibril angle (MFA) of each specimen was measured by X-ray diffraction following the methodology described by Cave [20]. A total of 18 specimens with similar MFA (11.8° ±0.6 S.D.) were selected for this experiment. Nine of them were stabilized at ambient air-dry conditions (approx. 40% RH), and will hereafter be referred to as "dry specimens". Their average moisture content was 6.0% (±0.4 S.D.). The other 9 specimens were water-saturated by immersion in water in a chamber submitted to vacuum cycles during 7 days, and will hereafter be referred to as "wet specimens". The modulus of elasticity (MOE) of the specimens was measured using a dedicated 4 point bending apparatus placed into a conventional testing machine. The specific modulus (E/d) was computed as the ratio of MOE to basic density.

2.2. Bending set-up and measurement of macroscopic strain

The specimens were used to perform *in situ* four-point bending tests while measuring axial lattice distance of crystalline cellulose. The set-up used is similar to that used by Montero et al. [12].

The bending device is illustrated in Fig. 1. Wood macroscopic strains were measured with 5 mm-long strain gages (Kyowa KFG-5-120-C1-11-L1M3R) glued with cyanoacrylate, in the middle of the top and bottom LxR surfaces of the specimen. The gages were connected to a data-logger (Vishay Model P3) recording the strain level. Four small pieces of steel were pasted on the upper and lower surfaces at the position of the loading points (Fig. 1) to minimize the effect of indentation of cylinders on wood during the test. A screw on the top of the bending device enabled the control of the displacement of the upper spans in the T direction, while the lower spans were fixed. The displacement was increased with the screw, until the desired strain level (average of the two strain gages = 0.15% and 0.24% for wet and dry specimens respectively) was reached. Temperature and relative humidity (RH) in the testing chamber were continuously measured during the tests, and ranged between 35 and 45% RH and 24–26 °C.

2.3. X-ray diffraction set-up for measuring cellulose strain

The change in lattice distance of crystalline cellulose was calculated from the shift of the 004 cellulose crystal plane during bending. The bending device was placed in an X-ray goniometer (Agilent Technologies Gemini S), using a CuKα source (wavelength $\lambda = 0.154$ nm, energy E = 8.05 keV) monitored at 40 kV and 40 mA. The spot size was 800 μm in diameter. In order to optimise the intensity of the $(004)_{cellulose}$ diffraction signal, the specimen was rotated at an angle $\theta = 17°$ to the X-rays (corresponding to the Bragg angle of the cellulose 004 plane with this wavelength), and the detector at an angle $2\theta = 34°$ (Fig. 2). Also, to maximize the resolution of the recorded pattern, the CCD detector (2048 × 2048; 31μm/pixel; bin1) was placed at a distance D = 135 mm of the specimen (Fig. 2), acquisition focusing on the 004 cellulose peak.

Gold powder (with particles of 5 μm diameter) was placed on the back LxT surface of the specimen, to calibrate the distance between the specimen surface and the CCD detector, by detection of the angle of the (111) reflection of gold on each image. Dry specimens were exposed to X-rays during 60 s. Wet specimens, yielding lower signal intensity due to water absorption, were exposed during 90 s. To avoid any drying during the test of wet specimens, the bending device was enveloped in a cellophane film with water-saturated absorbent paper, ensuring a water-saturated atmosphere surrounding the specimen.

The specimens were scanned at 30 positions along the Z-axis (Fig. 2) from top to bottom, by moving up the device between two scans. The Z-position of the bending device was measured using a displacement sensor (Mitutoyo Absolute Digimatic Indicator; 1 μm accuracy). Steps of 100 μm were used for the 10 first scans, then 200 μm for the 10 following scans, and then 100 μm for the 10 last scans. This procedure was chosen to maximize the precision near the upper and lower surfaces of the specimen, where the strain is the largest. Given the size of the steps and that of the spot, successive probed wood areas partly overlap. At each step, the scan produced a pattern of X-ray diffraction including reflection peaks of cellulose $(004)_{cellulose}$ and gold $(111)_{gold}$ as illustrated on Fig. 2.

2.4. Signal integration and processing

Each pattern was integrated in the azimuthal direction (from $\varphi = -20°$ to $\varphi = +20°$ and from $2\theta = 20°–24°$), yielding a one-dimensional signal expressing intensity variations as a function of the diffraction angle θ. The diffraction angle is related to the lattice distance via Bragg's law:

$$d = n\lambda/(2\sin\theta) \quad (1)$$

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