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ORIGINAL ARTICLE

Sensitivity of bamboo fiber longitudinal tensile properties to moisture content variation under the fiber saturation point

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Abstract The mechanical response of bamboo fibers to variation in moisture content (MC) has a direct influence on the performance of bamboo fiber-based products, both during their processing and final practical applications. However, due to the experimental difficulties involved in testing, this fundamental process remains poorly understood. In this paper, longitudinal tensile modulus (TE), ultimate tensile strength (UTS) and elongation at break (EB) for four different MC levels, ranging from approximately 4.97 to 26.2 %, were determined for single bamboo fibers aged 0.5, 1.5 and 2.5 years old, respectively. For each MC level, the results show that both TE and UTS of bamboo fibers vary little with age. A general linear reduction is observed for both TE and UTS when MC increases, while EB shows a slight increase. Furthermore, TE is found to be most sensitive to MC change, followed by UTS and then EB. A close examination revealed that 2.5-year-old bamboo fibers are more sensitive to MC change than younger specimens, which may partly be related to their relatively higher microfibrillar angel (MFA). A direct TE–MC plot comparison between bamboo fibers and solid bamboo not only demonstrates the decisive role of the fiber component in the overall mechanical response to MC of bamboo, but also reveals that the TE of the former is less sensitive to varying MC levels than the latter.

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Keywords Bamboo fiber · Mechanical properties · Moisture content · Sensitivity

Introduction

Bamboo fibers are renewable, abundantly available, biodegradable, combustible and have minimal environmental impact over the course of their whole life cycle [1]. These advantages explain why there has been a rapid expansion in global research on bamboo fiber-based composites in recent years [2–5]. To enhance developments in composite research, there is still a great need to measure and accumulate data on the basic mechanical properties of bamboo fibers. While several studies have been performed in this area, most of them focus on mechanical variation at either the bundle or single-fiber scale, with respect to ages or locations in the bamboo culms [6–9]. Recently, Yu et al. [9] also measured the mechanical properties of single bamboo fibers from eleven bamboo species at greater levels of statistical significance. These results suggest that bamboo fibers are significantly stronger and stiffer than most softwood fibers, as well as have smaller diameters and larger aspect ratio than the latter. The research highlights the potential of bamboo fibers to act as the reinforcing phase in polymer composites used for structural purposes. Although these findings provide invaluable information for efficiently selecting and utilizing bamboo fibers for the production of various composites, the influence of moisture content on mechanical properties is still poorly understood.

Due to their hydroscopic nature, bamboo fibers tend to change their mechanical properties according to the moisture content (MC). This feature cannot be ignored when one designs processing and utilization applications for bamboo fiber-based composites. The cell walls of bamboo fibers can be analogous to nanofiber-reinforced polymer composites in which the stiff and less moisture-sensitive microfibrils are helically arranged in as of more moisture-sensitive matrix of hemicellulose and lignin [10]. Furthermore, MC variation has the potential to significantly interfere with bonding between the reinforcing and matrix phases in bamboo fiber cell walls. To gain better insight into this fundamental relationship between the mechanical properties of bamboo fibers and MC, mechanical testing should be conducted at the single-fiber level. However, traditionally, due to the experimental difficulties involved in testing single fibers, very few studies have been undertaken.

However, in recent years barriers to conducting tests at the single-fiber level have been mainly overcome with the development of a new technique developed by the authors. Using this technique, Yu et al. [11] were able to measure the effect of MC on the mechanical properties of single Moso bamboo fibers of 4.5 years of age. They found that although an anticipated reduction in longitudinal tensile modulus (TE) and ultimate tensile strength (UTS) of bamboo fibers was clearly observed with rising MC, TE showed much less sensitivity to MC than UTS. Despite these initial findings, more experiments are needed to confirm whether this finding is applicable as a general rule. In this paper, we use our newly developed micro-tensile technique to extend studies on the effect of MC on single Moso bamboo fibers for fibers aged 0.5, 1.5 and 2.5 years. The study was conducted for MC ranging from 4.97 to about 26.2 %. Meanwhile, the microfibrillar angle (MFA) of these fibers were also tested and integrated into the discussion of the experimental results.

Experimental

Raw materials

Moso bamboos [*Phyllostachys edulis* (Carr.) H.de Lehaie] of approximately 0.5, 1.5, and 2.5 years of age were taken from a bamboo plantation located in Zhejiang Province, China.

MFA measurement

MFA refers to the angle between the cellulose orientation in the dominant layer of the cell wall and fiber axis, which is widely known to have significant effects on most physical and mechanical properties of bamboo fibers [9, 12]. An X-ray diffractometer (X'pert Pro, Panalytical, Netherlands) was used to determine the MFA of bamboo slices cut from the mid-height (approximately 4 m, Fig. 1a) of air-dried bamboo culms (Fig. 1b). The samples were cut to the dimensions of 1 mm (Radial) × 10 mm (Tangential) × 30 mm (Longitudinal) (Fig. 1c). A point-focused X-ray beam was applied to the tangential section with a scanning angle range of 0°–360°. From the obtained diffraction intensity curves, the MFA of the bamboo fibers was determined using the 0.6 T methods [13, 14].

Sample preparation

Small bamboo sticks (Fig. 1d) sized 1 mm (R) × 1 mm (T) × 30 mm (L) were split from the aforementioned samples to measure MFA. These sticks were macerated in a soft solution composed of one part 30 % hydrogen peroxide, five parts glacial acetic acid and four parts distilled water for 18 h at 60 °C. Subsequently, the macerated fibers were washed several times in distilled water and dried on a glass slide at room temperature. The fibers with minimal damage were carefully selected under a microscope and placed across a gap (width of roughly 1.8 mm) in an organic glass panel. Two epoxy droplets of approximately 200 μm in diameter were then placed at the ends of each fiber with an approximate spacing of 0.7–0.8 mm. The epoxy was allowed to solidify at 60 °C for 24 h followed by further conditioning for 24 h at room temperature as a further balance (Fig. 1e). More details about sample preparation can be found in Yu et al. [11]. All the samples were randomly divided into four groups for moisture conditioning in a range from 4.97 % MC to approximately 26.2 %.

Micro-tensile test

A small commercial high-resolution mechanical tester (Instron Microtester5848, USA) combined with a custom-built fiber gripping system was used to perform tensile tests on single bamboo fibers, which are often as short as 2 mm. The load cell used has a capacity of 5 N. A pre-tensioned force of roughly 10 mN was exerted on the sample and the tensile speed used was 0.048 mm/min. Elongation was recorded from the crosshead movement with a resolution of 0.08 μm. More than 50 fibers were tested for each group. For the calculations of cell wall mechanical properties, the cell wall areas of every broken fiber were determined with a confocal scanning laser microscope (Meta 510 CSLM, Zeiss, Germany). The broken fibers were first immersed in 0.1 % acridine orange solution for 20 s and then rinsed in distilled water several times. The fibers were then imaged with a 63× immersion oil objective. The load–elongation curves were then converted to stress–strain curves, with TE and UTS obtained based on the cell wall area and the initial span length.

Fig. 1 Flow diagram showing the process of fiber sample preparation

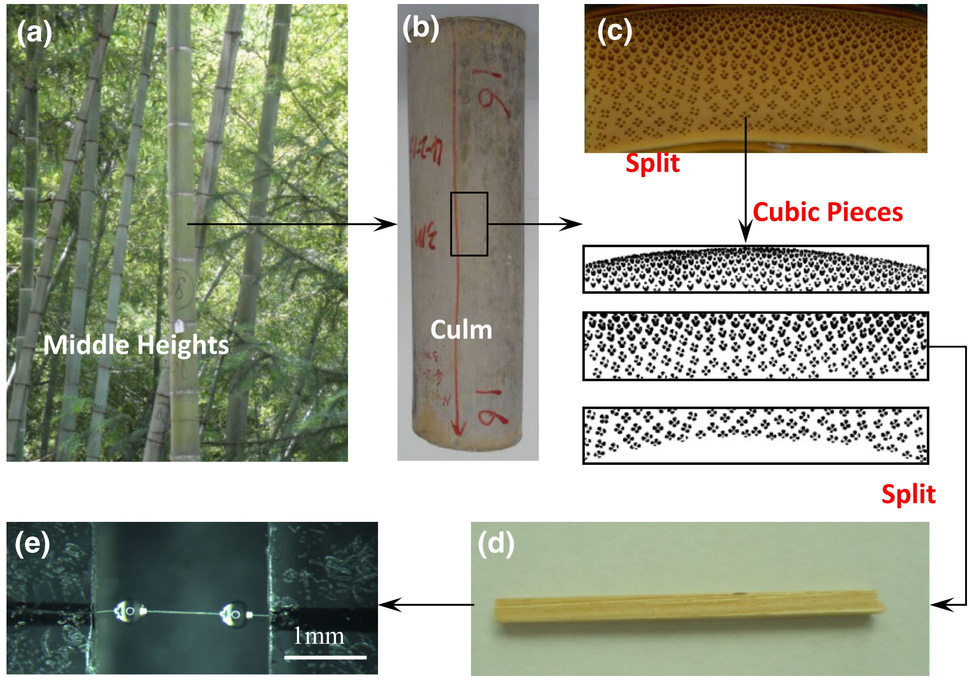
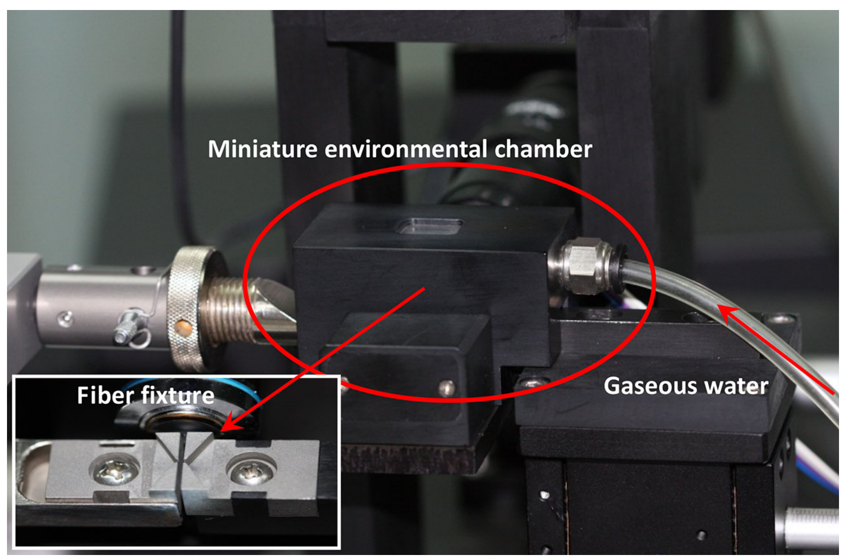


Fig. 2 The custom-built miniature environmental chamber for fiber testing under variable relative humidity



MC control of the tested bamboo fibers

To investigate the effect of MC on the mechanical properties of bamboo fibers, a custom-built miniature environmental chamber was developed (Fig. 2). The relative humidity (RH) in the chamber can be adjusted from 15 to 95 % by changing the speed of gaseous water going into and out of the chamber. In the present study, the RH in the chamber was, respectively, set at 11, 33, 64 and 90.8 %, which, based on the hydroscopic adsorption isotherm of chemically extracted bamboo fibers developed in our laboratory (Fig. 3), corresponded to the respective equilibrium moisture contents (EMC) of 4.97, 8.58, 11.1 and 26.2 %.

Results and discussion

Stress–strain curves of single bamboo fibers with different ages and MC

Figure 4a shows the typical stress–strain curves of single bamboo fibers taken from differently aged samples at a constant MC level of 8.58 %. All the tested fibers exhibited

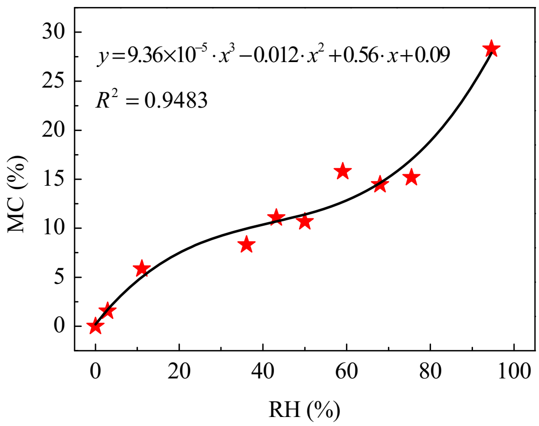


Fig. 3 The moisture sorption isotherms of bamboo fibers. *MC* moisture content, *RH* relative humidity

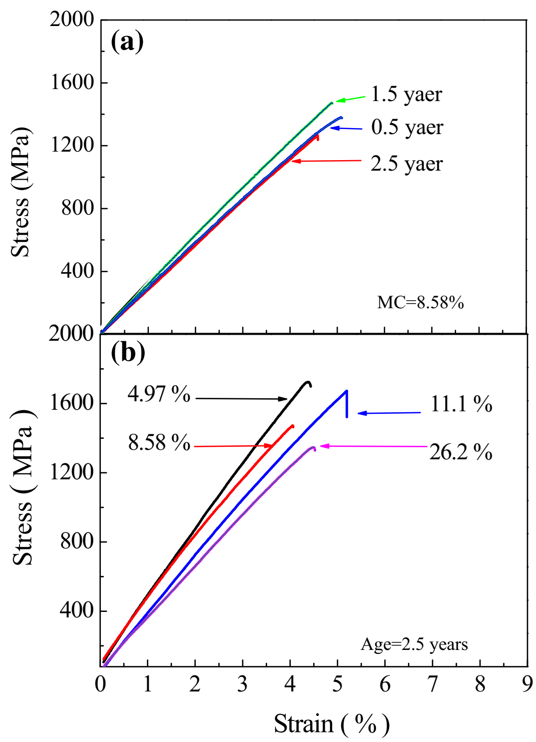


Fig. 4 The typical stress–strain curves of single bamboo fibers with different ages (**a**) and MC (**b**) tensioned in axial direction

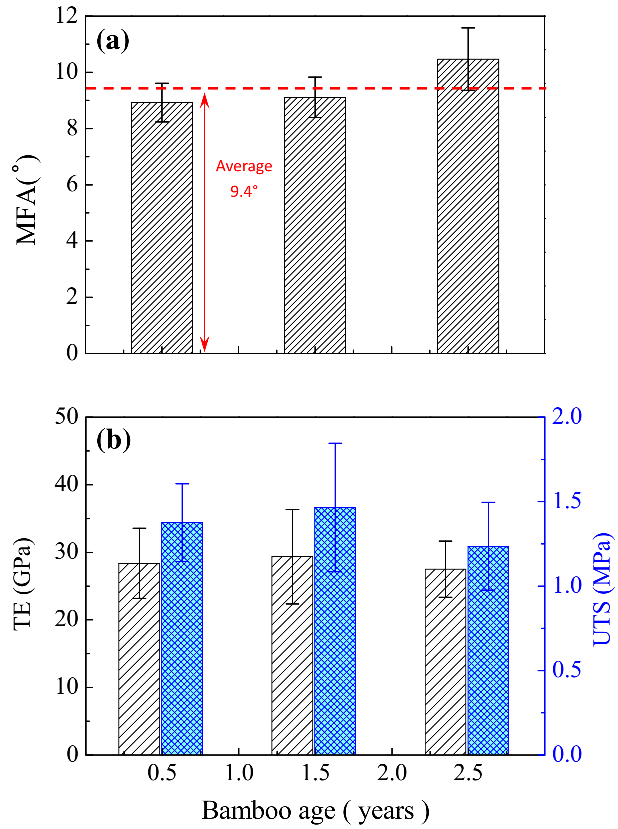


Fig. 5 Microfibril angle and mechanical properties of single bamboo fibers. **a** MFA of Moso bamboo; **b** histogram of TE and UTS of single bamboo fibers. *MFA* microfibrillar angle, *TE* longitudinal tensile modulus, *UTS* ultimate tensile strength

a quasi-linear stress–strain behavior to failure, which has been repeatedly observed in our previous studies [7, 9, 11, 15, 16] According to Köhler [17] and Groom et al. [18, 19], the shape of the tensile stress–strain curve of plant fibers is proposed to be to a large extent dependent on its MFA, with a linear relationship being the dominant outcome provided that the MFA of the fibers is less than 20°. Since the MFA of Moso bamboo in this research ranges from 8.2° to 11.7°, with an average of 9.4° (Fig. 5a); this explains why all the fibers display a quasi-linear stress–strain behavior. Figure 4b presents the typical stress–strain curves of 2.5-year-old single bamboo fibers under different MC. While increasing MC reduces the slope of the curve, there is no change in the linear nature of the results, indicating that MC has little effect on the shape of the tensile stress–strain curves of bamboo fibers.

Longitudinal tensile properties of single bamboo fibers with different ages

The effect of bamboo age on TE and UTS under 8.58 % MC is presented in Fig. 5b. Generally, age has little influence on both the TE and UTS of bamboo fibers. The average TE and UTS of the fibers across the three different age samples ranged from 28.89 to 31.35 GPa and 1.36 to 1.47 GPa, respectively. The mechanical properties of bamboo fibers aged 2.5 years were found to be slightly smaller than those of the other two groups, especially in modulus, which agrees well with the MFA measurement presented in Fig. 5a. The average MFA of 2.5-year-old fibers, 10.47°, was slightly higher than that of the 0.5 and 1.5-year-old bamboo fibers. Similar variation related to age was also observed by Huang et al. [6], who measured the tensile mechanical properties of Moso bamboo fibers in specimens ranging from 0.5 to 8 years of age. MFA has been shown to account for 87 % variation of TE in wood. When measured from the longitudinal axis, an MFA of 10° can increase TE by a factor of 2.5 compared to an MFA of 14° [20]. However, the hemicelluloses–lignin matrix also contributes significantly to the mechanical properties of the cell wall at larger MFA values, especially in the transverse direction [21].

The effect of MC on the longitudinal mechanical properties of single bamboo fibers

The correlations among TE, UTS and elongation at break (EB) of single bamboo fiber with MC are shown in Fig. 6. A general linear reduction of TE and UTS with increasing

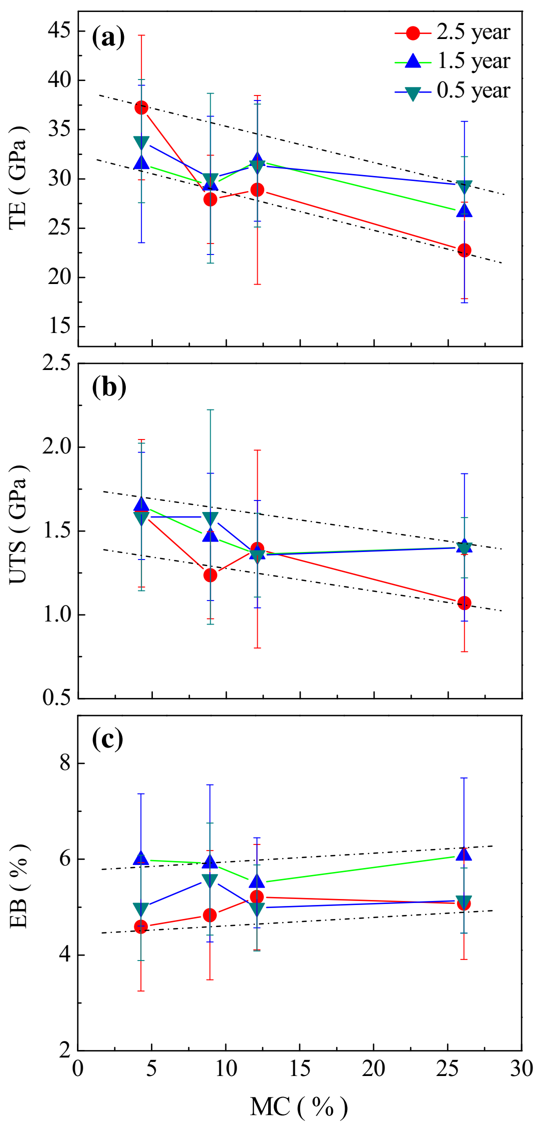


Fig. 6 The influence of MC on longitudinal tensile properties of single bamboo fibers. *TE* longitudinal tensile modulus, *UTS* ultimate tensile strength, *EB* elongation at break, *MC* moisture content

MC is clearly seen (Fig. 6a, b). This MC dependence can be attributed to the hygroscopic nature of lignin, hemicellulose and cellulose in the bamboo fiber cell wall. Experimental investigation of the effect of MC on the elastic modulus of lignin [22–24] and hemicellulose, which were separated chemically from wood, showed that the stiffness of both hemicelluloses and lignin significantly reduced with increases in MC. The mechanical properties of cellulose have also been measured by Sakurada et al. [25] and were found to be less sensitive to changes in MC. In addition to these findings, it was also assumed that MC might weaken the bonding between microfibrills and the matrix of lignin and hemicellulose, resulting in a reduction in both stiffness and strength of the cell wall. This assumption is partly supported by the response of EB to MC augmentation in our tests, which increased slightly with increasing MC although with less significance (Fig. 6c).

The average decreasing ratio of TE, UTS and EB of all the samples for MC increasing from 4.97 to 26.2 % are summarized in Table 1. The results suggest that different mechanical indicators of bamboo fibers show different sensitivities to MC variation. As MC increases from 4.97 to 26.2 %, the TE reduces by 23.23 % on average, followed by a 19.9 % reduction in UTS. MC has minimal effect on EB, with an average growth rate of only 3.25 % across the same MC range. Yu et al. [11] also found that the UTS for 4.5-year-old bamboo fibers show less sensitive to MC than TE, especially at MC levels lower than 10.8 %. Since no other similar publications were found for bamboo fibers, we have to compare our results with similar research conducted on wood species. Gerhards [26] summarized the relative effects of MC on TE and UTS at room temperature based on four previous works [27–30] and indicated that TE and UTS reduced by 21 and 23 %, respectively, with increasing MC from 6 to 20 %. These results show that the sensitivity to MC variability of TE and UTS for wood is rather close. Ozyhar et al. [31] also found that the longitudinal mechanical properties of European beechwood decreased to a different degree with increasing MC. Across a tested MC range of 5.9–16.3 %, the TE and UTS

Table 1 The decreasing ratio of longitudinal tensile mechanical properties of single bamboo fibers with MC increasing from 4.97 to 26.2 %

Bamboo ages	Decreasing ratio (%)		
	TE	UTS	EB
0.5	13.22	11.56	−3.07
1.5	15.46	15.03	−1.57
2.5	38.90	33.37	−5.86
Average	23.23	19.99	−3.35

TE longitudinal tensile modulus, *UTS* ultimate tensile strength, *EB* elongation at break

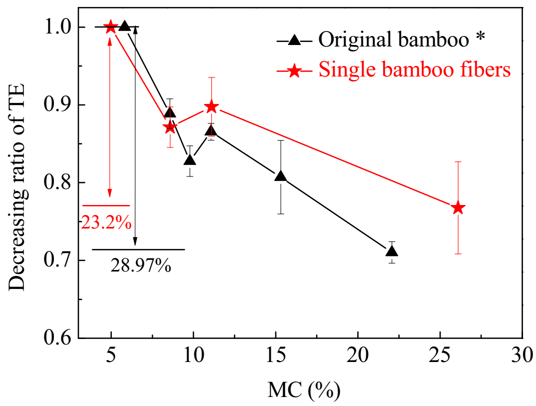


Fig. 7 The decreasing ratio of TE of single bamboo fiber and solid bamboo from 4.97 to 26.2 %. *Asterisk* TE and MC of solid bamboo quoted from Jiang et al. [34]. *TE* longitudinal tensile modulus, *MC* moisture content

decreased by 23 and 30 %, respectively. Therefore, the response to MC change of longitudinal tensile modulus and strength of solid bamboo might behave differently from that of wood, which needs to be confirmed in a future investigation.

The effect of age on the sensitivity to MC change of longitudinal tensile properties of single bamboo fibers

Table 1 also gives a more quantitative description of the effect that age has on TE, UTS and EB sensitivity of bamboo fibers to MC change in a range from 4.97 to 26.2 %. It was found both the changing ratio of TE and UTS increased with the age of bamboo, which indicates that mature bamboo fibers might have greater sensitivity to MC change than younger fibers. Currently, there is no definitive explanation for this phenomenon. However, the explanation is likely to be related to cell wall structure and chemical compositions. Despite this, the tiny difference in MFA among the differently aged bamboo fiber samples does not indicate why we would observe the above-mentioned difference in mechanical property responses to MC change. For wood, as MC increased from 4 % to wet state, Kojima and Yamamoto [32] reported 28.4 and 28.7 % reductions in macroscopic TE of wood (*Cryptomeria japonica* D. Don) with MFAs of 20.6° and 31.4°, respectively. Therefore, more systematic and extensive investigations are required to clarify causes of the variations observed in our research.

Comparison of sensitivity to MC change of longitudinal tensile modulus at single-fiber and macroscopic level

As the main mechanical supporting unit of bamboo, bamboo fibers help to determine the overall mechanical properties of bamboo. Therefore, the reduction of mechanical properties of bamboo fibers will lead to a corresponding reduction in the overall mechanical performance. Figure 7 shows the correlations between TE and MC, both at single-fiber and macroscopic levels for bamboo. Every data point represents the average value of samples for the three defined ages. The results clearly show that the TE variation with MC of solid bamboo agrees well with the mechanical response to MC found for bamboo fibers. However, a more careful examination reveals that there are different sensitivities to MC change for solid bamboo compared to fibers, especially at higher MC levels. For bamboo fibers, the average TE decreasing ratio is 23.2 %, when MC increases from 4.97 to 23.2 %, while the solid bamboo exhibits much greater sensitivity to MC with a decreasing rate of 28.97 % observed in relation to an increase from 5.85 to 22.3 % in MC. Bamboo fibers showed less sensitivity to MC change than bulk bamboo, which maybe partially explained by the unique two-phase composite structure of bamboo, with its reinforcing fiber sheaths embedded in soft parenchyma cells (Fig. 8). The distinct structure and chemical compositions between bamboo fiber sheaths and parenchyma cells indicates different swelling behaviors to MC change, leading to easier internal sliding and greater reduction in stiffness during moisture augmentation. Secondly, the interfacial bonding between fibers or parenchyma cells will also be weakened. Finally, parenchyma cells might suffer from more reduction in stiffness than bamboo fibers due to their much higher MFA and lower lignin content [33].

Conclusion

From our comprehensive investigation of longitudinal tensile properties of single bamboo fibers across three separate ages, as well as four different MC levels, we reach the following main conclusions. Both longitudinal tensile modulus (TE) and ultimate tensile strength (UTS) of bamboo fibers vary little with age, when MC is kept constant. These properties show a linear decrease in relationship to increasing MC from 4.97 to 26.2 % as a whole, while elongation at break (EB) increased slightly. TE was found to be most sensitive to MC change, followed by UTS and then EB. Bamboo age may have a negative influence on the sensitivity to MC change of longitudinal tensile properties of single bamboo fibers. A direct comparison between the TE–MC correlation of bamboo fibers and solid bamboo not only demonstrated that the fiber component in bamboo determined the overall mechanical response to MC, but also revealed that there was smaller sensitivity to MC change at the fiber level.

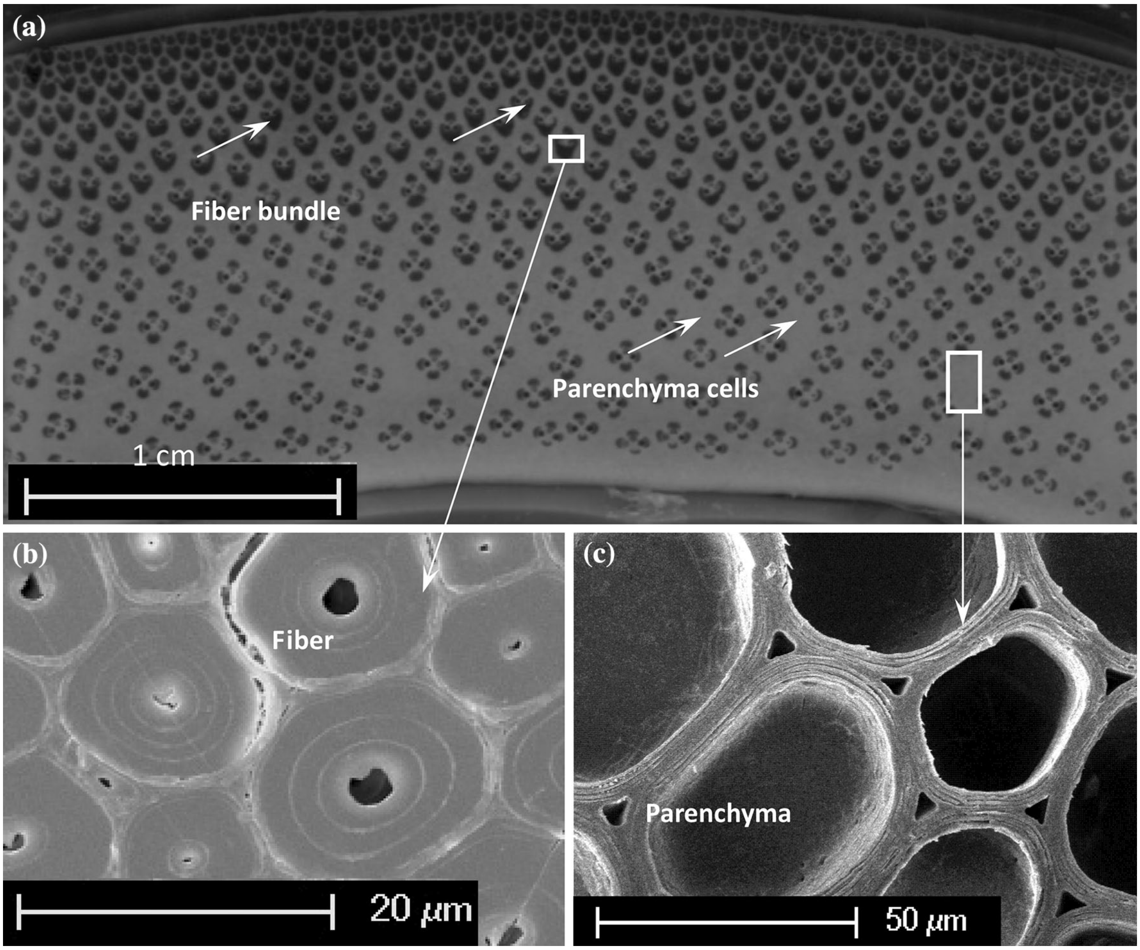


Fig. 8 SEM images showing the morphologies of bamboo transverse section (**a**), bamboo fibers (**b**) and parenchyma cells (**c**)

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References

1. Robson D, Hague J, Newman G, Jeronomidis G, Ansell M (1996) Survey of natural materials for use in structural composites as reinforcement and matrices. Woodland Publishing Ltd, Abingdon
2. Faruk O, Bledzki AK, Fink HP, Sain M (2012) Biocomposites reinforced with natural fibers: 2000-2010. Prog Polym Sci 37:1552–1596
3. Liu DG, Song JW, Anderson DP, Chang PR, Hua Y (2012) Bamboo fiber and its reinforced composites: structure and properties. Cellulose 19:1449–1480
4. Khalil HPSA, Bhat IUH, Jawaid M, Zaidon A, Hermawan D, Hadi YS (2012) Bamboo fibre reinforced biocomposites: a review. Mater Des 42:353–368
5. Gamon G, Evon P, Rigal L (2013) Twin-screw extrusion impact on natural fibre morphology and material properties in poly (lactic acid) based biocomposites. Ind Crop Prod 46:173–185
6. Huang Y, Fei B, Yu Y, Zhao R (2012) Plant age effect on mechanical properties of Moso bamboo single fibers. Wood Fiber Sci 44:1–6
7. Wang H, An X, Li W, Wang H, Yu Y (2014) Variation of mechanical properties of single bamboo fibers (*Dendrocalamus latiflorus* Munro) with respect to age and location in culms. Holzforschung 68:291–297
8. Shao Z, Fang C, Huang S, Tian G (2010) Tensile properties of Moso bamboo (*Phyllostachys pubescens*) and its composites with respect to its fiber-reinforced composites structure. Wood Sci Technol 44:655–666
9. Yu Y, Wang H, Lu F, Tian G, Lin J (2014) Bamboo fibers for composite applications: a mechanical and morphological investigation. J Mater Sci 49:2559–2566
10. Ashby MF, Gibson LJ, Wegst U, Olive R (1995) The mechanical properties of natural materials. I. Material property charts. Proc R Soc Lond Ser A Math Phys Sci 450:123–140
11. Yu Y, Jiang Z, Fei B, Wang G, Wang H (2011) An improved microtensile technique for mechanical characterization of short plant fibers: a case study on bamboo fibers. J Mater Sci 46:739–746
12. Lefeuvre A, Bourmaud A, Morvan C, Baley C (2014) Elementary flax fibre tensile properties: correlation between stress-strain behaviour and fibre composition. Ind Crop Prod 52:762–769
13. Cave ID (1966) Theory of X-ray measurement of microfibril angle in wood. For Prod J 16:37–42

14. Wang XQ, Li XZ, Ren HQ (2010) Variation of microfibril angle and density in moso bamboo (*Phyllostachys pubescens*). J Trop For Sci 22:88–96
15. Wang G, Yu Y, Wang J, Cao S, Cheng H (2011) Microtension test method for measuring tensile properties of individual cellulosic fibers. Wood Fiber Sci 43:251–261
16. Yu Y, Tian G, Wang H, Fei B, Wang G (2011) Mechanical characterization of single bamboo fibers with nanoindentation and microtensile technique. Holzforschung 65:113–119
17. Köhler L (2000) Biphasic mechanical behaviour of plant tissues. Mater Sci Eng C 11:51–56
18. Groom LH, Mott L, Shaler SM (2002) Mechanical properties of individual southern pine fibers. Part I: determination and variability of stress-strain curves with respect to tree height and juvenility. Wood Fiber Sci 34:14–27
19. Groom LH, Shaler SM, Mott L (2002) Mechanical properties of individual southern pine fibers. Part III. Global relationships between fiber properties and fiber location within an individual tree. Wood Fiber Sci 34:238–250
20. Yang JL, Evans R (2003) Predicition of MOE of eucalypt wood from microfibril angle and density. Holz Roh Werkst 61:487–499
21. Bergander A, Salmén L (2000) Variations in transverse fiber wall properties: relations between elastic properties and structure. Holzforschung 54:654–660
22. Salmén L, Kolseth P, Rigdahl M (1986) Modelling of small-strain properties and environmental effects on paper and cellulosic fibers. Composite systems from natural and synthetic polymers. Elsevier, Amsterdam, PAYS-BAS, pp 211–223
23. Cousins WJ (1976) Elastic modulus of lignin as related to moisture content. Wood Sci Technol 10:9–17
24. Cousins WJ (1978) Young's modulus of hemicellulose as related to moisture content. Wood Sci Technol 12:161–167
25. Sakurada I, Nukushima Y, Ito T (1962) Experimental determination of the elastic modulus of crystalline regions in oriented polymers. J Polym Sci 57:651–660
26. Gerhards CC (1982) Effect of moisture content and temperature on the mechanical properties of wood: an analysis of immediate effects. Wood Fiber Sci 14:4–36
27. Kufner M (1978) Modulus of elasticity and tensile strength of wood species with different density and their dependence on moisture content. Holz Roh-Werkst 36:435–439
28. Schniewind AP, Pozniak RA (1971) On the fracture toughness of Douglas fir wood. Fract Mech 2:223–233
29. Sulzberger PH (1953) The effect of temperature on the strength of wood, plywood and glue joints. Aeronaut Res Consultative Com Rep ACA-46. Melbourne, Australia
30. Wilsion TRC (1932) Strength-moisture relations for wood. USDA Tech Bull. No. 282 US Dep Agric, Washington DC
31. Ozyhar T, Hering S, Niemz P (2012) Moisture-dependent elastic and strength anisotropy of European beech wood in tension. J Mater Sci 47:6141–6150
32. Kojima Y, Yamamoto H (2004) Properties of the cell wall constituents in relation to the longitudinal elasticity of wood. Part 2: origin of the moisture dependency of the longitudinal elasticity of wood. Wood Sci Technol 37:427–434
33. Wang H (2010) Study the mechanism of moisture affecting the bamboo fibre cell walls and macroscopic mechanical behavior. Central South University of Forestry and Technology. Changsha Hunan, PR China pp 47–50
34. Jiang Z, Wang H, Yu Y, Tian G (2012) Hygroscopic behavior of bamboo and its blocking units. J Nanjing For Univ (Nat Sci Edn) 36:11–14