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An improved microtensile technique for mechanical characterization of short plant fibers: a case study on bamboo fibers

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Abstract This article aims to present an improved technique for the mechanical characterization of single plant fibers. In particular, our efforts lie in resolving the difficulties involved in the testing of short plant fibers with only a few millimeters in length and tens of microns in width. Such fibers are potentially used as reinforcement phase in polymer composites. A special fiber gripping system, consisting of a pair of fiber clamps, a 3D positioning stage and two digital microscopes, was developed and combined to a small commercial mechanical tester. The resultant testing system possesses great convenience and reliability for the installation, orientation adjustment, and gripping of short plant fibers, as well as powerful control and data analysis functions. The equipment was then applied to the mechanical characterization of single bamboo fibers under air-dried state. Furthermore, the capability of this equipment for studying moisture content-related mechanical and viscoelastic properties of bamboo fibers was also successfully demonstrated.

Introduction

Plant fibers are renewable, easily available, biodegradable, and combustible with minimal environmental impact during their whole life cycle [1]. Plant fibers possess versatile advantages of high aspect ratio, high strength to weight ratio, low density, which makes them potential replacement for man-made fiber in fiber-reinforced composites [2–5]. However, an enhanced utilization of plant fibers for composites or other fiber-based products requires more knowledge of the basic properties of fiber itself [6]. Especially, an increased understanding of the mechanical behavior is needed before they can be used in confidence as the load-bearing constituent in the structural components. Although many studies have been performed on the mechanical characterization of plant fibers [7, 8], most of them are measured at fiber bundles level, other than single fiber level. The study of mechanical behavior of single plant fibers has gained momentum in recent years not only because of the valuable engineering mechanical data for the optimal design of fiber-based composite materials, but also the capability of this technique to establish a direct correlation between fiber mechanical properties with plant cell wall microstructure and chemical compositions. The latter is particularly important in biomaterials science with focus on learning from the hierarchically organized structure and from specific molecular mechanistic phenomena at the cell wall level [9–11].

The most challengeable issue involved in mechanical characterization of single plant fibers is how to properly grip fibers, which is especially difficult for some short plant fibers, such as fibers from wood and bamboo. These fibers normally range from 0.5 to 5 mm in length and 5 to 40 μm in diameter, which means specialized techniques and equipments must be developed to obtain precise and repeatable results. After numerous reports demonstrated conventional mechanical gripping and methods of gluing fibers to medium usually gave unacceptable results, Groom et al. [12] developed a rapid single fiber testing protocol based on a ball and socket assembly, which made it possible for the first time to map whole tree variation in

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fiber mechanical properties [13, 14]. In their study, a custom-built microtensile tester equipped with a pair of delicate clamps was developed. The clamps were actually a pair of thin metal plates with miniature "V" shape slots to grasp the epoxy droplets applied near the ends of single wood fibers in advance. It seems the thickness of metal plate should be carefully selected to minimize the length of fibers required for testing and meanwhile ensure minimal deflection during tension. In order to investigate shorter fiber types, like compression wood tracheids or libriform fibers of deciduous trees, Burgert et al [15] had to use a foliar frame with 200-μm thick as a medium to carry single wood fibers. However, the fiber elongation had to be measured by a video extensometer to reduce the errors caused by indirect fiber gripping. Except for fiber gripping, stress concentration caused by deviation of fibers from tensile direction is another important source of error in single fiber testing [16], which was not effectively dealt with in most previous investigations. Furthermore, little efforts have been performed to control the moisture content of fibers during tensile testing.

In this article, a custom-built fiber gripping system was developed and combined to a small, high-resolution commercial mechanical tester to give an unprecedented accuracy, and convenience for mechanical testing of single plant fibers. A matched miniature environmental chamber was also developed to facilitate the study of moisture dependence of fiber mechanical properties. Bamboo fibers are selected here for the case study based on the following reasons. First, bamboo has long been the subject of extensive researches of biomimetic design of composites due to its hierarchical structure and superior mechanical properties [17]. Bamboo fibers were thought to be the key to reveal the mystery of bamboo in both extraordinary strength and toughness [18]. Secondly, bamboo fiber reinforced composites (BFRC) have been attracting intensive attentions in the materials science field [19, 20], which highlights the necessity of understanding of fiber mechanical properties to improve the quality of bamboo fiber-based composites. Finally, the length of bamboo fibers normally ranges from 1 to 3 mm with an average value less than 2 mm, which make them suitable for evaluating the capability of the developed microtensile technique for short plant fibers.

Materials and methods

Sample preparation

Moso bamboo (*Phyllostachys pubescens* Mazei ex H.de Lebaie) aging with 1.5, 2.5, and 4.5 years was taken from a bamboo plantation located in Zhejiang Province, China. Figure 1 shows the microstructure of Moso bamboo. Vascular bundles that perform the function of mechanical reinforcement are embedded in the matrix of ground parenchyma with higher distribution density approaching to the outer layer of a culm wall (Fig. 1a, b). The sheaths of sclerenchyma fibers in the vascular bundles (Fig. 1c) are composed of long and thick-walled fibers that are much stiffer, and stronger than the thin-walled parenchymal cells (Fig. 1d).

Cubic blocks were cut out from the 20th internodes of a bamboo culm and then splitted into sticks measuring about 1 (radial) × 1 (tangential) × 15 (longitudinal) mm^3. Bamboo fibers were chemically extracted from these sticks in a solution comprised hydrogen peroxide and glacial acetic acid [12]. Figure 2a shows the microscope image of chemically isolated bamboo fibers. All the fibers are straight, whereas wood fibers tend to curl after drying. Under a microscope, bamboo fibers with minimal damage were carefully selected and placed across a gap in an organic glass panel. Two epoxy droplets with approximate 200 μm in diameter were then placed at the ends of each fiber with an approximate spacing of 0.7–0.8 mm via a super fine tweezers (Fig. 2b). The epoxy was allowed to solidify at 60 °C for 24 h, followed by an additional balance at room condition for 24 h. More details for sample preparation could be found in Groom et al. [12].

Microtensile assembly

A special fiber gripping system was developed and combined to a small high-resolution commercial mechanical tester (Microtester 5848, Instron, USA) to measure the mechanical properties of bamboo fibers (Fig. 3a, b). The purpose of this design is to fully utilize the advantages of high resolution in load and displacement, as well as the powerful control and data analysis functions of the commercial machine. A miniature environmental chamber was also developed to facilitate the study of moisture dependence of fiber mechanical properties (Fig. 3c). The key part of the fiber gripping system is a pair of innovative fiber clamps that can effectively grasp the epoxy droplets at the ends of bamboo fibers during tension (Fig. 3d, e). The fiber clamps are designed to deal with fibers as short as 1.0 mm in length. Figure 4 gives a detailed schematic representation of the fiber clamps. High-resolution laser cutting technique was adopted to produce slots ranging from 100 to 120 μm in width. The "θ" and "a" values are, respectively, the angle and the length of shorter side of the trapezoid shown in Fig. 4. Both of them can be adjusted according to the length of fibers to be tested and the stiffness requirement for the clamps, minimizing the possible deflection during tension. For bamboo and other longer fibers, the typical "a" value is 200 μm. The value can be reduced for shorter fibers.

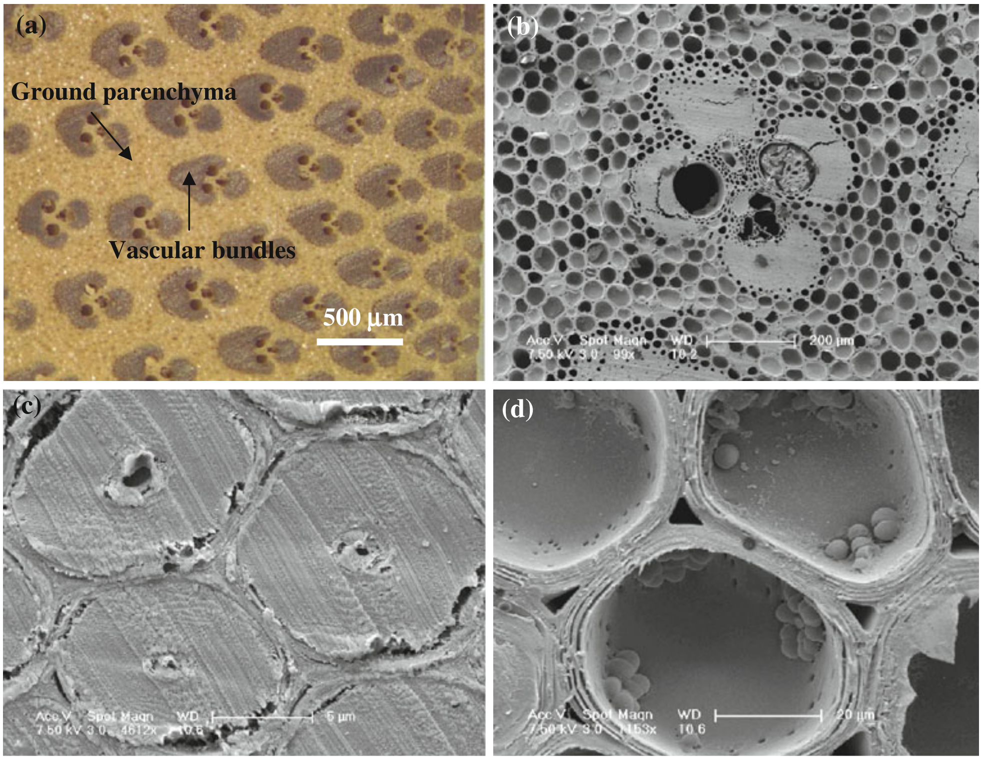


Fig. 1 Transverse section of bamboo showing vascular bundles embedded in the ground parenchyma. **a** the graded distribution of vascular bundles, **b** vascular bundles embedded in the ground parenchyma, **c** bamboo fibers, **d** parenchymal cells

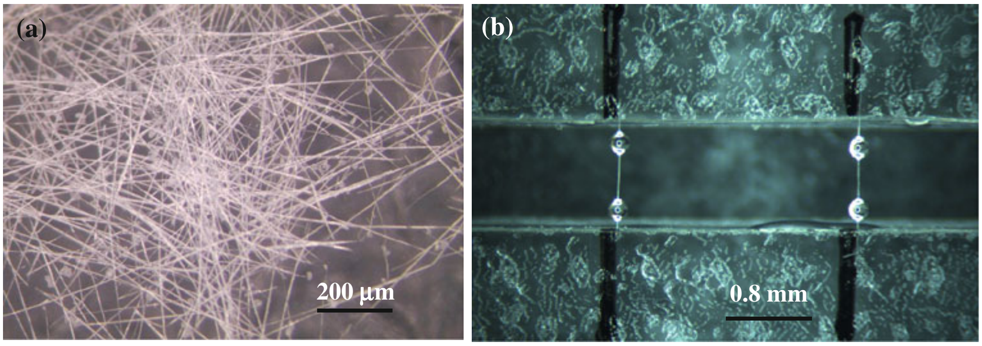


Fig. 2 Preparation of bamboo fibers for microtensile test. **a** the chemically isolated bamboo fibers, **b** epoxy droplets placed at the ends of bamboo fibers

Microtensile testing at air-dried state

During a typical fiber testing experiment, the prepared bamboo fiber was first installed in the fiber clamps with an ultra-fine tweezers under the vertical microscope. The fiber was then pre-tensioned at 5 mN. Because of the small size of the specimens, it is difficult to place a fiber with its axis perfectly aligned with the tensile direction. An integrated 3D positioning stage was used to ensure the tensile direction aligned exactly with the fiber axis (Fig. 5). The initial tensile span of bamboo fibers between the two epoxy droplets was then measured directly with the vertical microscope. The capacity of load cell used was 5 N. Elongation was recorded from the crosshead movement with a resolution of 0.02 µm. The tensile speed was 48 µm/min. Tests were carried out under an environment of 23 °C and 35% in relative humidity (RH). More than 40 bamboo fibers were tested for each bamboo age.

To calculate the tensile strength and modulus of bamboo fibers, the cell wall area of every broken fiber was determined with a confocal scanning laser microscope (Meta 510 CSLM, Zeiss). The broken fibers were first immersed

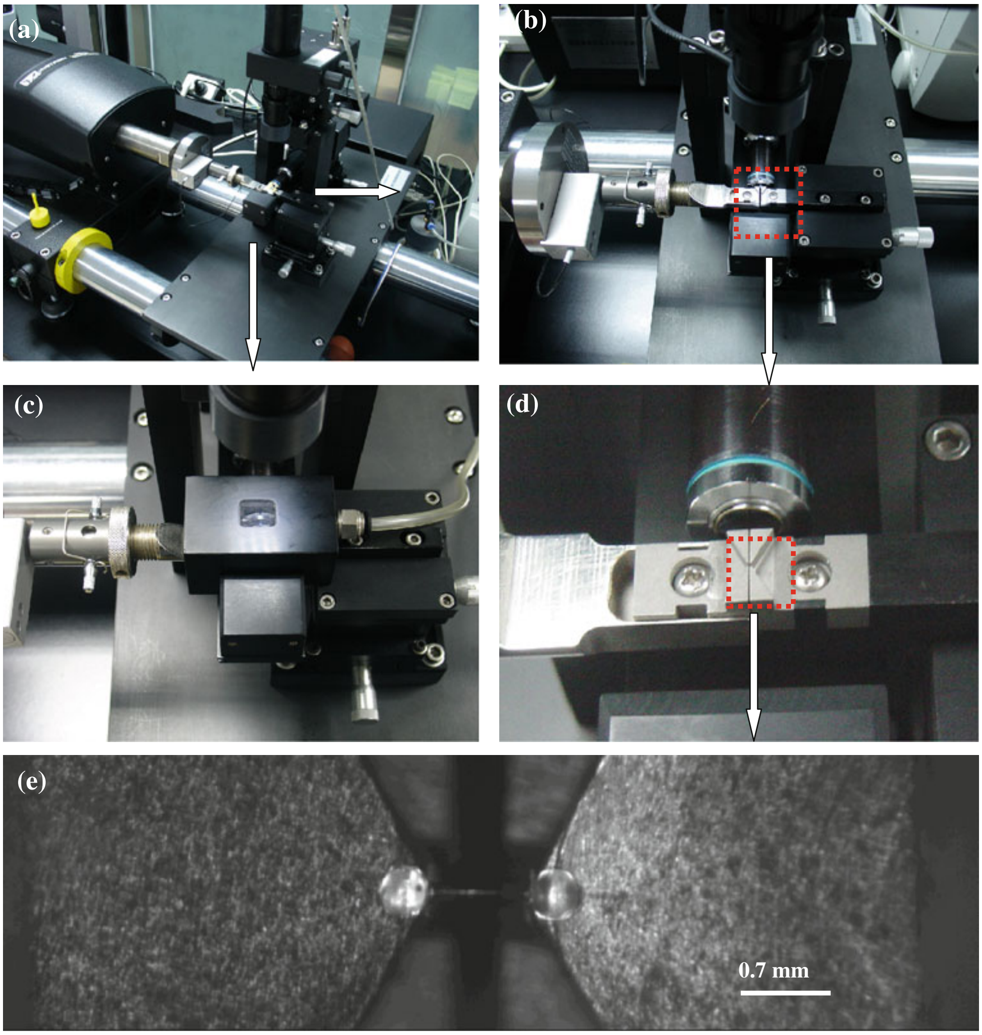
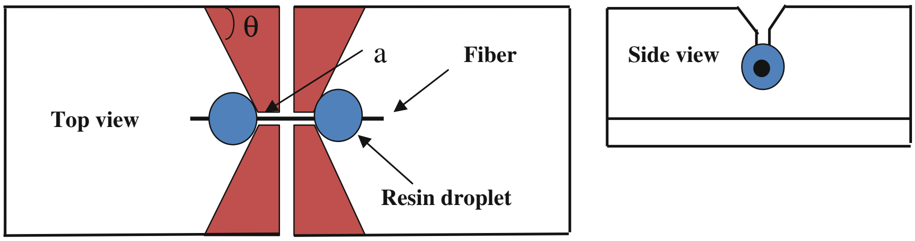


Fig. 3 **a** Overview of the custom-built microtensile testing system, **b** a magnified view of the tensile zone, **c** a magnified view of the tensile zone with miniature environmental chamber, **d** fiber clamps, **e** single bamboo fibers installed in the fiber clamps

Fig. 4 Schematic diagrams of fiber clamps for short plant fiber testing. *Left* top view, *right* side view



in 0.1% acridine orange solution for 20 s and then rinsed in distilled water for several times [12]. The fibers were then imaged with a 63× immersion oil objective. The cell wall area of the fibers was then measured with image analysis software Image Pro 6 (Fig. 6). With the value of cell wall area and the initial span, load–elongation curves can be converted to stress–strain curves to extract tensile strength and modulus. Finally, some of the broken bamboo fibers were coated with conductive platinum film and observed with a field-emission scanning electron microscope (XL30-FEG-SEM, FEI, USA).

Microtensile testing at elevated moisture content

Before proceeding with bamboo fiber testing, RH in the miniature environmental chamber was, respectively, kept at 11, 33, 64, and 90.8% for more than 30 min, which corresponded to 4.1, 8.2, 10.8, and 20.7%, respectively, in

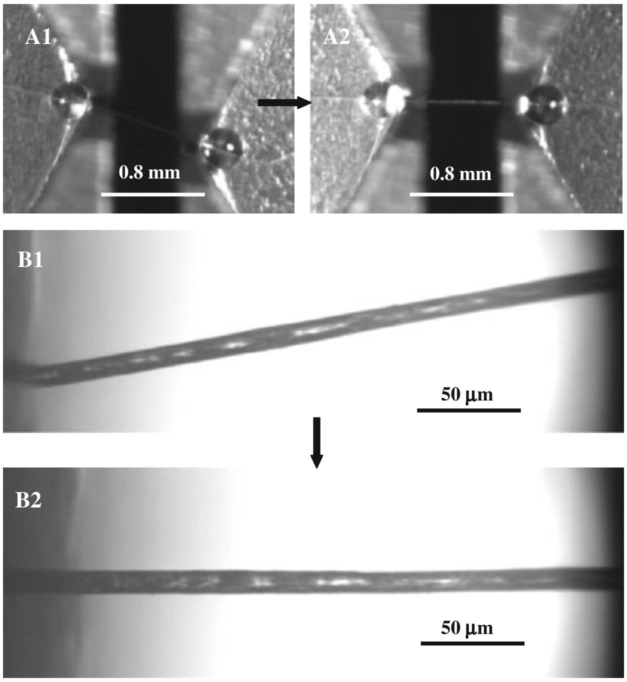


Fig. 5 Micro-adjustment of fiber orientation to align with loading direction. Fiber adjustment in **a1–2** horizontal and **b1–2** vertical directions

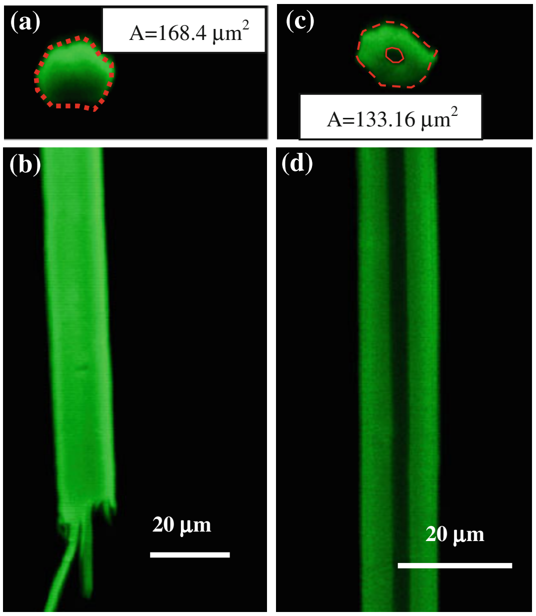


Fig. 6 CLSM images of single broken bamboo fibers for cell wall area measurement. **a** Cell lumen invisible, **b** small cell lumen discernable

terms of equilibrium moisture content (EMC) based on the sorption isotherm of chemically extracted bamboo fibers developed in our lab. For fiber testing at wet state, liquid water was carefully dropped on the bamboo fibers under 90.8% RH before tension. For each moisture content level, 16–29 bamboo fibers with 4.5 years old were tested.

Short-term creep testing at elevated moisture content

For creep test, bamboo fibers were first conditioned for 30 min in the environmental chamber to reach EMC of 7.5, 10.2, and 18.7%. The method to test bamboo fibers at wet state was the same as that mentioned above. After conditioning, fibers were loaded to 100 mN and kept for 30 min. Though 30 min is a little short for a normal creep test, it is reasonable since our purpose is to demonstrate the capability of the apparatus developed to perform creep test on short plant fibers. All the tested fibers had similar size in diameter ranging from 13 to 15 µm, which resulted in similar tensile stress under the same load. For every moisture content level, 5–7 fibers were tested.

Results and discussion

Mechanical properties of bamboo fibers at air-dried state

The typical stress–strain curves of single bamboo fibers with ages from 1.5 to 4.5 years are presented in Fig. 7. All the fibers tested exhibit linear stress–strain behavior to failure, which is somewhat different from that of wood fibers. Groom et al. [12] found the shape of the stress–strain curve of softwood fibers depended on microfibrillar angle (MFA). Fibers with MFA larger than 20° exhibited curvilinear behavior up to 60% of the load-carrying capacity of fibers followed by linear behavior to failure, while fibers with MFA less than 20° appeared to be full linear during the test. Since the MFA of Moso bamboo fibers tested here varied from 8° to 13° [21], which could explain why all the bamboo fibers display linear stress–strain behavior. Furthermore, stress–strain curves can be used as additional evidence to evaluate whether slipping between fiber and resin droplet occurs during tension,

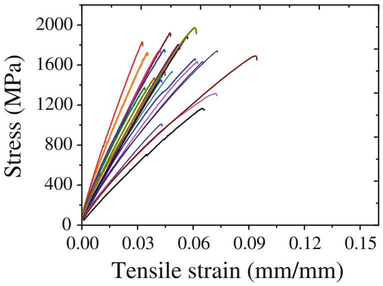


Fig. 7 Stress–Strain curves of single bamboo fibers with ages from 1.5 to 4.5 years

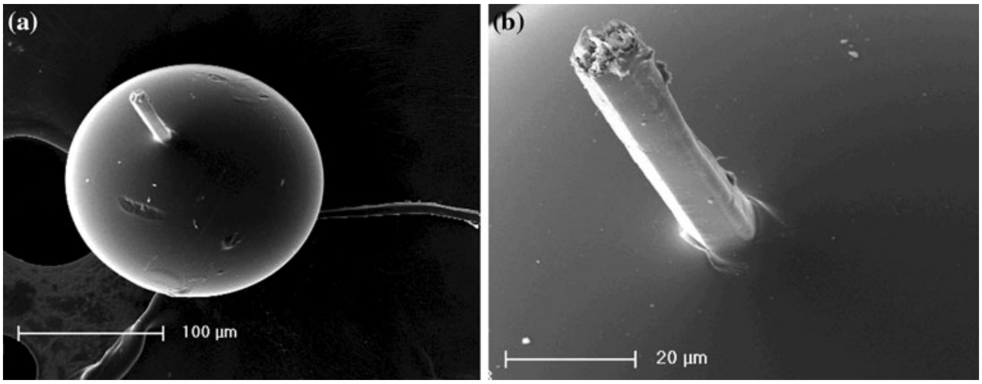


Fig. 8 Fracture surfaces of single broken bamboo fibers at different magnifications

especially when the normal curves should be linear. Our results indicate the method of fiber gripping adopted here causes negligible slipping.

In order to further make sure no or insignificant slipping occurred at normal conditions of tension, some broken fibers were carefully observed under a high-resolution SEM. Figure 8 shows the micrographs of a single broken bamboo fiber at different magnification. It can be concluded no epoxy resin runs onto the testing length of the fiber, which may strengthen the mechanical properties of tested fibers. Moreover, no visible "pulling out" of fiber could be observed at the proximity of interface between fiber and resin.

Cell wall areas, tensile span, maximum load, ultimate tensile strength, and tensile modulus of elasticity of bamboo fibers are summarized in Table 1. A large variation is found both for cell wall area and ultimate load, ranging from 65.01 to 289.65 µm^2 and from 84.15 to 387.41 mN, respectively. The coefficient of variation (COV) for them is 33%. A much less COV 19% is found for tensile span. Since tensile span may has negative effect on the tensile strength and modulus of fibers, the technique of sample preparation should be further improved to reduce the variability of tensile span. The tensile strength of bamboo fibers measured is found to range from 0.85 to 2.94 GPa with a average value of 1.56 GPa, while tensile elastic modulus ranges from 20.34 to 60.01 GPa with a average value of 33.03 GPa. As far as we know, no experimental data on the mechanical properties of single bamboo fibers have been reported. However, the tensile strength and the modulus of bamboo fiber bundles could be found in several publications. Amada et al. [22] found a tensile strength of 0.61 GPa and a tensile modulus of 46 GPa for bamboo fiber bundles, which was actually calculated based on the volume ratio of fibers to parenchymal cells as well as the macroscopic tensile modulus and strength of bamboo. Recently, Shao et al. [23] experimentally measured the tensile strength and modulus of Moso bamboo fiber bundles to be 0.482 and 33.9 GPa, respectively. It can be found the tensile modulus measured on single bamboo fibers agrees well with fiber bundles, whereas the tensile strength is much higher than the latter. This is reasonable since the debonding between fibers during tension will significantly reduce the loading capacity of bamboo fiber bundles.

The effect of moisture content on the mechanical properties of bamboo fibers

Figure 9 presents the typical stress–strain curves of single bamboo fibers with 4.5 years old under different moisture contents. Increased moisture content reduces the slope of curve but do not change its linearity, indicating no slipping occurs with increase of moisture content. This demonstrates the technique developed here is capable of evaluating the mechanical performances of plant fibers at variable moisture content. Figure 10 gives more quantitative description on the effect of moisture content on the tensile modulus and strength of bamboo fibers. The

Table 1 Statistical results of tensile testing of single bamboo fibers

	Cell wall area (µm^2)	Tensile span (mm)	Ultimate load (mN)	Tensile strength (GPa)	Tensile modulus (GPa)
Max	289.65	1.09	387.41	2.94	60.01
Min	65.01	0.42	84.15	0.85	20.34
Average	140.58	0.70	212.06	1.56	33.03
CV (%)	0.33	0.19	0.33	0.23	0.23
N	126	126	126	126	126

COV coefficient of variation, *N* number of measurements

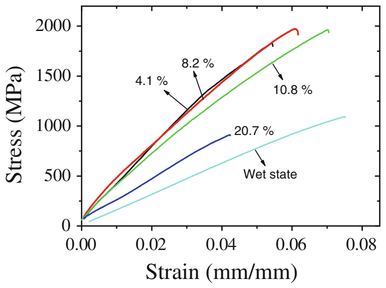


Fig. 9 Stress–strain curves of single bamboo fibers in tension under different moisture contents

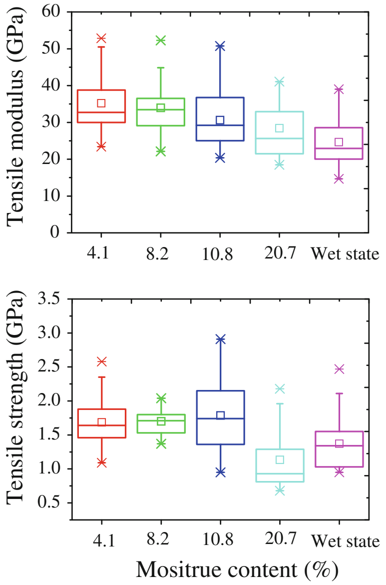


Fig. 10 Effects of moisture content on the tensile modulus and strength of bamboo fibers with 4.5 years old

anticipated reduction of tensile strength and modulus of bamboo fibers with the increase of moisture content was clearly observed. Furthermore, it seems tensile strength shows less sensitive to moisture content than tensile modulus especially when moisture content is less than 10.8%. More experiments are needed to confirm if this is a general rule.

The effect of moisture content on the short-term tensile creep behavior of bamboo fibers

Figure 11 shows the time dependency of tensile creep deformation of bamboo fibers in relation to the moisture content. The instantaneous strain appeared immediately after the load was applied, varying with moisture content. Subsequently, the creep deformation increased approximately linearly with time. These observations are similar to a recent report on the tensile creep behavior of single wood fibers [24]. In order to better describe the effect of moisture content on the creep behavior of bamboo fibers, substantial creep is calculated by subtracting the initial instantaneous strain from the total tensile strain. Figure 12 shows the effect of moisture content on the substantial creep of bamboo fibers. It is clear that substantial creep of bamboo fibers increase with moisture content. A much faster increase in substantial creep was found for the range from 7.5 to 10.2%, compared to the range from 10.2% to fibers saturation. Since 10.2% is very close to the value 12% that is a critical moisture content at which BET multilayer adsorption of water molecular is finished and capillary condensed water begins to form in the micropores of wood cell wall [25], it was assumed the creep of bamboo fiber might be controlled by the plasticizing effect caused by the monolayer and multilayer adsorption of water molecular in the cell wall. More systematic investigations are needed before the effect of moisture content on the creep of plant cell wall can be well described.

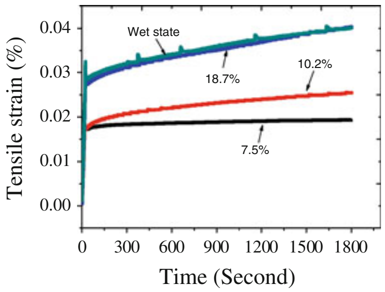


Fig. 11 Time–creep curves of bamboo fibers with 1.5 years old under different moisture contents

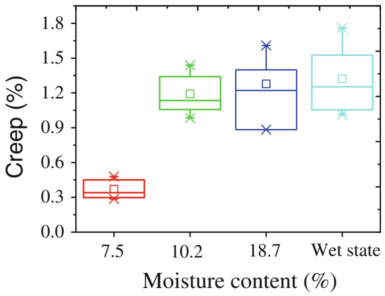


Fig. 12 Effect of moisture content on the creep of bamboo fibers with 1.5 years old

Conclusions

A custom-built fiber gripping system was developed and combined to a small commercial mechanical tester to furnish it with the capability to characterize the tensile mechanical properties of short plant fibers with excellent convenience and reliability. Mechanical characterization of bamboo fibers at constant and changeable moisture content both demonstrates the function and reliability of the modified mechanical tester. Measurements on other plant fibers are in progress, to extract more mechanical and bionic information from cell walls with a wide range of structural and chemical features.

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