

Variation of Tensile Properties of Single Fibres of *Dendrocalamus farinosus* Bamboo

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This study investigated the mechanical behavior and the variation of *Dendrocalamus farinosus* single fibres, which were isolated from fibre bundles using a chemical method. A micro-tester was applied to determine the influence of the age of the bamboo sampled, as well as the longitudinal and radial positions, on three indicators featuring tensile properties at the fibre level. The results indicated that the single fibres had a brittle failure mode, resulting in average tensile strength and tensile modulus (MOE) values of 1.06 and 26.93 GPa, respectively. The differing ages and positions across the whole culm appeared to be minor in relation to their tensile properties, which reached a near-optimal state at 2 years old and remained fairly constant. This work could provide basic-data for further research on bamboo properties and increase attention to a potential supplementary material to moso bamboo in industrial utilization.

Key words: Variation; *Dendrocalamus farinosus*; Single bamboo fibres; Microtensile test

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INTRODUCTION

In recent years, researchers have begun to pay more attention to potential applications of cellulosic materials due to their renewable nature and other properties. Bamboo is a natural-fibre reinforced bio-composite, which provides an abundant source of cellulose fibril resources. It consists of unidirectional fibres as a reinforcement, and parenchymatous ground tissue as a matrix. Owing to its fast growth, superior mechanical properties, rapid regeneration, and eco-friendly properties, bamboo fibres have been applied in numerous industries, ranging from pulp, paper, and textiles through to construction (Dirceu 1971; Jain *et al.* 1992; Li 2006). However, to ensure that bamboo material remains stable during its use in load-bearing structural applications, an increased understanding of the mechanical behavior of the bamboo fibre itself is needed.

To date, many previous studies have characterized the mechanical properties of plant fibres. The main variables among them are the method used to isolate the single plant fibres and a suitable gripping system used to conduct tests. These two variables have effects on studies of single fibres in plants, especially for those with short fibres, such as bamboo and some wood. For example, previous studies had shown that fibres isolated from spruce using either a mechanical pull-out method or soft chemical treatment had notably different mechanical properties (Burgert *et al.* 2002, 2005a). These studies discovered that chemically isolated fibres were lower in strength than those isolated physically, while the stress-strain curves for both isolation methods were broadly similar. The chemical isolation methods allow one to recover a large quantity of intact cell materials and obtain a great

quantity of fibre, while the artificial pull-out method requires a skilled technical expertise and is based on observation of a few single fibres.

Recently, Chen *et al.* (2011) tested the static contact angle and mechanical properties of bamboo single fibres isolated using four different chemical isolation methods. Furthermore, numerous studies have demonstrated that mechanical gripping, as well as methods of gluing short fibres, can be applied to achieve precise and repeatable testing results (Groom *et al.* 2002a; Burgert *et al.* 2003). Among available methods for gripping single short bamboo fibres, Yu *et al.* (2010) showed that by combining a custom-built fibre gripping system to a small, high-resolution mechanical tester, it is possible to achieve previously unprecedented levels of accuracy, while at the same time reducing the difficulty of conducting mechanical tests on single short fibres.

The mechanical properties of single fibres may be influenced by many factors, such as moisture content, microfibril angle (MFA), plant species, as well as the age and location of fibres. For wood fibres, tensile behavior in relation to their mean MFA has been extensively studied (Page and El-Hosseiny 1983). In the case of wood, the modulus of elasticity (MOE) decreases with increases in the mean MFA, namely wood fibres with small MFA have a high MOE. There was also some experimental evidence to show that wood fibres' behavior were to some degree dependent on growth ring and their location along the height of a tree stem (Groom *et al.* 2002b; Mott *et al.* 2002).

For bamboo fibres, most previous studies have focused on monopodial bamboo, especially moso bamboo (*Phyllostachys pubescens*) fibres. The results indicated that there was little variation across samples of different ages for tensile strength and MOE (Yu *et al.* 2011a; Huang *et al.* 2012). The mechanical characterization of bamboo fibres at changeable moisture contents has also been studied (Wang 2010; Yu *et al.* 2011b). Although sympodial bamboo fibres with a high content of cellulose and seemingly favorable mechanical properties were demonstrated (Ren *et al.* 2014; Wang *et al.* 2014), especially, two fibres of this species were observed to reach their optimal mechanical performance after just 1 year. However, little research has been conducted on how culm height and radial positions affect its mechanical properties. Critically, no mechanical behavior properties have been reported in relation to correlated variables such as fibre morphology, MFA, ages, and different positions for single fibres isolated from *D. farinosus* or other sympodial bamboo species. These properties could be potentially important to help identify whether mechanical properties reach a near optimal state at a young age, as well as whether these properties are uniform across the cross section and the length of bamboo culm. Sympodial bamboos have not been fully developed in Chinese industry, while monopodial ones (moso bamboo) are almost popularly used. The difference may be connected with the fact that there has been relatively little fundamental study of sympodial bamboos. This information is critical for providing further support data to broaden the commercial application of fibres from those traditionally used in pulp production to the construction industry.

In order to fill the mechanical experimental gaps with variables of fibre morphology, ages, and positions in the sympodial bamboo, the paper presents the results of tensile mechanical properties of a sympodial bamboo culm from *D. farinosus* (Keng et keng f.) Chia et H. L. Fung. Microtensile techniques were used to demonstrate the variations of the fibre tensile strength, MOE, and elongation at break at different ages, height positions, and radial positions along the functionally graded structure of a bamboo culm.

EXPERIMENTAL

Materials

D. farinosus samples (2, 3, 4, and 5 years old) were collected from Zhuhai town, Sichuan Province, China. The selected bamboo culms had an average external diameter of 65 mm, average length of 602 mm, and had no obvious visible decay or defects. Bamboo blocks of approximately 20 mm × 10 mm × *t* mm (thickness of the bamboo culm wall) were cut from the base (1.5 to 3.5 m from the ground) for each of the sampled ages. Blocks from the base, middle, and upper positions of the 4-year-old culm were taken to observe potential variations along different culm height positions (Fig. 1A, B), with blocks also taken from the outer, middle, and inner positions of the 4-year-old culm to analyze variations along different radial positions (Fig. 1C).

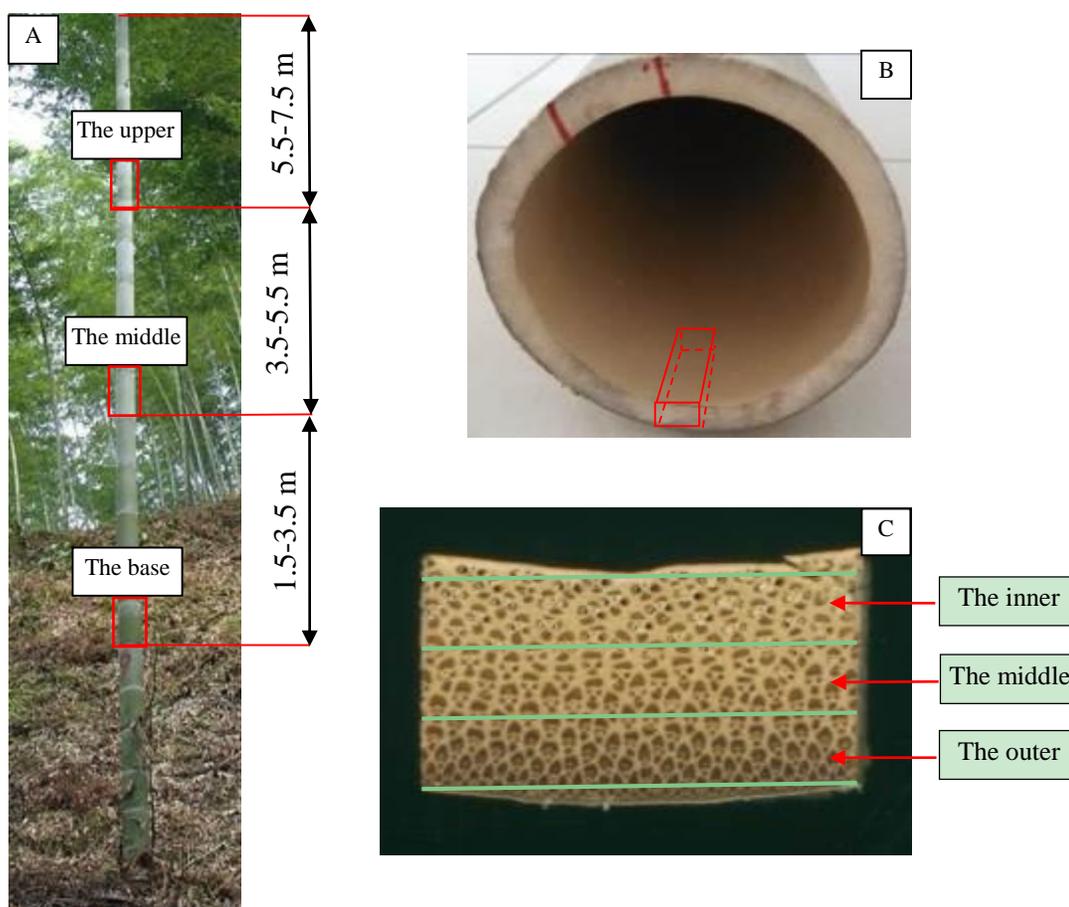


Fig. 1. The methods of splitting *D. farinosus* bamboo blocks (A and B) along different culm height positions and (C) along radial direction

Methods

Extraction of bamboo single fibres

All the fibre extractions were carried out based on the procedure established by Groom *et al.* (2002). Bamboo blocks were cut into small strips (1 × 1 × 10 mm³) and treated in the mixture of hydrogen peroxide (H₂O₂, 30%) and glacial acetic acid (CH₃COOH) in a 1:1 volume for fibre separation. The solutions were heated to 60 °C, and the bamboo strips were immersed for 15 h. The softened sticks were artificially stirred into separated single

fibres by using a glass rod and washed in distilled water until a neutral pH was reached. The fibre solution was then dropped on glass slides to be air-dried and kept for sample preparation.

Sample preparation

Under a vertical microscope, single fibres with minimal damage were carefully selected and placed across a gap of 1.8 mm in width on an organic plastic panel. Two epoxy droplets with an approximate diameter of 200 μm were then placed at two ends of each fibre using an ultra-fine set of tweezers to ensure that no epoxy resin was placed on the testing fibre between the two droplets. The thermoset epoxy was cured in an oven at a temperature of 60 $^{\circ}\text{C}$ for 24 h, with an additional balance at room conditions applied for 24 h. More details on the sample preparation applied are provided in Yu *et al.* (2010).

Microtensile tests

The prepared fibres were placed in the fibre clamps using an ultra-fine set of tweezers under a vertical microscope. The fibre orientations were aligned in the loading direction, with XYZ micro-adjustment used to prevent tensile shear failure. All tests were conducted using an Instron 5848 Micro-Tester (USA). The advantages of this type of Micro-Tester are covered in Yu *et al.* (2011b). Tests were carried out under a load cell with a 5 N capacity at a constant speed of 0.0008 mm/s and in environmental conditions of 23 $^{\circ}\text{C}$ and relative humidity (RH) of 20-35%. More than 60 bamboo fibres were tested for each age group and each bamboo position, thus ensuring a minimum of 30 reliable measurements were recorded for each test sample.

In order to obtain the tensile strength and MOE of single fibres, the cross-sectional area and image of each broken fibre were measured using a confocal scanning laser microscope (CLSM, Meta 510, Zeiss, Germany). After tests, the broken fibres were first immersed in 0.1% acridine orange solution for 3 min and then rinsed in distilled water several times. The tensile span is represented by the length of the fibre between the epoxy spheres and was measured under the microscope before the tension test (Fig. 3a). The value of the cross-sectional area, tensile span, and stress–strain curves were derived to calculate the tensile strength, MOE, and elongation of bamboo fibres.

A certain number of broken bamboo fibres were carefully observed under a field-emission scanning electron microscope (FESEM, XL30, USA) to make sure that no epoxy resin had run onto the testing length of the fibre, and that no visible “pulling out” of fibre at the proximity of interface between the fibre and resin had occurred. This was done to ensure that the mechanical properties of tested fibres had neither been artificially strengthened nor weakened.

RESULTS AND DISCUSSION

Morphology and Tensile Behavior of Bamboo Single fibre

The fibres prepared with hydrogen peroxide and glacial acetic acid ($\text{H}_2\text{O}_2 + \text{CH}_3\text{COOH}$) had experienced extensive loss of lignin in addition to partial hydrolysis of some hemicellulose (Burgert *et al.* 2005b; Xu and Tang 2006). In the process of dehydration under air drying, the surface of a fiber becomes buckled, and the orientation of the microfibrils is revealed, which can be clearly seen in Fig. 2c, due to the degradation of cell wall components. Moreover, a negligible lumen can be found in Fig. 3b and the cross section

of a single fibre appears almost as a circle, which may be due to the intrinsic small lumen and its collapse. As a means of calculating the diameter of a single fiber, it was assumed that the cross section could be accurately described as a circle or concentric circles.

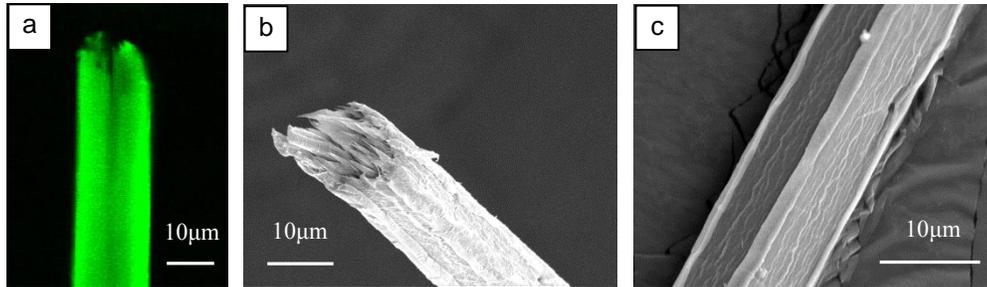


Fig. 2. Fracture surface under CLSM (a) and under FESEM (b), and the morphology (c) of single bamboo fibre

The statistical results of the tensile span and cross-sectional area of all single fibres are shown in Fig. 3 and Table 1. It can be seen that the tensile span was concentrated in the range of 0.7 to 1.0 mm, with an average value of 0.87 mm. Generally, longer fibres have more defects, which renders such fibres more easily damaged and negatively affects the tensile strength and modulus of fibres. Effective cross-sectional areas of testing fibres ranged from 83.88 to 382.05 μm^2 , with an average value of 175.23 μm^2 and coefficient of variation (CV) of 29%.

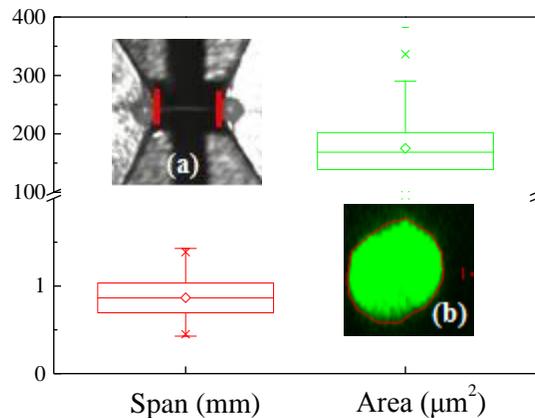


Fig. 3. Tensile span (a) and cross-sectional area (b) of a single bamboo fibre

The typical load–displacement curve and the converted stress–strain curve in relation to the tensile span and cross-sectional area of the fibres are shown in Fig. 4. The stress increases linearly with respect to strain for all specimens. The curves are important to evaluate whether slipping between fibre and resin droplets occurred during tension, because if slippage occurs, the relationship of stress and strain will depart from linearity. Furthermore, the tensile modulus is calculated by taking the corresponding values of stress and strain from the linear portion of the graph. The mean MFA for *D. farinosus* based on X-ray diffraction was reported as 8.5° (Liu *et al.* 2014), while the reported MFA for most wood, calculated using the same method, was 20°.

The previous results indicated that fibres with large MFA showed ductile behavior, having the potential for coping with a large strain, while fibres with small MFA showed brittle failure and high elastic modulus (Sedighi 2006). The linear curve and the smooth fracture surface respectively observed in Figs. 2a and 2b, are consistent with a brittle failure mode and suggest the potential for high elastic modulus.

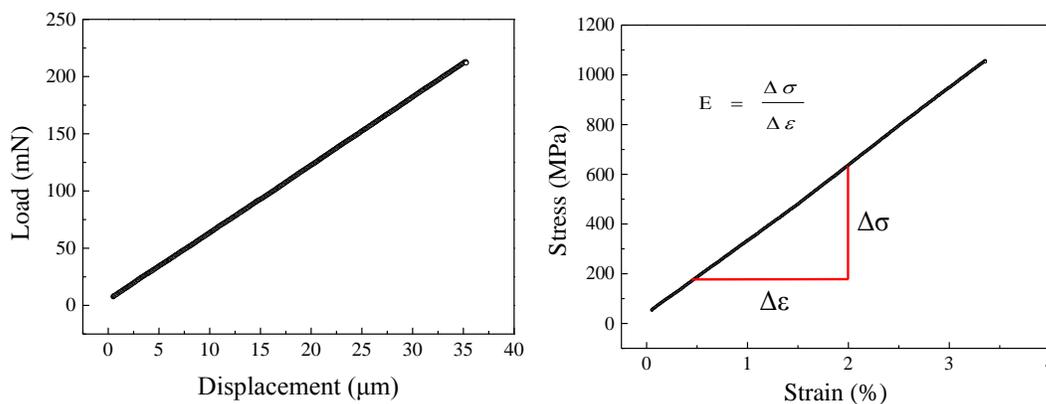


Fig. 4. Load-displacement and stress-strain curves

Ultimate load, tensile strength, MOE, and elongation of bamboo single fibres are also summarized in Table 1. The bamboo fibres exhibited a large range of values for ultimate load, tensile strength, and MOE. This variability might be explained by the morphological and structural parameters of the fibres. For example, the diameter and lumen sizes of the tested fibres ranged from 3.98 to 22.75 µm and 0.856 to 11.296 µm, respectively.

In general, many parameters influence the mechanical properties of natural fibres, such as, surrounding growth conditions and testing conditions (humidity, temperature), position of the fibre in the stem (Duval *et al.* 2011), fibre diameter and lumen size (Placet *et al.* 2012), and fibre MFA (Page and El-Hosseiny 1983). The simultaneous effects of the multiple parameters could explain the large variation of the mechanical properties observed within one individual bamboo culm.

The tensile strength of bamboo fibres measured was found to range from 0.52 to 1.92 GPa, with an average value of 1.06, which is less than the value of moso bamboo (0.85 to 2.94 GPa). At the same time, the tensile elastic modulus ranged from 10.91 to 54.63 GPa with an average value of 26.93 GPa, which is close to the value of moso bamboo (20.34 to 60.01 GPa). The elongation of *D. farinosus* ranged from 2.23% to 6.49%, which is also slightly smaller than the values observed for moso bamboo.

Table 1. Statistical Results from Tensile Test of Single Bamboo fibre

	Tensile span (mm)	Cross-sectional area (µm ²)	Ultimate load (mN)	Strength (GPa)	MOE (GPa)	Elongation (%)
Average	0.87	175.23	177.87	1.06	26.93	3.84
CV (%)	26	29	32	29	29	26
N	417	394	337	330	330	342

CV: coefficient of variability; N: valid numbers of sample

Effect of Bamboo Age on Tensile Properties of Single Fibre

Figure 5 gives a quantitative description of the effect of ages from 2 to 5 years old on the tensile strength, modulus, and elongation of bamboo fibres, respectively.

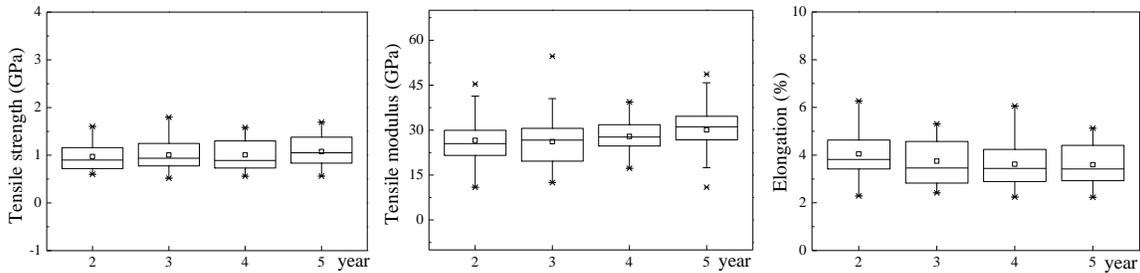


Fig. 5. Trends of tensile properties of single fibre with bamboo age

The figure clearly shows that tensile strength, MOE, and elongation varied slightly with bamboo age. The highest tensile strength and MOE were observed in 5-year-old culms, while the lower tensile strength and MOE values were found in younger culms. Similar variation also was noted by Cao (2010) for moso bamboo, in which the mean values of tensile strength were observed to increase with age from 2 to 6 years old and tensile MOE increased with age from 2 to 4 years old.

For *D. farinosus*, although the tensile strength and MOE respectively increased by 10.3% and 13.3% in 5 years old compared to 2 years old culms, no statistically significant difference could be confirmed from the results of analysis of variance (ANVOA) between all four ages for the tensile strength ($F = 0.644$, $df = 3$, $p > 0.05$), MOE ($F = 2.033$, $df = 3$, $p > 0.05$), and elongation data ($F = 1.994$, $df = 3$, $p > 0.05$). The tensile properties were found to reach a near optimal state at 2 years old. The result may be related to the finding that by the end of the first growing season, lignification of bamboo is already completed and fibres reach relatively constant mechanical properties values that alter little over time (Yu *et al.* 2011a).

Longitudinal Variation in Tensile Properties of Bamboo Single Fibre

To analyze the relationship between fibre tensile properties and the culm height position, 4-year-old *D. farinosus* bamboo culms were chosen. The statistical results of tensile strength, MOE, and elongation at different height positions are shown in Fig. 6.

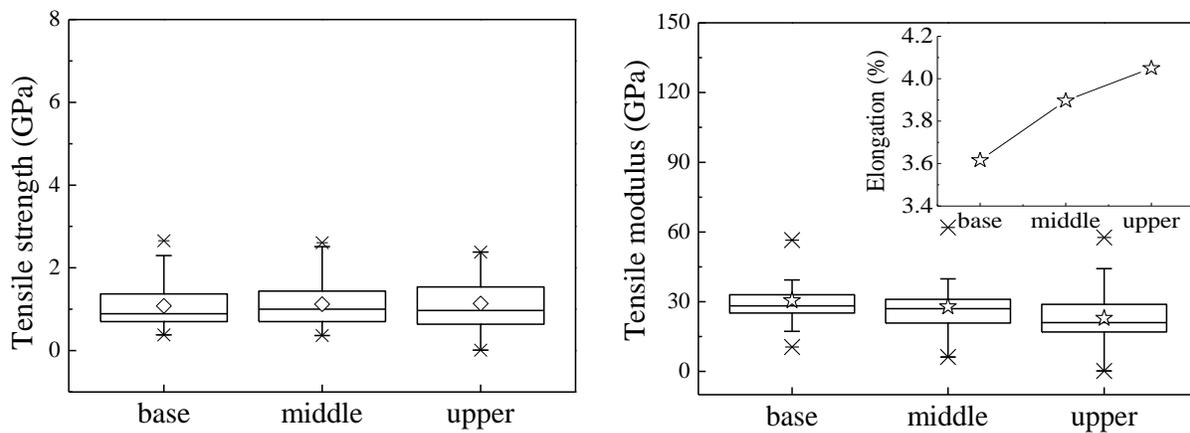


Fig. 6. Trends of tensile properties of bamboo fibres along culm height

In this work, the average tensile strength was concentrated in the range of 1.00 to 1.12 GPa, showing little deviation among the base, the middle, and the upper positions along the culm height. A weak increasing trend of elongation of 11.9% from the base to the upper part was observed. Conversely, MOE decreased by 23.2% from the base, where it was valued at 27.86 GPa, to the upper position, where it dropped to 21.41 GPa. ANOVA results showed that the variation was highly significant along the culm height for MOE ($F = 8.825$, $df = 2$, $p < 0.01$), while there was no significant relationship for tensile strength ($F = 1.147$, $df = 2$, $p > 0.05$) and elongation ($F = 2.028$, $df = 2$, $p > 0.05$). It can be concluded that the effects of culm height position on tensile strength and MOE at fibre level are less obvious than those at blocks level.

Radial Variation in Tensile Properties of Bamboo Single fibre

Irrespective of bamboo age and height position, 4-year-old fibres from the middle height position were chosen to assess changes of tensile properties from the outer to the inner periphery along the radius, as shown in Fig. 7. Shigeyasu *et al.* (1997) indicated that the mechanical distribution in bamboo was consistent with the distribution of fibrils, which meant that a decrease of fibril density gave rise to a decrease of macro-mechanic properties from the outer to the inner radial section. But the changes in mechanical properties of single bamboo fibre may not follow the same pattern.

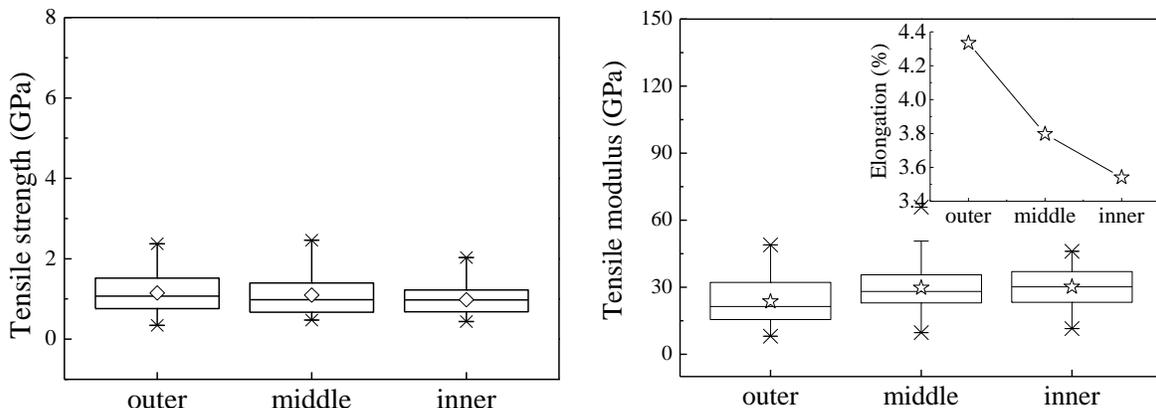


Fig. 7. Trends of tensile properties of bamboo fibres along the radius

Figure 7 shows no obvious change in strength, with a range of 1.09 to 1.24 GPa recorded. In addition, elongation exhibited a decrease of 18.2% from the outer (4.33%) to the inner position (3.54%), which means that the MOE increased slightly (by 23.2%) from the outer (24.08 GPa) to the inner section (29.66 GPa). The variation was significant along the radius for MOE ($F = 5.727$, $df = 2$, $p < 0.01$), elongation ($F = 6.744$, $df = 2$, $p < 0.01$), and tensile strength ($F = 4.128$, $df = 2$, $p < 0.05$). The effect of morphological parameters, but not the radial position, might explain the variation of these properties within one bamboo block.

CONCLUSIONS

1. It can be concluded that bamboo age has little effect on the three indicators used to characterize fibrous microtensile properties. While the culm height and radial positions were shown to have a pronounced influence on MOE indicator, they did not have much influence on the tensile strength at the fiber level. The results might be explained by the

morphology and structural parameters of the fibre itself, but are not reflected in bamboo's macro-mechanical properties because of the dependence of the latter on variance in the fibre density along different sections of the culm.

2. The average values for tensile strength and MOE of *D. farinosus* are close to those of moso bamboo, which suggests that more attention should be paid to sympodial bamboo to develop their diversified industrial utilization, as a potential supplement to moso bamboo. These properties were found to reach a near optimal state at 2 years old, after which they remained fairly uniform across the whole culm. It follows that one can use 2-year-old bamboo instead of 5- or more year-old bamboo from the tip to the base to shorten materials' cycling time and improve their utilization, when they are used in industry.

ACKNOWLEDGEMENTS

The authors are grateful for the support of Forestry Public Research Foundation (201304513). Mr. Oliver Frith of the International Network for Bamboo and Rattan (INBAR) also kindly assisted the authors with preparation of the manuscript. The constructive comments from the anonymous reviewers are greatly appreciated.

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Article submitted: July 30, 2015; Peer review completed: September 20, 2015; Revised version received and accepted: October 15, 2015; Published: December 18, 2015.

DOI: 10.15376/biores.11.1.1609-1619