Variability in the Mechanical Properties of Cell Walls of *Bambusa arundinacea* (Retz.) Willd. Based on Nanoindentation Method

Fukuan Dai, a,b Ziwei Wang, a,b Linpeng Yu, c Tuhua Zhong, a,b Hankun Wang, a,b and Genlin Tian a,b,*

*Bambusa arundinacea* (Retz.) Willd. is a large and thorny bamboo species used for construction and scaffolding purposes. Bamboo fibers, which are an important part of bamboo, give bamboo its excellent mechanical properties. The goal of this work was to get more information about fiber in order to learn more about bamboo material. It was tested for different ages, heights, and radial positions using the nanoindentation method. The results showed that the mean values of indentation modulus of elasticity and hardness of this species were 20.79 GPa and 497.01 MPa, respectively, and that the mechanical properties of the fiber cell walls were less affected by the three factors and were generally consistent. In regards to the age factor, it had little effect on the indentation modulus of elasticity, while the hardness increased slightly with age. The indentation modulus of elasticity and hardness tended to increase with increasing height. The fiber strength was relatively stable in different parts of the radial direction.

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Contact information: a: Institute of New Bamboo and Rattan Based Biomaterials, International Center for Bamboo and Rattan, Beijing 100102, China; b: Key Laboratory of National Forestry and Grassland Administration/Beijing for Bamboo & Rattan Science and Technology, Beijing 100102, China; c: School of Forestry and Landscape Architecture, Anhui Agricultural University, Hefei, Anhui 230036, China; * Corresponding author: tiangenlin@icbr.ac.cn

INTRODUCTION

Bamboo is an extremely fast-growing and ecologically valuable material, and importantly, its mechanical properties can reach a stable state in a short period of time (Zhang et al. 2020). It is widely used as a green building material, with direct bamboo culm utilization for bamboo buildings, bamboo scaffolding, etc., and is also processed into bamboo flooring and bamboo furniture (Chung and Yu 2002; Lee et al. 2012; Hong et al. 2019; Zheng and Zhu 2021). Bamboo, as a typical anisotropic material, has a tissue structure and other factors that make its mechanical properties exhibit significant differences both radially and longitudinally (Wang and Shao 2020; Liu et al. 2022). In order to further grasp the complex mechanical behavior of bamboo materials and to optimize the design of bamboo fiber-based composites, more information on fiber mechanics is needed (Yu et al. 2011).

Fibers as a reinforcing phase are distributed in a gradient structure in the basic tissue, resulting in the distinctive macroscopic properties of bamboo. Bamboo fibers are also widely used in the textile and paper industries as well as in advanced engineering.
materials (Wang et al. 2010; Guan et al. 2019; Lin et al. 2022). The excellent mechanical properties of bamboo originate from bamboo fibers. Through gaining a deeper understanding of fiber, a more thorough grasp of the properties of bamboo can be obtained (Hao et al. 2018). Fibers have thick cell walls. Characterization of the mechanical properties of small-sized cell walls is difficult to perform using conventional mechanical testing instruments. The birth of the nanoindentation technique overcame this difficulty and pioneered the study of the mechanical properties of cell walls, adding to the study of the properties of wood (Wimmer et al. 1997). Since then, the technique has become increasingly sophisticated and has been applied to the study of the fibers within the cell walls of bamboo (Dixon et al. 2015). The nanoindentation technique involves pressing a very fine diamond indenter needle into the surface of the object to be measured, continuously collecting the load during loading and unloading as well as the depth of indentation, and finally obtaining the hardness and indentation modulus of the sample to be measured through a specific theoretical model. Since the radius of curvature of the indenter is generally less than 100 nm, much smaller than the thickness of the bamboo fiber cell wall, it is well suited for the study of cell wall properties. *Bambusa arundinacea* (Retz.) Willd. is a large and spiny bamboo with poles 10 to 17 m high, 6 to 9 cm in diameter, 10 to 23 cm long between nodes, and 1 to 1.5 cm thick. It has a straight, thick, and solid culm that can be used for construction and scaffolding. This species is native to India and is now cultivated in Guangzhou and Hong Kong, China. Previous studies on the root distribution, phytohormones, and medicinal value of *Bambusa arundinacea* (Retz.) Willd. have been published (Joshi and Nadgauda 1997; Divakara et al. 2001; Kumar and Divakara 2001; Venkatachalam et al. 2015; Patil et al. 2023). As a construction material, there is little research on its mechanical properties. Bamboo is a biomass material with natural functional gradient (Habibi et al. 2015), which is anisotropic and heterogeneity in nature, resulting in macroscopic mechanical properties that exhibit variability and anisotropy (Ahmad and Kamke 2005; Hu et al. 2018). This work addresses whether there is variability in the fibers of different parts. Therefore, this study investigated the mechanical properties of individual fibers within the bamboo cell wall in terms of age, radial, and axial direction.

**EXPERIMENTAL**

**Materials**

The vascular bundle type of *Bambusa arundinacea* (Retz.) Willd. is mainly broken-waist type with sporadic distribution of double broken-waist type (Fig. 1). Samples were collected from the Hua’an Bamboo Garden in Zhangzhou City, Fujian Province, China, a region with a subtropical monsoon climate (Fig. 2a). Randomly selected healthy two-, three-, and four-year-old normal-growth bamboos were felled, and sections at heights of 1.5 m, 7.0 m, and 12.5 m were intercepted for the base, middle, and top specimens (Fig. 2b), respectively. The middle wall of a 1.5 m high bamboo section of two-, three-, and four-year-old bamboos was used to study age variation. Different wall layers along the radial section of the 1.5 m high bamboo segment of three-year-old bamboo were used to study radial variation. The middle walls of bamboo sections at 1.5 m, 7.0 m, and 12.5 m height of three-year-old bamboo were used to study the longitudinal variation.
Fig. 1. Transverse section and vascular bundle types of *Bambusa arundinacea* (Retz.) Willd.

**Nanoindentation Test**

The bamboo section with the fibers to be tested was first prepared into a rectangle, and the fibers to be tested were located in the center of the rectangle end face (Fig. 2c). Then, the end face of the rectangle was prepared into a pyramid shape using a slicer, and the tip of the tower was the fiber to be tested (Fig. 2d). The top was polished with a diamond blade to produce a nanoindentation test sample.

Fig. 2. a: Sample collection, preparation, and testing *Bambusa arundinacea* (Retz.) Willd. in living condition; b: Cut off bamboo segments; c: Rectangular specimens containing fibers of interest; d: Pyramid-shaped specimens on an iron stand; e: Testing using nanoindentation equipment

The prepared samples were equilibrated in a sealed chamber for more than 24 h at a temperature of 23 ± 5 °C and a relative humidity of 40 ± 0.5% (Li *et al.* 2020). The test apparatus was a Triboindenter (Hysitron, Minneapolis, MN, USA) with a Berkovich diamond tip (diameter of the tip ≤ 100 nm) (Fig. 2e). A three-stage segmental application of load to the fiber of interest in force control mode was selected. The set nanoindentation test procedure is shown in Fig. 3, first loading at 50 μN·s⁻¹, loading for 5 s to reach a maximum load of 250 μN, and the maximum load was held for 6 s. The holding load was for viscoelastic materials to avoid experimental errors due to creep; finally, after a 3 s unloading segment, and collecting data from the unloading segment for computational
analysis. The indentation modulus of elasticity and hardness of the samples were calculated using the equations of Oliver and Pharr (1992), based on specific data and procedures described by Eder et al. (2013) and Yan et al. (2011),

\[
1 / E_r = (1 - \nu^2) / E + (1 - \nu_i^2) / E_i
\]

\[
H = P / A_c
\]

where \(H\) is hardness; \(P\) is peak load; \(A_c\) is the projected area at peak load; \(E_r\) is the composite response modulus, which can be calculated based on the load-displacement curve and elastic contact theory; \(E_i\) and \(\nu_i\) are the elastic modulus and Poisson ratio of the tips, respectively. For diamond tips, \(E_i\) is 1141 GPa, and \(\nu_i\) is 0.07. \(E\) and \(\nu\) are, respectively, the indentation modulus of elasticity and Poisson ratio of samples, where the Poisson ratio of samples is 0.22 (Yu et al. 2011; Yuan et al. 2021).

Fig. 3. a: Load-Time control schematic (loading section, loading holding section, and unloading section in that order); b: Typical force-displacement curves (corresponding in turn to the three loading processes in Fig. 3.a)

**RESULTS AND DISCUSSION**

**Strength of Fibers of Different Ages**

Figure 4 illustrates the strength of the fibers in the middle of the bamboo wall of 1.5-m-high sections of two-, three-, and four-year-old bamboos. Overall, the indentation modulus of elasticity of the fibers of the different ages of the bamboos did not differ greatly in value at around 20.5 GPa. The indentation modulus of elasticity of the four-year-old fibers was the highest, with a mean value of 21.4 GPa. The stiffness of the fiber cell wall showed a small increase with age. The hardness of two-year-old bamboo fibers was the smallest at 441 MPa, while the hardness of four-year-old bamboo fibers increased to 536 MPa. It has been shown that for wood science, microfibril angle, cellulose rate, and density are considered to be the main determinants of mechanical properties (Evans and Elic 2001). The cell wall density and cellulose proportional content of mature wood fibers are almost the same, so the microfibril angle is the most important factor affecting the properties for mature wood fibers (Evans and Elic 2001). There is a negative correlation between the microfibril angle and two mechanical properties of the cell wall, which are the indentation
modulus of elasticity and hardness. That is, the indentation modulus of elasticity and hardness decreases with increasing microfibril angle (Tian et al. 2010). The difference in microfibril angle between bamboo fibers of different ages was not significant (Liu et al. 2014). Therefore, the age factor had little effect on fiber cell wall strength.

![Graph showing the indentation modulus of elasticity (IMOE) and hardness of cell walls of fibers of different ages over time.](image1)

**Fig. 4.** Indentation modulus of elasticity (IMOE) and hardness of cell walls of fibers of different ages

**Variability of the Strength of the Fibers in the Height Direction**

The nanoindentation data were analyzed for the middle part of the three-year-old bamboo wall at different heights (Fig. 5). The indentation modulus of elasticity and hardness increased with height in a relatively smooth trend. The indentation modulus of elasticity and hardness of the top fiber cell wall were 21.9 GPa and 550 MPa, respectively, which were slightly higher than the strength of the other two parts. The age factor had little effect on the microfibril angle, but in the height direction, the average microfibril angle decreased with increasing height of bamboo culms, which may make the cell wall strength increase with increasing height (Zhang et al. 2020).

![Graph showing the variability of indentation modulus of elasticity and hardness of cell walls of fibers in the height direction.](image2)

**Fig. 5.** Variability of indentation modulus of elasticity and hardness of cell walls of fibers in the height direction
Variability of the Strength of Fibers in the Radial Direction of the Bamboo Wall

Figure 6 clearly shows the trend of the fiber cell wall strength from the outer skin to the inner skin. Both the indentation modulus of elasticity and the hardness were almost unchanged, with only small fluctuations that did not produce significant differences. In general, the cell wall strength was stable from the outside to the inside of the bamboo wall, with 95% confidence intervals for the mean values of the indentation modulus of elasticity ranging from 20.2 to 20.5 GPa. Similarly, 95% confidence intervals for the mean values of the hardness ranged from 464.7 to 477.5 MPa. The strength of the fiber cell wall remained almost constant along the radial direction of the bamboo wall, probably because the microfibril angle along the radial direction was almost constant (Yu et al. 2007).

The mechanical properties of the cell walls of the fibres of Bambusa arundinacea (Retz.) Willd. were found to be somewhat lower than those of the more widely used fibres of moso bamboo (21.69 GPa and 0.61 GPa in compressive modulus of elasticity and hardness, respectively) (Huang and Fei 2017). However, Dendrocalamus farinosus, which is also a clumping bamboo, has an indentation modulus and hardness of only 18.56 GPa and 410.72 MPa respectively, which are lower than those of Bambusa arundinacea (Retz.) Willd. (Yang et al. 2014). This shows that from the point of view of the mechanical properties of the cell wall, the fibres of Bambusa arundinacea (Retz.) Willd. have great potential for industrial use.

CONCLUSIONS

1. The mechanical properties of fibrous cell walls of Indian cambium were tested by nanoindentation method at different ages, heights, and radially superior sites. The mean values of indentation modulus and hardness for all specimens of Bambusa arundinacea (Retz.) Willd. were 20.8 GPa and 497 MPa, respectively, and the results indicated that the three factors studied had little effect on the mechanical properties of the fiber cell walls.
2. The age factor had little effect on the indentation modulus of elasticity, while the hardness increased slightly with age. The indentation modulus and hardness tended to increase with increasing height. The strength of the fibers was relatively stable in different parts of the radial direction.

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**Author Contributions Statement**

F. D. wrote the main manuscript, Z. W. was responsible for data curation, L. Y. performed formal analysis, T. Z. supplied the resources, H. W. came up with the methodology, G. T. was responsible for funding acquisition, methodology, and project administration. All authors reviewed the manuscript.

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

**Data Availability Statements**

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

**REFERENCES CITED**


