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Mechanical properties of bamboo fiber cell walls during the culm development by nanoindentation

Yanhui Huang[a,*], Benhua Fei[b], Penglian Wei[b], Chang Zhao[a]

[a] Key Laboratory of Wooden Material Science and Application, Ministry of Education, Beijing Forestry University, 100083 Beijing, China
[b] International Center for Bamboo and Rattan, 100102 Beijing, China

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ABSTRACT

Bamboo fibers have excellent mechanical properties, and as such are a popular raw material in papermaking, textile, and various high-tech industries. In this study, the longitudinal mechanical properties of Moso bamboo (*Phyllostachys Heterocycla* Var. Pubescens) fiber cell walls during development were systematically investigated via nano-indentation technique at the sub-cellular level. Several influencing factors were also analyzed, particularly the effects of different locations within the vascular bundle and age. The results showed that the longitudinal nano-indentationmodulus (NI modulus) of the 1-month-old bamboo fiber cell walls was relatively high and changed very little in different locations of the vascular bundle, only fluctuating near 22 GPa, while that at the interface between fibers and parenchyma cells (i.e., the edge of the vascular bundle) was smaller and less stable (15.61 GPa on average); nano-indentation hardness (NI hardness) decreased from the center of the vascular bundle to the outside, ranging between 0.4665 and 0.5603 GPa. Age had no significant effect on NI modulus from 1 month to 36 months, while NI hardness did increase with age ($p < 0.05$), showing mean values of 0.5452–0.6142 GPa across the samples. Our observations of longitudinal NI modulus and hardness were mainly affected by the microfibril angles and lignin content, respectively. These results may provide mechanical evidence for bamboo growth and lignin deposition, and also provide a scientific basis for the successful utilization of bamboo fibers.

1. Introduction

Bamboo fibers have unique and favorable properties, and are readily available, renewable, and environmentally friendly; as of now, they already are commonly utilized in papermaking, textile, and a variety of advanced tech industries including composite and nano-material manufacturing (Takagi et al., 2003; Abdul Khalil et al., 2012). As a constituent or reinforcing phase of materials, the mechanical properties of bamboo fibers inextricably affect the final performance of the products containing them. To this effect, it is very important to appropriately select and properly utilize bamboo fibers to ensure high-performing products (Okubo et al., 2004).

Moso bamboo (*Phyllostachys Heterocycla* Var. Pubescens) is an important bamboo species in terms of material production. Both its stand volume and cultivated area are among the highest in the world. The cell wall structure of Moso bamboo fibers is quite complex, as shown in Fig. 1, where the outermost layer of the cell wall is the primary wall *P*, and its microfibril arrangement is reticular and disordered; below the outer layer is secondary wall *S*, which is comprised of up to nine (occasionally more) alternately repeated thick, micro-scale layers and thin, nano-scale layers. The orientations of thick-layer microfibrils are mostly along the fiber axis, where the microfibril angle (MFA) shows an increasing trend from the intercellular layer to the cell lumen; in thin layer, the orientations of microfibrils are basically perpendicular to the fiber axis. The thin layers have significantly higher lignin content than the thick layers and accordingly exhibit more lignification (Parameswaran and Liese, 1976, 1980; Liese 1998; Crow and Murphy, 2000).

Typically, bamboo fibers are smaller than other plant fibers with diameters less than 15 μm and lengths no more than 2 mm, also featuring micro- or nano-natural characteristics on the cell walls such as pits, uneven microfibril arrangement, and complex layer structures. These characteristics make it rather difficult to quantify the micro- or nano-mechanical properties of the fibers (Huang et al., 2012). Prior to the emergence of nano-indentation techniques, the micro- or nano-mechanical properties were quantified through traditional macro-mechanics or by mechanical testing of the single fibers.

The longitudinal NI modulus and hardness of mature bamboo fiber cell walls were first obtained by Yu et al. (2007) via an in situ

* Corresponding author.
E-mail address: huangyanhui@bjfu.edu.cn (Y. Huang).

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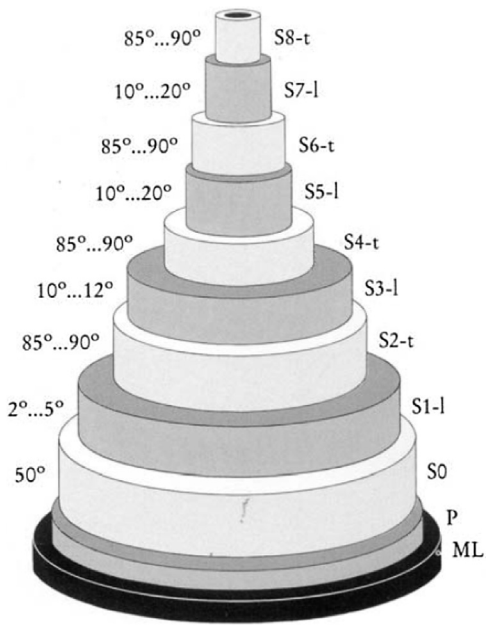

Fig. 1. Structure of bamboo fiber cell wall(Liese, 1998).

nano-indentation imaging technique. Then, Zou et al. (2009) also successfully defined the mechanical properties of mature bamboo fiber cell walls through nano-indentation testing. However, their results departed considerably from those published prior due to restricted sample preparation techniques and test conditions. With the development of research, Wang et al. (2012) found that the favorable mechanical properties of bamboo were due to the orientation of microfibrils from bamboo fiber cell walls laying basically along the fiber axis, which maximizes the longitudinal NI modulus of the cell wall, while lignification enhances the transverse stiffness of the fibers. The same research team also compared the cell wall mechanical properties of bamboo fibers with those of wood fibers to confirm that the former is superior mechanically (Wang et al., 2014). The influence factors of mechanical properties of bamboo fiber cell walls were also studied, such as moist, heat treatment, age, and so on. For example, there were a negative correlation between the longitudinal NI modulus and hardness of mature bamboo fiber cell walls and moisture (Wang, 2010); the heat treatment temperature had important influences on the mechanical properties of mature bamboo fiber cell walls (Li et al., 2015). For age, Liu (2008) preliminarily tested the mechanical properties of 17-day- and 4-year-old bamboo fiber cell walls, and confirmed that the mechanical properties of the cell walls were affected by the development process (though the related age choosing and measurement data was extremely limited).

Compared to wood fiber cell walls, extant research on mechanical properties of bamboo fiber cell walls is very limited, especially in terms of the growth development of bamboo fibers. These development-related cell wall mechanical characteristics have important theoretical and practical significance, however, as they can provide mechanical evidence for the growth and development of bamboo and lignin deposition; research on the subject also can create quantitative objectives and indexes for the selection and utilization of bamboo fibers, as well as the genetic modification and oriented cultivation of bamboo. In this study, we systematically investigated the mechanical properties of bamboo fiber cell walls during growth development via nano-indentation, and analyzed the impact of age and different locations in the 1-month-old vascular bundle on the mechanical properties.

2. Materials and methods

2.1. Materials

Bamboo was collected and felled from Miaoshanwu Tree Farm, Fuyang City, Zhejiang Province, China (120.02′E, 30.06′N), which is located in the north of China's subtropical region at 50–536.9 m altitude. The soil in the area is red, having been developed by arkose or quartz sandstone. The annual average temperature is 16–17 °C, and the annual rainfall is 1200–1700 mm (mostly occurring during the 5–6 month rainy season.)

The selected Moso bamboos were 1, 2, 6, 18, and 36 months old; all were straight and growing well at the time (in 2008) they were harvested. The bamboos were cut into 2 m sections and air dried prior to analysis.

2.2. Methods

2.2.1. nano-indentation

The nano-indentation technique is an effective method for measuring the mechanical properties of biomass materials. These mechanical properties, including NI modulus, NI hardness, yield strength, and creep, can be obtained by calculating the load-displacement curve derived from nano-indenter loading and unloading on the materials (Wang et al., 2006).

The theoretical calculation method most commonly adopted for nano-indentation is the Oliver-Pharr method (O & P method). For more detail on this method, interested readers can refer to our Works Cited (Oliver and Pharr, 1992).

Under O & P methodology, NI hardness (H) is defined by the following equation:

$$H = P_{max} / A \tag{1}$$

The sample NI modulus (E) can be calculated as follows:

$$1 / E_r = (1-\nu^2) / E + (1-\nu_i^2) / E_i \tag{2}$$

where P_{max} is the peak load and A is the projected contact area, which can be calculated from the empirical formula 24.5 h_c^2, where h_c is the contact depth of the indent. E_r is the composite response modulus, which can be calculated according to the load-displacement curve and elastic contact theory. E_i and ν_i are, respectively, the elasticity modulus and Poisson ratio of the tips. For Berkovich tips, E_i is 1141 GPa, and ν_i is 0.07. To calculate E (NI modulus of the sample), the Poisson ratio of sample must be known in the current direction. Generally, the elasticity modulus is not sensitive to the Poisson ratio value; the bamboo cell wall is much softer than diamond, of course, so the effect of the samples' Poisson ratio was negligible.

2.2.2. Sample preparation

When applying the nano-indentation technique to test the mechanical properties of cell walls, appropriate sample preparation is crucial. A nominal 20-mm-long cylinder was cut from halfway between adjacent bamboo nodes at about 2 m height immediately after harvesting each age of bamboo, then a 1 mm wide (tangential) × 1 mm thick (radial) stick was cut from the cylinder with the distance between the stick and the outer surface of the bamboo as 1 mm in radius (Fig. 2). At least one complete vascular bundle was contained in each stick. All 1 × 1 × 20 mm (radial * tangential * longitudinal) sticks were conditioned for at least 24 h at 22 °C and 40% relative humidity in the nano-indenter testing chamber, then embedded in Spurr resin (Spurr, 1969). The embedding was performed in a flat mould with the specimen in the middle of the flat surface, then the mould containing the samples and the resin was placed in a vacuum oven. After being kept in a vacuum for a minimum of 12 h, the embedded samples were heated to 70 °C for 8 h