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Mechanical characterization of single bamboo fibers with nanoindentation and microtensile technique

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Abstract

More mechanical information on fibers is needed for better understanding of the complex mechanical behavior of bamboo as well as optimizing design of bamboo fiber based composites. In this paper, *in situ* imaging nanoindentation and an improved microtensile technique were jointly used to characterize the longitudinal mechanical behavior of fibers of Moso bamboo (*Phyllostachys pubescens* Mazei ex H. de Lebaie) aged between 0.5 and 4 years. These methods show that 0.5-year-old fibers have similar mechanical performances to their older counterparts. The average longitudinal tensile modulus and tensile strength of Moso bamboo fibers ranges from 32 to 34.6 GPa and 1.43 to 1.69 GPa, respectively, significantly higher than nearly all the published data for wood fibers. This finding could be attributed to the microstructural characteristics of the small microfibrillar angle and scarcity of pits in bamboo fibers. Furthermore, our results directly support the assumption that the widely used Oliver-Pharr analysis method in nanoindentation test significantly underestimates the longitudinal elastic modulus of anisotropic plant cell wall.

Keywords: bamboo fibers; *in situ* nanoindentation; mechanical properties; microtension.

Introduction

It has been well-recognized that the extraordinary mechanical performances of bamboo mainly originate from its fiber components (Amada and Untao 2001; Lo et al. 2004). In addition, reinforced composites from bamboo fibers have been the focus of intensive attention in the field of materials science. Microtension and nanoindentation of single fibers belong to the powerful tools for mechanical characterization of plant fibers. In this context, the pioneer work of Jayne (1959) should be pointed out. Since then, the emphasis was on the improvement of the accuracy and efficiency of this method (Page et al. 1971; Burgert et al. 2002, 2003; Groom et al. 2002a). The major challenges of this method are fiber gripping, alignment of fibers to tensile direction, and determination of the cell wall area of single broken fibers.

Nanoindentation, a method of hardness and modulus testing at the micrometer level or even at the nanometer scale, is increasingly applied in the mechanical characterization of plant fibers. Wimmer et al. (1997) and Wimmer and Lucas (1997) first introduced nanoindentation in the field of wood science by estimating the mechanical properties of the secondary wall and the cell corner middle lamella of spruce tracheids. The subsequent investigations conducted by Gindl and Gupta (2002a) and Gindl et al. (2004) focused on microfibril angle (MFA) and lignification related to longitudinal hardness and elastic modulus of the secondary cell wall of softwood tracheids. Recent publications demonstrate that there are many applications for nanoindentation in the field of wood research, such as paint films on wood products (Jiang et al. 2006), bonding interface of wood-adhesive (Konnerth and Gindl 2006; Konnerth et al. 2007; Konnerth and Gindl 2008; Follrich et al. 2010; Stöckel et al. 2010), wood modification (Gindl and Gupta 2002b; Stanzl-Tschegg et al. 2009; Konnerth et al. 2010), wood fiber polymer composites (Gindl et al. 2006; Lee et al. 2007a), creep behavior of lyocell fibers (Lee et al. 2007b), and thermo-mechanical refining (Xing et al. 2008). However, it was suggested based on a theoretical calculation that the longitudinal elastic modulus of wood cell wall measured by nanoindentation might be seriously underestimated (Gindl and Schoberl 2004). Furthermore, nanoindentation does not permit the calculation of the ultimate tensile strength.

To obtain a better and more comprehensive understanding of the mechanical behavior of bamboo fibers, in the present study nanoindentation and an improved technique of microtension of single fiber were jointly investigated. Such investigations are scarce (Lee et al. 2007b). The working hypothesis was that the former can be calibrated by the latter. Thus, the aim of this study was to investigate the experimental evaluation of the extent of underestimation in modulus by nanoindentation compared to traditional tensile testing.

Materials and methods

Sample preparation

Moso bamboo, approximately 0.5, 2, and 4 years old, was taken from a bamboo plantation located in Zhejiang Province, China. Cubic blocks were cut out from three heights (2 m, 4 m, and 6 m) of a bamboo culm and then split into sticks measuring approximately 1(R)×1(T)×15(L) mm^3. For nanoindentation, three sticks were evenly selected along the radial direction of culm wall at every height, and 27 sticks were collected and tested in total. The procedure of sample preparation for nanoindentation was similar to that proposed by Wimmer et al. (1997). In brief, bamboo sticks were embedded in Spurr resin and cured in a plastic mold. After curing, the cross-section of samples was cut with an ultramicrotome equipped with a diamond knife to obtain a very smooth surface for indenting.

The matched bamboo sticks was macerated in a soft solution comprising one part 30% H_2O_2, four parts distilled water, and five parts glacial acetic acid for 24 h. Subsequently, fibers were washed several times in distilled water and dried on glass slides at room temperature. Twisting of bamboo fibers during drying is negligible due to the small MFA as well as the geometrical feature of the extremely large ratio of cell wall to cavity. The widely accepted methodology of ''ball and socket'' type fiber gripping was adopted for microtensile testing. Two epoxy droplets approximately 200 μm in diameter were placed in the center portion of each fiber with an approximate spacing of 0.7–0.8 mm via a fine tweezers. 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 can be found in the paper by Groom et al. (2002a).

In situ imaging nanoindentation testing

Nanoindentation was developed from conventional impression hardness testing, but it gives rise to much higher resolution both in load and depth measurement. During a nanoindentation test, the load and indentation depth are continuously recorded from loading to unloading. Hardness and elastic modulus can be inferred from the pure elastic unloading segment of the load-depth trace based on the most widely accepted method developed by Oliver and Pharr (1992).

Instrument: Triboindenter (Hysitron Incorporation, USA). A Berkovich diamond tip with radius less than 100 nm was selected for indenting. The target peak load and loading-unloading rate was 250 μN and 50 μN s^{-1}, respectively. The hold time at peak load was 6 s. During the test period, the relative humidity (RH) in the chamber of the instrument ranged from 15% to 37% (changing with the environmental humidity), but the temperature was kept between 22.2°C and 24.1°C. In total, 200–250 indents were made for each bamboo age.

The reduced elastic modulus E_r can be obtained from Eq. (1). Then the MOE and hardness of materials can be calculated from Eqs. (2) and (3):

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \tag{1}$$

$$\frac{1}{E_r} = \frac{1-\upsilon^2}{E} + \frac{1-\upsilon_i^2}{E_i} \tag{2}$$

$$\mathrm{H} = \frac{P}{A_\mathrm{c}} \tag{3}$$

where E_i and υ_i are, respectively, the elastic modulus and Poisson ratio of the tips. For diamond tips, E_i is 1141 GPa, and υ_i is 0.07. E and υ are, respectively, the elastic modulus and Poisson ratio of samples. Although there is no experimental data υ for the cell wall of bamboo or wood fibers presently, a value of 0.4–0.45 has been adopted for wood cell wall (Wimmer et al. 1997; Gindl and Gupta 2002b). However, we calculated ν based on the ply structure and chemical composition of wood cell wall and a ν value of 0.2 was obtained (Yu 2003). This value is very close to that of 0.25 adopted by a recently published paper (Wu et al. 2010). Therefore, an average value of 0.22 was applied here as the Poisson ratio of bamboo fibers.

Microtension of single bamboo fibers

A custom-built fiber gripping system was specially developed and combined to a small commercial high resolution mechanical tester (Instron Microtester 5848, USA) to measure the mechanical properties of short single plant fibers such as bamboo (with length more than 1.2 mm and diameter larger than 7 μm) (Figure 1a and b). The key part of the fiber gripping system is a pair of patented fiber grips that can grasp the epoxy droplets at the ends of bamboo fibers during tension (Figure 1c). The position of grips could be finely adjusted in X, Y, and Z direction through an integrated 3D micro-adjustment stage by means of a horizontal and a vertical digital microscope; so it was ensured that the tensile direction is aligned exactly with the fiber axis. The initial length of fibers can be 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 and a constant speed of 48 μm min^{-1}. In total, 76–100 fibers were tested for each bamboo age. Tensile testing was carried out under an environment of 23°C and at 15–35% RH.

To calculate the tensile strength and modulus of bamboo fibers, the cell wall areas of every broken fiber were determined with a

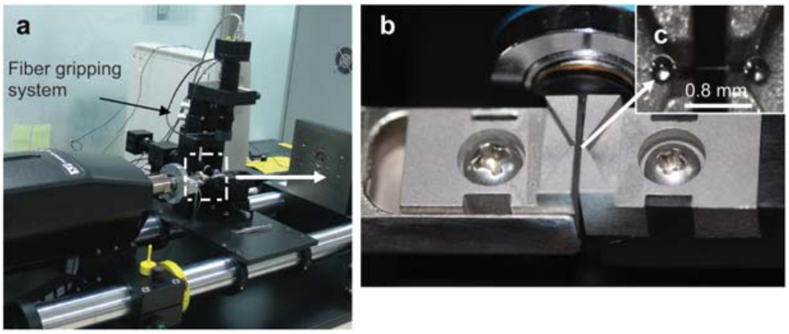

Figure 1 (a) Instron 5848 microtester combined with a specially developed fiber gripping system for fiber mechanical testing. (b) Patented fiber grips. (c) Bamboo fiber with epoxy resin droplets formed at its both ends was installed in the grips.

confocal scanning laser microscope (Meta 510 CSLM, Zeiss). The broken fibers were firstly 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 cell wall area of the fibers was then measured with software provided by the instrument producer. Load-elongation curves can be converted to stress-strain curves and the tensile strength and modulus can be obtained based on the cell wall area and the initial span length.

Results and discussions

Nanoindentation results

Figure 2 describes the procedure of an *in situ* imaging nanoindentation test on bamboo fibers. A sample holder, which is actually a modified holder of the ultramicrotome used for sample polishing, was used to keep the polished surface of sample vertical to the indenting direction as good as possible (Figure 2a). This sample holder makes adhesive unnecessary during sample installation. A target region was firstly selected under a light microscope integrated to the instrument (Figure 2b). The indenter tip was then used to image the bamboo fiber cell wall, from which the locations to be indented were carefully selected (Figure 2c). The indentation impression was also imaged with the same tip (Figure 2d). *In situ* imaging verifies that the indentations are performed in the anticipated locations and this improves the reliability of the data and helps in the explanation of unexpected test results. This function is particularly important for the reliable mechanical characterization of heterogeneous biomaterials such as bamboo and wood.

Figure 3a shows the elastic modulus of bamboo fibers measured with nanoindentation. No statistically significant difference was found with changing bamboo ages (analysis of variance, $P<0.01$). Because the longitudinal elastic modulus of plant cell wall is highly dependent on MFA (Cave 1969; Page et al. 1977), it can be inferred that the MFA of bamboo fibers should vary little with age, which has actually been demonstrated in one of our previous papers (Yu et al. 2007a). The elastic modulus measured was almost double of the value 10.4 GPa obtained by Zou et al. (2009) and higher than our previously reported value of 16.01 GPa (Yu et al. 2007b) for the same bamboo species. We attributed this discrepancy to the different methods adopted for sample preparation. In the quoted papers, sliding microtomes equipped with steel knives were applied to cut the surface of bamboo, whereas an ultramicrotome combined with ultra sharp diamond knife was applied here to reduce significantly surface damages and roughness during sample preparation. Furthermore, the elastic moduli measured here are a little higher than that of mature wood cell wall with similar MFA, but the values are significantly larger than that of juvenile cell wall with large MFA (Gindl and Gupta 2002a; Gindl and Schoberl 2004; Tze et al. 2007).

The hardness of bamboo fibers determined by nanoindentation is presented in Figure 3b as a function of age. Hardness is an important parameter also in context of the permanent plastic deformation of materials. The definition of hardness obtained in a nanoindentation test is somewhat different from that of conventional Brinell hardness. In the first case, the hardness is found by dividing the indenter peak load by the projected area under contact, which can be calculated from the penetration depth of indenter and the known

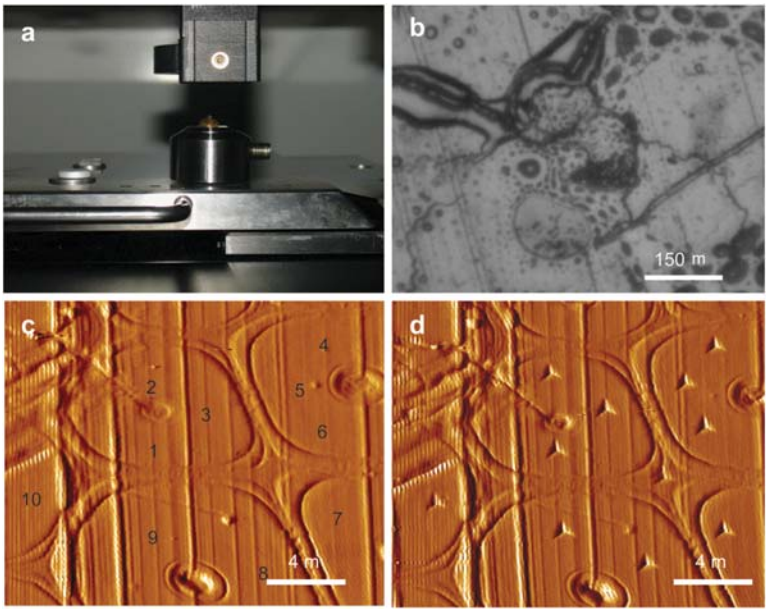

Figure 2 The procedure of an *in situ* imaging nanoindentation test on bamboo fibers. (a) A custom built sample holder for nanoindentation testing. (b) A region of interest selected from the light microscope image. (c) High magnification image of the same area obtained with indenter tips before indenting. Arabic numbers indicate the locations of indents. (d) The image obtained after indenting shows the actual position of indents.

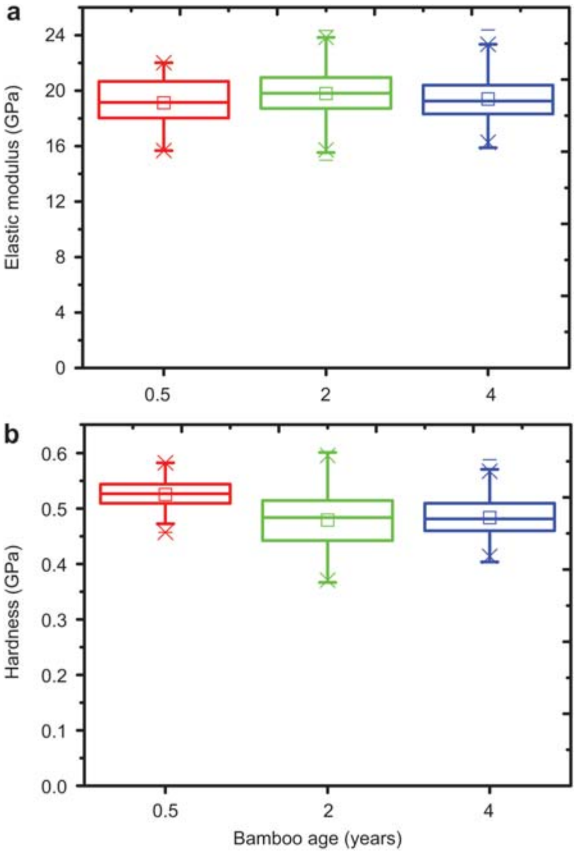

Figure 3 Box-and-whisker plots of (a) elastic modulus and (b) hardness of bamboo fibers with different ages measured with nanoindentation.

area function of the indenter tip as shown in Eq. (3). The Brinell hardness is calculated by dividing the peak load by the residual impression of an indenter after it was withdrawn from the surface. Usually, the area given by the shape of the residual impression and that given by nanoindentation are almost identical, but this is not the case for highly elastic materials that tend to result in a very small residual impression. We have compared the area of residual impression in the cell wall of bamboo fibers measured with atomic force microscopy and the projected area of contact under peak load obtained with the Olive-Pharr method. No significant differences were found (unpublished data). Thus, nanoindentation hardness is almost equal to Brinell hardness for bamboo fibers. As demonstrated in Figure 3b, little difference was found in hardness between the 2- and 4-year-old bamboo fibers. The slightly higher hardness (0.53 GPa) of the 0.5-year-old bamboo is probably due to experimental uncertainties. Gindl and Gupta (2002a) found that the cell wall hardness of Norway spruce increases significantly with lignification, and then it becomes stable when lignification is finished. This finding is helpful for explaining our experimental results. It is known from previous investigations that lignification of bamboo is completed at the end of the first growing season (approximately 6 months) with no further lignification occurring later (Itoh 1990).

The present nanoindentation tests reveal that bamboo fibers can reach mechanical maturity both in stiffness and hardness at the age of 6 months. However, Moso bamboo normally reaches its optimal macroscopic mechanical performances after 4 years, and this age is also regarded as the best harvesting time in practice. Accordingly, the improving macroscopic mechanical properties of Moso bamboo with aging should be mainly attributed to the increase of specific density caused by cell wall thickening and not to the mechanical improvement of the fiber cell wall itself.

Although the tested bamboo fibers come from different heights of a bamboo culm, the coefficient of variability (COV) in both modulus and hardness is shown to be less than 10%, which implies that the mechanical performances of bamboo fibers are rather stable in the whole culm. This could be attributed to the small variability in MFA of fibers. Yu et al. (2007a) demonstrated that Moso bamboo fibers have a MFA variation ranging from 8° to 13° with an average value of 9°, and the corresponding COV is 6.7%.

Microtensile results

The typical stress-strain curves of single bamboo fibers in tension are presented in Figure 4. All the fibers tested exhibited a quasi linear stress-strain behavior to failure, which is somewhat different from that of wood fibers. The shape of the stress-strain curve of softwood fibers is a function of MFA (Groom et al. 2002a). 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, whereas fibers with MFA less than 20° appeared to be full linear during the test. The small variability of the MFA of Moso bamboo fibers (8°–13°) (Yu et al. 2007a,b) explains why all the bamboo fibers display linear stress-strain behavior.

Figure 5 shows the tensile modulus and strength of bamboo fibers measured by microtension of single fibers. The average tensile strength of bamboo fibers with different ages is found to range from 1.43 GPa to 1.69 GPa, whereas their elastic modulus ranges from 32 GPa to 34.6 GPa. Bamboo age has little effect on these properties, a finding which is

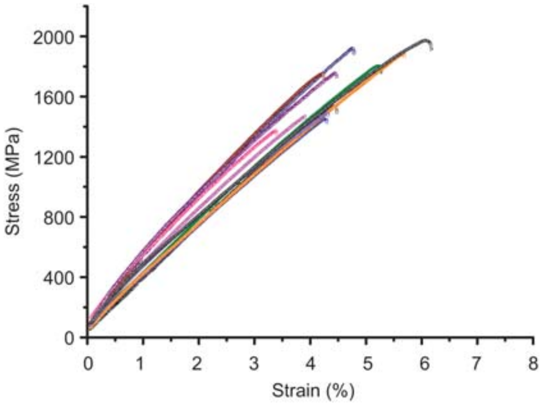

Figure 4 The typical stress-strain curves of single bamboo fibers tensioned in axial direction.

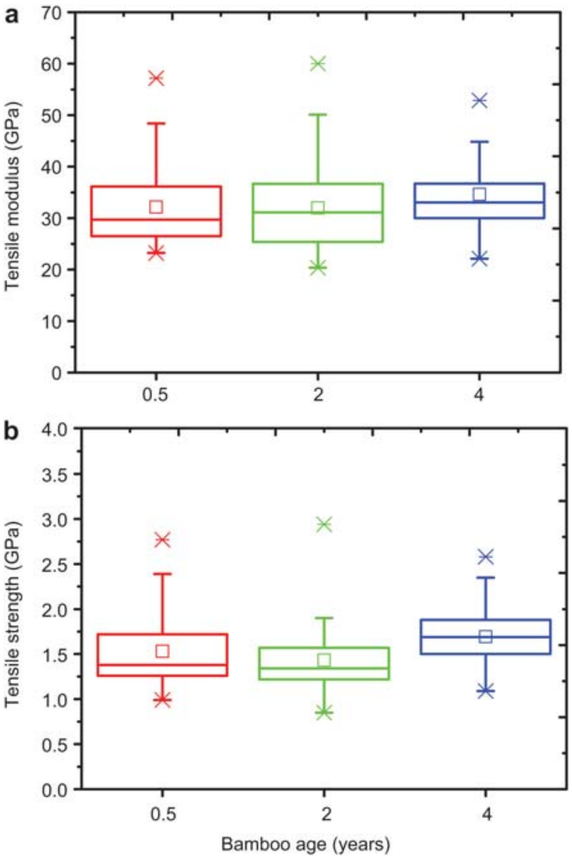

Figure 5 Box-and-whisker plots of (a) elastic modulus and (b) tensile strength of bamboo fibers with different ages measured with microtension of single fibers.

consistent with the results of nanoindentation measurements. To the best of our knowledge, no experimental data on the mechanical properties of single bamboo fibers have been reported in the literature. However, the data concerning tensile strength and modulus of bamboo fiber bundles can be found in several publications. Amada et al. (1997) found a tensile strength of 0.61 GPa and a tensile modulus of 46 GPa for bamboo fiber bundles, which were indirectly calculated based on the volume ratio of fibers to parenchymal cells and based on the macroscopic tensile modulus and strength of bamboo. Recently, Shao et al. (2010) experimentally found the tensile strength and modulus of fiber bundles of Moso bamboo to be 0.482 GPa and 33.9 GPa, respectively. Accordingly, tensile modulus measured on single bamboo fibers is comparable to that of fiber bundles, whereas the tensile strength is much higher than the latter. This is reasonable as the debonding between fibers during tension will significantly reduce the loading capacity of bamboo fiber bundles.

Although the statistical results of bamboo fibers presented in Figure 5 are based on limited numbers of tests (76–100 fibers for each bamboo age), a preliminary comparison with wood fibers can be made. Several commercially important wood species have been tested for their fiber mechanical properties. A large variability was found, dependent on the testing protocol adopted, the type of fibers (earlywood or latewood fibers), the MFA or the juvenility, etc. Generally, the earlier studies presented relatively lower values, possibly caused by the improper fiber gripping and insufficient instrument accuracy. For examples, 11.4 GPa in elastic modulus and 0.324 GPa in tensile strength for slash pine earlywood fibers (Jayne 1960), and 8.8 GPa and 0.47 GPa for Douglas-fir earlywood fibers (Hardacker 1962). Statistically reliable and precise measurements have been conducted by Groom et al. (2002a,b) and Mott et al. (2002) on loblolly pine fibers. Higher elastic modulus and tensile strength were observed on mature latewood fibers, ranging from 15.4 GPa and 0.747 GPa, and these properties were improved in the fifth growth ring to 21.1 GPa and 1.3 GPa in the 48th growth ring, respectively. The average elastic modulus and ultimate tensile strength of all loblolly pine fibers, distributed in equal proportion to earlywood and latewood, was 17.3 GPa and 0.824 GPa, respectively. Thus, Moso bamboo fibers are superior to wood fibers in terms of mechanical performances, and this finding explains the extraordinary longitudinal tensile strength of bamboo. It is widely accepted that larger MFA and pits tend to weaken the mechanical performance of plant fibers. Thus, the superior mechanical performances of bamboo fibers can easily be attributed to the scarcity and small size of pits with diameters <1 μm (Figure 6) as well as to the smaller MFA (Yu et al. 2007a).

Comparison between nanoindentation and microtension

A comparison between Figures 3a and 5a permits an important conclusion: nanoindentation significantly underestimates the longitudinal elastic modulus of bamboo fibers, with the former value being only 55% of the latter value. This type of underestimation seems to be less serious for softwood fibers. An average longitudinal modulus can be deduced from the literature for mature softwood cell walls as being 16 GPa to 22 GPa measured by nanoindentation (Wimmer et al. 1997; Gindl et al. 2004; Tze et al. 2007). These data are comparable to the more recent value of 16–23 GPa obtained by tension test of single wood fibers (Groom et al. 2002a,b; Mott et al. 2002; Burgert et al. 2003). However, Gindl et al. (2004) proposed that the longitudinal elastic modulus of wood cell walls measured by nanoindentation is only 19–24% of the theoretical value based on cell wall mechanic calculations. We assume that this huge discrepancy mainly comes from the overestimation obtained by theoretical calculations which do not account for the presence of pits in the wood cell wall. Moreover, the microtension of single wood fibers might underestimate the real mechanical performances of wood cell wall because the thin wood cell wall tends to be more seriously damaged than bamboo fibers. The underestimation of moduli obtained by nanoindentation has also been observed in other anisotropic biological materials, such as bone (Swadener et al. 2001; Fan et al. 2002). This was thought to correlate with the extent of anisotropy of the tested materials. Finally, a much larger COV of 21% was observed for the elastic modulus measured by microtension, which could be attributed to the sample preparation for single fiber testing. In this context, it should be noted that

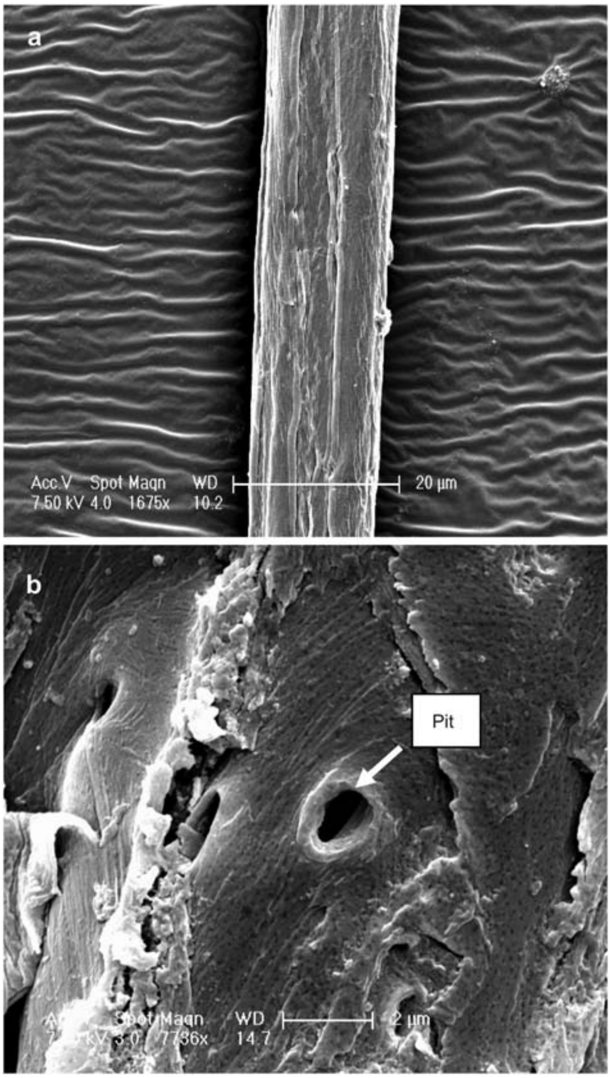

Figure 6 SEM micrographs of bamboo fibers. (a) Few pits are distributed along bamboo fibers. (b) The diameter of pits in bamboo fibers is normally <1 μm, much smaller than that in wood fibers.

Lee et al. (2007a,b) found that, by testing of lyocell fibers, there was no significant difference between modulus values inferred from nanoindentation and those obtained from singe fiber tensile testing. Of course, the behavior of lyocell fibers cannot be compared with that of native fibers of bamboo.

Conclusions

The results of combined investigations of bamboo fibers by means of *in situ* imaging nanoindentation and microtension of single fiber permit the following conclusions:

- Nanoindentation by Oliver-Pharr analysis significantly underestimates the real elastic modulus of anisotropic plant cell walls. For Moso bamboo fibers, the elastic modulus measured by nanoindentation was only 55% of the value obtained by microtension.
- Moso bamboo fibers can reach optimal mechanical performances at the age of 6 months or less. In the following growth period, the macroscopic mechanical performances of bamboo are improved by cell wall thickening, more than by enhancing the mechanical performances of cell wall itself.
- Moso bamboo fibers are much stronger and stiffer than most wood fibers tested, indicating that more attention should be focused on the utilization of bamboo fibers in the production of high performance fiber reinforced composites.

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