Effects of Moisture Content on the Mechanical Properties of Moso Bamboo at the Macroscopic and Cellular Levels

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To better understand how moisture content (MC) affects the longitudinal compressive mechanical properties of bamboo, mechanical tests on both the macroscopic and cellular levels were performed on Moso bamboo (*Phyllostachys pubescens* Mazei ex H. de Lebaie) at different MCs. At the macroscopic level, the compressive modulus of elasticity (CMOE) was determined using a common mechanical tester, while the indentation modulus of elasticity (EIT) and the hardness (HIT) of the bamboo fiber cell walls were obtained using nanoindentation. The results showed that CMOE, EIT, and HIT were all negatively correlated with the change in MC below the fiber saturation point (FSP) with strong linear relationships. However, the CMOE was found to be more sensitive to a change in MC than was EIT, which indicated that the bamboo was more sensitive to MC at the macro level than at the cellular level, at least in terms of longitudinal compression stiffness. Moreover, EIT was found to be much less sensitive to a change in MC than was HIT, which may explain why the longitudinal compression strength of bamboo was much more sensitive to changes in MC than was the compression modulus of elasticity on the macro scale.

Keywords: Moso bamboo; Moisture content; Compressive modulus of elasticity; Nanoindentation; Modulus of elasticity; Nanoindentation hardness

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INTRODUCTION

Due to the significant role that water plays in the application of wood and wood-based composites in building construction and other fields, numerous investigations regarding the effects of moisture content (MC) on the mechanical and physical performances of wood have been conducted in recent decades. The results have revealed that almost all the mechanical properties, such as compressive strength, hardness, and modulus of elasticity, decrease with increasing MC below the fiber saturation point (FSP) (Cave 1978; Green *et al*. 1986, 1988; Kretschmann and Green 1996; Wang and Wang 1999; Kojima and Yamamoto 2004). Moreover, the sensitivities of different mechanical properties to changes in the MC of wood have also been investigated both at the cell wall level (Yu *et al*. 2011) and at the macroscopic level (Ishimaru *et al*. 2001; Sudijono *et al*. 2004).

Bamboo is one of the most important non-timber forest resources in the world because it grows faster than almost any other tree on earth. As a lignocellulosic material, bamboo is also significantly affected by changes in MC, especially with regard to mechanical properties. However, the structure of bamboo is remarkably different from that of wood. At the cellular level, bamboo is a natural fiber-reinforced composite with bamboo fibers as the reinforcement and parenchyma cells as the matrix. The radial gradient distribution of fibers imparts to bamboo the characteristics of natural functional gradient materials (Parameswaran and Liese 1976; Ray *et al*. 2005). Therefore, the relationship between the MC and mechanical properties of wood may not be completely transferable to bamboo.

In comparison to that of wood, knowledge of the correlation between the MC and mechanical properties of bamboo is rather limited. In an earlier study, Godbole and Lakkad (1986) concluded that water absorption by bamboo caused considerable reduction in the strength and rigidity of the material. Recently, Jiang *et al.* (2012a) found further differences in the ways that the mechanical properties of bamboo exhibited sensitivity to changes in the MC. However, the authors ignored the sensitivity of these mechanical properties to changes in MC at the cellular level, and additional relevant research has not been reported.

In the present study, mechanical tests at both the cellular and macroscopic levels were performed on Moso bamboo (*Phyllostachys pubescens* Mazei ex H. de Lebaie) at different MCs to obtain a better understanding of the manner in which MC affects the longitudinal compressive mechanical properties of bamboo. The hardness (H_{IT}) and the elastic modulus (E_{IT}) of bamboo fiber cell walls were determined using nanoindentation. Nanoindentation is a powerful technique used to directly evaluate the mechanical properties of plant cell walls, first used by Wimmer *et al.* (1997) in the mechanical characterization of wood cell walls. Since then, this technique has found many more applications in the area of wood science and technology, including wood development (Gindl *et al.* 2004), wood fiber composites (Lee *et al*. 2007), and wood adhesion (Konnerth *et al.* 2006, 2007). The mechanical testing at the macroscopic level involved the investigation of the compressive modulus of elasticity (CMOE), which is thought to be related to some extent to the cell wall indentation modulus obtained through nanoindentation testing (Yu *et al.* 2011).

MATERIALS AND METHODS

Sample Preparation

Moso Bamboo aged 4.5 years, at which age the performance of Moso bamboo is very stable and excellent, was collected from a bamboo plantation located in Zhejiang Province, China. All the samples used for mechanical testing were prepared from the 15th to 25th internodes of eight culms, according to the Chinese national standard for bamboo (GB/T 15780-1995). The culms were split into bamboo strips with widths of 30 mm, as shown in Fig. 1. These strips were then further processed into samples with a final size of 20 mm × 20 mm × *t* mm (thickness of the bamboo culm wall) to be used for compression testing parallel to the grain. All the samples were air-dried in environmental conditions (relative humidity 40% to 60%) for more than four months before undergoing moisture conditioning.

Several bamboo blocks were cut from the same internodes and then further cut into sticks with dimensions of approximately 1 × 1 × 5 mm^3 (R × T × L) for nanoindentation testing. The sample preparation procedure for nanoindentation was similar to that proposed by Wimmer *et al.* (1997). In brief, the bamboo sticks were embedded in Spurr resin and cured in a plastic mold. After curing, the cross-section of the samples was polished with an ultramicrotome equipped with a diamond knife to obtain a very smooth surface for indenting.

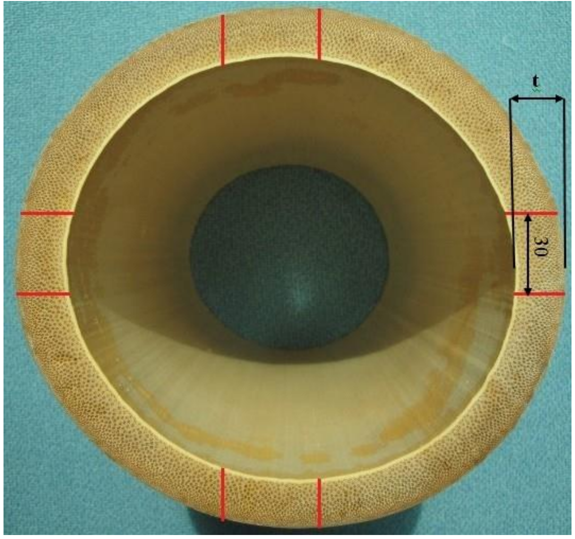

Fig. 1. The method of splitting bamboo into strips for compression sample preparation

Table 1. Relative Humidity (RH) in the Experiment at 20 °C and the Corresponding Equilibrium Moisture Content (EMC) of Samples after Conditioning in the Desiccators

Chemicals for Conditioning	RH (%)	EMC, Average (%)
Silica gel	2.90	0.44
Lithium chloride	12.50	4.94
Sodium bromide	68.7	10.02
Sodium chloride	74.1	13.62
Nitrate of potash	97.3	20.09
Water	-	23.04 (FSP)
Water	-	33.69

Macroscopic Compression Testing

The target MCs of the samples prepared for macroscopic compression testing were 0%, 5%, 10%, 13%, 20%, 23%, and 33%. The MC for the first 5 groups was achieved by placing these samples into desiccators containing different saturated salt solutions or a silica gel, as listed in Table 1. The relative humidity (RH) was measured by a hygrothermograph (TESTO 608-H1). The conditioning time was more than 40 days at a constant temperature of 20 °C. The actual equilibrium moisture content (EMC) of each sample was measured by weighing after the conditioning, in accordance with Jiang *et al.* (2012a). To achieve FSP, the bamboo samples were first soaked in water for a period of time until they had reached the target weight gain of about 23.5%. The soaked samples were then sealed in plastic bags until they had reached an EMC of about 23%, the FSP of mature Moso bamboo (Wang *et al.* 2010).

The compressive experiments were conducted using an electronic universal mechanical testing machine (Instron 5582, USA), according to the Chinese national standard (GB/T 15780-1995). To minimize the loss of moisture, the specimens were tested immediately after being taken out of the desiccators.

In Situ Imaging during Nanoindentation Testing

The MCs of the samples prepared for nanoindentation testing were adjusted by placing different saturated salt solutions or a silica gel in the chamber of the nanoindenter. The RH was measured with a hygrothermograph placed in the sample chamber, and the EMC values shown in Table 2 were calculated based on the sorption isotherm curve of the bamboo, as proposed by Jiang *et al*. (2012b).

Because it is impossible to maintain 100% RH in the chamber of a nanoindenter, the test at 100% RH was actually performed by conducting the nanoindentation test on a sample that was saturated with water. The detailed procedure for nanoindentation testing in water can be found by referring to Yu *et al.* (2011). All the samples were conditioned overnight before testing. During the tests, the temperature was kept between 22 °C and 24 °C.

Table 2. Relative Humidity and Corresponding Equilibrium Moisture Content (EMC) of Samples Prepared for Nanoindentation Testing

RH (%)	15.22	27.19	36.11	54.98	68.78	100
EMC, average (%)	4.30	6.04	6.91	8.66	10.92	23.04

The instrument used for nanoindentation testing was a Triboindenter with an *in situ* imaging function produced by Hysitron Company, USA, for which the resolution of the load and displacement were 1 nN and 0.01 nm, respectively. A Berkovich tip with a radius of less than 100 nm was used. Figure 2 shows the typical loading and unloading curves of the nanoindentation test. An “advanced feedback force control mode” was applied for a more accurate control of the peak load, and the target peak load and loading-unloading rate were 250 μN and 50 $\mu N\cdot s^{-1}$, respectively. The hold time at the peak load was 6 s. Only the cell walls of bamboo fibers were tested.

Based on the load-depth curve shown in Fig. 3, the key parameters of peak load (P_{max}), depth at peak load (h_{max}), and initial slope (S) of the unloading curve were determined. Contrary to conventional hardness, which is determined from the area of the residual imprint after unloading, the nanoindentation hardness is determined at peak load. The nanoindentation hardness (H_{IT}) was calculated based on the following equations,

$$\mathrm{H}_{\mathrm{IT}} = \frac{P_{\max}}{24.5 h_c^{2}}, \tag{1}$$

$$\mathrm{h_c} = \mathrm{h_{max}} - \varepsilon \frac{P_{\max}}{S} \tag{2}$$

where h_c is the contact depth of the indentation and ε is a constant that depends on the geometry of the indenter ($\varepsilon = 0.75$ for a Berkovich indenter).

The nanoindentation elastic modulus (E_{IT}) of a sample is defined as follows,

$$\frac{1}{E_r} = \frac{1-v^2}{E_{IT}} + \frac{1-v_i^{\,2}}{E_i} \ , \qquad (3)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta h_c}\frac{S}{\sqrt{24.5}} \qquad (4)$$

where E_i and v_i are the elastic modulus and Poisson ratio of the tips, respectively (for the diamond tips, E_i was 1141 GPa and v_i was 0.07); E_r is the reduced elastic modulus; and β is a constant (β=10.34 for a Berkovich indenter).

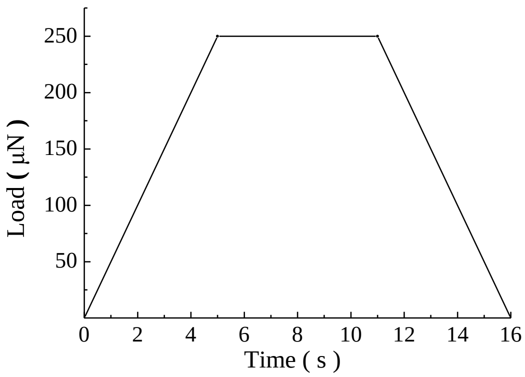

Fig. 2. Load-time curve of a nanoindentation test in Moso bamboo cell wall performed in feedback force control mode

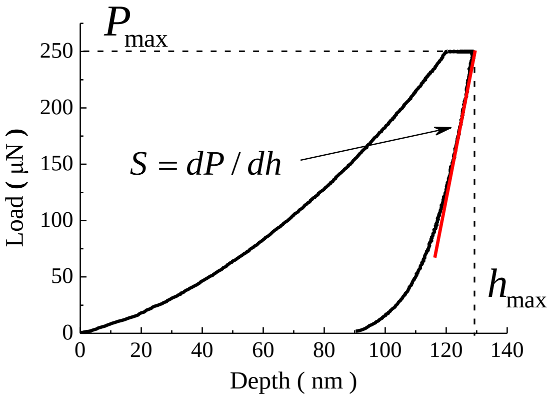

Fig. 3. Typical loading and unloading curves of the nanoindentation test and the relevant parameters

The residual indentations in the bamboo fiber cell wall are shown in Fig. 4, from which valid indentations for data analysis could be identified. For instance, the indentation indicated by the arrow in the image was invalid, as it was located too close to the middle lamella.

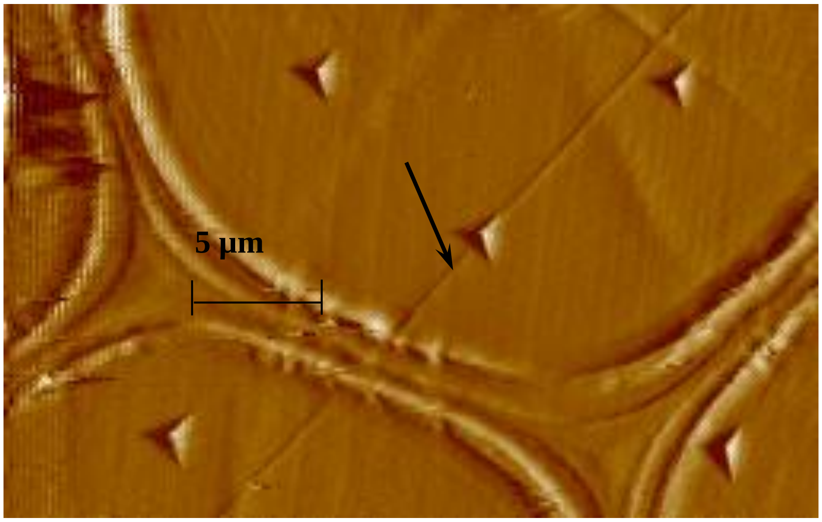

Fig. 4. Image showing the residual indentations on the cell walls of Moso bamboo fibers

RESULTS AND DISCUSSION

Effects of MC on the Compressive Modulus of Elasticity Parallel to the Grain

Figure 5 shows the correlation between the CMOE of bamboo and MC in the range of 0% to 35%. In previous studies, it was demonstrated that the FSP of mature Moso bamboo was approximately 23% (Wang *et al.* 2010; Jiang *et al.* 2012b). Therefore, a linear fit was performed on the data below FSP to render a better quantitative description. It was found that the CMOE linearly decreased by nearly 51%, from 7.27 GPa to 3.58 GPa, as the MC increased from 0.44% to 23%. The CMOE remained basically unchanged as the MC was continuously increased above FSP. From this linear equation, it could be inferred that a 1% change in MC would result in an increase or decrease in the CMOE of 0.15 GPa.

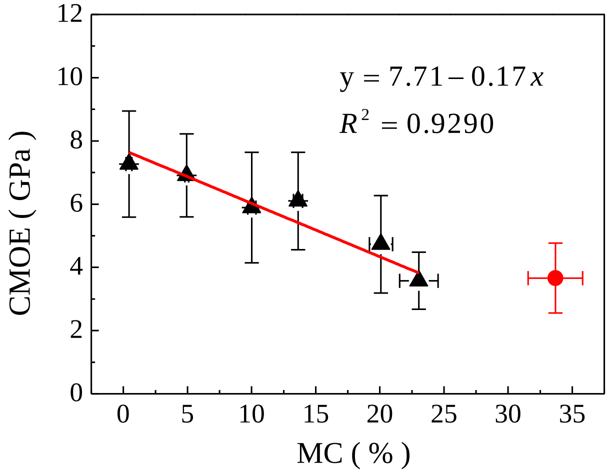

Fig. 5. The relationship between the compressive modulus of elasticity (CMOE) parallel to the grain and MC

To better evaluate the sensitivity of the CMOE to changes in MC, a reference value had to be obtained in advance. Here, the CMOE at 12% MC was selected as the reference value, calculated according to Equation 5. The moisture sensitivity K, namely the change rate of the properties per 1% MC change, could then be defined by the ratio between the slope of the linear equation and the CMOE at 12% MC (P_{12}).

$$K = 100\% \times \frac{\text{Scope}}{P_{12}} \tag{5}$$

From Equation 5, the K value of the CMOE was calculated to be 3.0%, significantly higher than the 1.09% value of the bending modulus and the 1.93% value of the tensile modulus measured by Jiang *et al*. (2012a). This could be partially explained by the different responses to changes in MC of the three main components (cellulose, hemicelluloses, and lignin) in the plant cell walls. The mechanical properties of the lignin/hemicellulose matrix have been experimentally (Cousins 1976, 1978) and theoretically (Sakurada *et al*. 1962; Koponen *et al*. 1989) proven to be much more sensitive to changes in MC than those of cellulose. In the process of compression testing, the hemicellulose/lignin matrix contributes considerably to the compressive deformation, while for the stiffness measurement in both the tensile and bending modes, cellulose undoubtedly dominates the whole process.

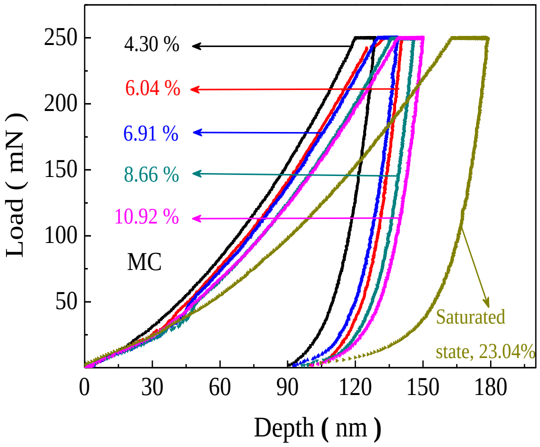

Fig. 6. Typical load-depth curves of nanoindentation tests in cell walls of Moso bamboo with different MCs

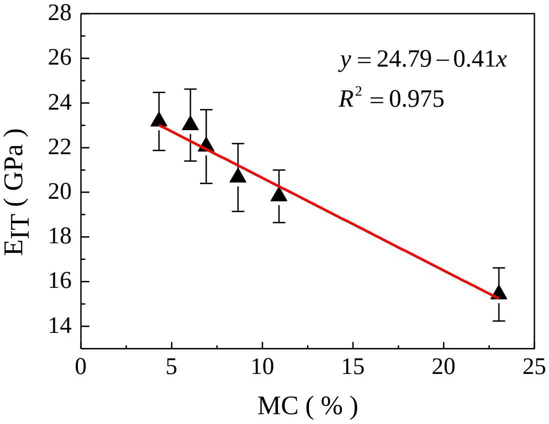

Fig. 7. Correlation between MC and indentation elastic modulus (E_{IT})

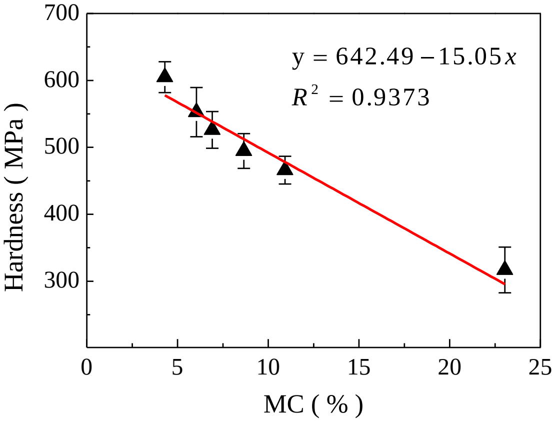

Fig. 8. Correlation between MC and indentation hardness (H_{IT})

Effects of MC on E_{IT} and H_{IT} of Bamboo Fiber Cell Walls

Figure 6 shows typical load-depth curves of nanoindentation tests in the cell walls of bamboo fibers with different MCs. The feedback force control mode ensured that the actual peak load of all the indentations was nearly equal to the set value of 250 μN. The maximum depth was positively correlated with MC, especially at the FSP.

The effects of MC on the E_{IT} and H_{IT} of Moso bamboo fibers are shown in Figs. 7 and 8, respectively. As the MC increased from 4.3% to the FSP, the E_{IT} decreased nearly linearly by 34%, from 23.17 GPa to 15.43 GPa. As shown before, at the macroscopic level, the relation between the longitudinal compression modulus of bamboo and the MC below the FSP could be well fitted to a linear equation, which implies that the mechanical dependence of bamboo on MC is largely determined by the mechanical response of the fiber cell walls to changes in MC. To determine the quantitative relationship between the E_{IT} of Moso bamboo fibers and the MC below the FSP, a linear fit was performed. A strong linear correlation (R^2=0.9750) was obtained. From the obtained linear equation, the *K* value for E_{IT} was found to be 2.06%.

A strong linear correlation also existed between the H_{IT} of the bamboo fiber cell walls and the MC below the FSP. The hardness decreased by 48% between 604.85 MPa and 316.80 MPa for the same MC range reported for E_{IT}. The K value for H_{IT} reached as high as 3.26%, significantly higher than that of E_{IT}. This indicated that the hardness of the bamboo fiber cell walls was much more sensitive to MC than was the elastic modulus. Similar results were also found for Masson Pine cell walls (Yu *et al*. 2011). The difference in K value between H_{IT} and E_{IT} could be used to explain the response of the longitudinal compression properties of bamboo to the MC at the macro level, as hardness can provide valuable information about longitudinal compression yield stress (Fischer-Cripps 2004). For Moso bamboo, Jiang *et al.* (2012a) found that the K value for longitudinal compression strength was 3.76%, higher than the K value of 3.0% that was found for the longitudinal compression modulus measured in the present study.

Comparison of the Effects of MC on the CMOE and E_{IT}

The sensitivities of CMOE and E_{IT} to changes in MC were significantly different in the present study. The K value was 3% for CMOE, while that for E_{IT} was only 2.06%, which meant that the indentation modulus of bamboo fiber cell walls was less sensitive to changes in MC than was the macroscopic compression modulus of bamboo. Different testing principles and the differences in testing scale were to a large extent responsible for the differences in K values. The E_{IT} determined by nanoindentation only reflected the cell wall S_2 layers of Moso bamboo fibers, while the CMOE represented an average result for both bamboo fibers and parenchyma cells. It is therefore hypothesized that the parenchyma cells in bamboo may have exhibited higher sensitivity to changes in MC in compression than did the bamboo fibers due to the much higher microfibrillar angle in the cell parenchyma walls. Additionally, the nanoindentation testing was only for the elastic modulus and hardness of the bamboo cell wall S_2 layers and did not test those of the other layers in the bamboo cell walls. Further research on the other layers of bamboo cell walls needs to be conducted; this is presently technically hindered by the surface quality of the samples and the spatial resolution of the present nanoindentation technique.

CONCLUSIONS

1. The mechanical dependence of bamboo on MC at the macroscopic level was largely determined by the response of its fiber cell walls to MC, at least for the longitudinal compression.

2. The E_{IT} of the bamboo fiber cell wall was less sensitive to changes in MC than was the CMOE, based on which observation it was hypothesized that the parenchyma cells in bamboo are more sensitive to changes in MC in compression than are bamboo fibers.

3. Both the E_{IT} and H_{IT} of the bamboo fiber cell walls showed a highly linear reduction with increasing MC. Meanwhile, the former was much less sensitive to changes in MC than the latter, which may explain the different response of the longitudinal compression modulus and the strength of the bamboo to changes in MC.

ACKNOWLEDGEMENTS

The authors are grateful to the National Natural and Science Foundation of China (31070491) for financially supporting this research.

REFERENCES CITED

Cave, I. D. (1978). “Modeling moisture-related mechanical properties of wood. Part I: Properties of the wood constituents,” *Wood Sci. Technol*. 12(1), 75-86.

Cousins, W. J. (1976). “Elastic modulus of lignin as related to moisture content,” *Wood Sci. Technol*. 10(1), 9-17.

Cousins, W. J. (1978). “Young’s modulus of hemicelluloses as related to moisture content,” *Wood Sci. Technol*. 12(3), 161-167.

Fischer-Cripps, A. C. (2004). *Nanoindentation*, 2nd Ed., Springer Verlag, New York.

Gindl, W., and Schöberl, T. (2004). “The significance of the elastic modulus of wood cell walls obtained from nanoindentation measurements,” *Compos. Part A* 35(11), 1345-1349.

Godbole, V. S., and Lakkad, S. C. (1986). “Effect of water absorption on the mechanical properties of bamboo,” *J. Mater. Sci. Lett.* 5(3), 303-304.

Green, D. W., Link, C. L., DeBonis, A. L., and McLain, T. E. (1986). “Predicting the effect of moisture content on the flexural properties of southern pine dimension lumber,” *Wood Fiber Sci.* 18(1), 134-156.

Green, D. W., Evans, J. W., Barrett, J. D., and Aplin, E. N. (1988). “Predicting the effect of moisture content on the flexural properties of Douglas-fir dimension lumber,” *Wood Fiber Sci.* 20(1), 107-131.

Ishimaru, Y., Arai, K., Mizutani, M., Oshima, K., and Iida, I. (2001). “Physical and mechanical properties of wood after moisture conditioning,” *J. Wood Sci.* 47(3), 185-191.

Jiang, Z., Wang, H., Tian, G., Liu, X., and Yu, Y. (2012a). “Sensitivity of several selected mechanical properties of Moso bamboo to moisture content change under the fiber saturation point,” *BioResources* 7(4), 5048-5058.

Jiang, Z., Wang, H., Yu, Y., Tian, G., and Liu, X. (2012b). “Hygroscopic behavior of bamboo and its blocking units,” *J. Nanjing Forestry University (Natural Science Edition)* 36(2), 11-14.

Kojima, Y., and Yamamoto, H. (2004). “Properties of the cell wall constituents in relation to the longitudinal elasticity of wood,” *Wood Sci. Technol.* 37(5), 427-434.

Koponen, S., Toratti, T., and Kanerva, P. (1989). “Modeling longitudinal elasticity and shrinkage properties of wood,” *Wood Sci. Technol*. 23(1), 55-63.

Konnerth, J., Valla, A., and Gindl, W. (2007). “Nanoindentation mapping of a wood-adhesive bond,” *Appl. Phys. A* 88(2), 371-375.

Konnerth, J., and Gindl, W. (2006). “Mechanical characterization of wood-adhesive interphase cell walls by nanoindentation,” *Holzforschung* 60(4), 429-433.

Kretschmann, D. E., and Green, D. W. (1996). “Modeling moisture content - Mechanical property relationships for clear Southern pine,” *Wood Fiber Sci*. 28(3), 320-337.

Lee, S. H., Wang, S., Pharr, G. M., and Xu, H. (2007). “Evaluation of interphase properties in a cellulose fiber-reinforced polypropylene composite by nanoindentation and finite element analysis,” *Compos. Part A* 38(6), 1517-1524.

Parameswaran, N., and Liese, W. (1976). "On the fine structure of bamboo fibers," *Wood Sci. Technol.* 10(4), 231-246.

Ray, A. K., Mondal, S., Das, S. K., and Ramachandrarao, P. (2005). "Bamboo - A functionally graded composite - Correlation between microstructure and mechanical strength," *J. Mater. Sci.* 40(19), 5249-5253.

Sakurada, I., Nukushima, Y., and Ito, T. (1962). "Experimental determination of the elastic modulus of crystalline regions in oriented polymers," *J. Polym. Sci.* 57(165), 651-660.

Sudijono, Dwianto, W., Yusuf, S., Iida, I., Furuta, Y., and Minato, K. (2004). "Characterization of major, unused, and unvalued Indonesian wood species I. Dependencies of mechanical properties in transverse direction on the changes of moisture content and/or temperature," *J. Wood Sci.* 50(4), 371-374.

Wang, S. Y., and Wang, H. L. (1999). "Effects of moisture content and specific gravity on static bending properties and hardness of six wood species," *J. Wood Sci.* 45(2), 127-133.

Wang, H., Yu, Y., Yu, Y., and Sun, F. (2010). "Variation of the fiber saturation point of bamboo with age," *Journal of Central South University of Forestry and Technology* 32(2), 112-115.

Wimmer, R., Lucas, B. N., Tsui, T. Y., and Oliver, W. C. (1997). "Longitudinal hardness and Young's modulus of spruce tracheid secondary walls using nanoindentation technique," *Wood Sci. Technol.* 31(2), 131-141.

Yu, Y., Fei, B., Wang, H., and Tian, G. (2011). "Longitudinal mechanical properties of cell wall of Masson pine (*Pinus massoniana* Lamb) as related to moisture content: A nanoindentation study," *Holzforschung* 65(1), 121-126.

Article submitted: July 29, 2013; Peer review completed: Sept. 9, 2013; Revised version received and accepted: Sept. 10, 2013; Published: September 12, 2013.