VARIATION OF MICROFIBRIL ANGLE AND DENSITY IN MOSO BAMBOO (*PHYLLOSTACHYS PUBESCENS*)

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WANG XQ, LI XZ & REN HQ. 2010. Variation of microfibril angle and density in moso bamboo (*Phyllostachys pubescens*). The variation patterns of microfibril angle (MFA) and air-dry density with respect to age as well as to different vertical and horizontal positions within the culm were investigated in moso bamboo (*Phyllostachys pubescens*) of different ages grown in south-eastern China. X-ray diffraction was used to determine the MFA of the culms. Although the analysis of variance indicated that age, height location as well as horizontal layer had significant effects on MFA, the mean MFA varied slightly from 8.17° to 10.51°, regardless of age or positions within the culm, with a coefficient of variation of only 6.7%. The air-dry density varied considerably with age and also within the culm, with the mean values ranging from 0.41 to 1.14 g cm⁻³. The air-dry density increased substantially during the initial four years of growth but was almost constant at older ages. The outer layers of the culm had significantly higher density than the inner layers and the top portion of the culm showed generally higher density than the lower portions.

Keywords: Air-dry density, vertical position, horizontal position

WANG XQ, LI XZ & REN HQ. 2010. Variasi sudut mikrofibril dan ketumpatan buluh *Phyllostachys pubescens*. Corak variasi sudut mikrofibril (MFA) dan ketumpatan kering udara buluh *Phyllostachys pubescens* di tenggara China dikaji berdasarkan usia serta kedudukan lapisan menegak dan mendatar yang berbeza dalam kulma. Pembelauan sinar-X diguna untuk menentukan MFA kulma. Walaupun analisis varians menunjukkan bahawa usia, lokasi ketinggian dan lapisan mendatar mempunyai kesan signifikan terhadap MFA, min MFA berubah sedikit dari 8.17° ke 10.51° tanpa mengira usia atau kedudukan dalam kulma dan koefisien variasi hanyalah sebanyak 6.7%. Ketumpatan kering udara berubah dengan agak banyak mengikut usia serta kedudukan dalam kulma. Min ketumpatan yang diperoleh berjulat antara 0.41 g cm⁻³ hingga 1.14 g cm⁻³. Ketumpatan kering udara meningkat mendadak semasa pertumbuhan pada empat tahun yang awal namun tidak banyak berubah dalam buluh yang lebih tua. Lapisan luar kulma mempunyai ketumpatan yang lebih tinggi secara signifikan berbanding dengan lapisan dalam. Bahagian atas kulma juga menunjukkan ketumpatan yang lebih tinggi daripada bahagian bawah.

INTRODUCTION

Bamboo is an important non-timber forest resource growing mainly in many tropical and subtropical countries. Owing to its fast growth rate, high strength and stiffness, easy machinability and local availability, bamboo has been widely used as a traditional material for furniture, construction and household products. Recently, bamboo has gained increasing interest as an alternative material to wood or wood-based materials and is utilised to manufacture various types of composted products such as fibreboard, zephyr board and oriented strandboard for industrial application (Nugroho & Ando 2000, Li 2004, Sumardi *et al.* 2007). To maximise the utilisation of bamboo, many studies have been performed on its fundamental anatomical, physical and mechanical properties (Liese 1987, Wang 2001, Jiang 2002).

From a practical point of view, density is the single most important physical characteristic affecting wood properties such as stiffness, strength and shrinkage behaviour and thus, to a large extent, determines the final application of woody material. Previous studies have demonstrated the variation of specific gravity of some bamboo species with respect to age and vertical height location as well as horizontal layer in the culms (Ahmad 2000, Li 2004). On the other hand, however, little attention has been paid to the cellulose microfibril orientation of

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bamboo fibres. Studies of wood have clearly shown that the microfibril angle (MFA) in the second cell wall layer is one of the main determinants of wood stiffness and strength as well as shrinkage properties. Compared with wood, the ultrastructure and microfibril orientation of fibre walls in bamboo are more complicated. The fibre wall of bamboo is characterised by a polylamellate wall structure with alternating broad and narrow lamellae of differing microfibril orientation (Liese 1998). The narrow lamellae are reported to show fibril angle of $85-90^{\circ}$ to the axis and the broader ones have fibril angle almost parallel to the axis (Parameswaran & Liese 1976). This peculiar ultrastructural organisation and microfibril orientation of bamboo fibres may have a decisive effect on the physical and mechanical properties of bamboo culms. X-ray diffraction (XRD) is a well-established method for the determination of mean MFA of wood. Recently, this method has also been used to measure MFAs of some bamboo species (Wang 2001, Yu et al. 2007, Xu 2008). However, so far, the variation pattern of MFA within bamboo culms as well as with respect to age is scarcely reported in the literature.

Due to the importance of density and MFA in determining the physical and mechanical properties of woody material, the present study was carried out to clarify their variation patterns with respect to the horizontal and vertical positions of culms in moso bamboo (*Phyllostachys*) *pubescens*) of different ages. This bamboo is one of the most popular and valuable bamboo species in China. This information will definitely contribute to the optimal utilisation of moso bamboo for industrial application.

MATERIALS AND METHODS

Sampling

Bamboo culms (P. pubescens) were collected from an experimental forest located in Zhejiang Province, China. Five representative culms for each age class (0.5, 1, 2, 4 and 6 years old) were harvested for this study. The culms were 13 to 15 m in length with a diameter at breast height (dbh) ranging from 9 to 11 cm. From each culm, the 15th, 23rd and 30th internodes were selected to represent three different vertical height locations (bottom, middle and top respectively) of the culm. After air drying, bamboo blocks with dimensions of 20 (longitudinal) \times 10 $(tangential) \times t mm$ (radial) were cut from the internodes. These blocks were then split evenly into five thin layers of 1 ± 0.2 mm thick along the radial direction, designated as layers 1, 2, 3, 4 and 5, from the inner layer to the outer layer of the culms (Figure 1). All the specimens were conditioned at 20 °C and 65% relative humidity until a moisture content of approximately 10% was achieved.



Figure 1 Sampling for the bamboo specimens

Measurement of density and microfibril angle

The air-dry densities of the specimens were determined based on the air-dry (10% moisture content) weight and volume calculated from their actual dimensions. Following density test, the same specimens were also used to determine the mean MFA of bamboo fibres by an X-ray diffractometer (X' Pert Pro, Panalytical, USA). MFA measurements were performed in the symmetrical transmission mode. The main experimental conditions were as follows: Cu Ka radiation ($\lambda = 0.154$), tube voltage 40 kV, tube current 40 mA, scanning angle range 0°-360° and scanning step 1°. From the obtained intensity curves of X-ray diffraction, the mean MFA of bamboo fibres was determined based on the method developed by Cave (1966) using the formula:

$$MFA = 0.6 \times T \tag{1}$$

where T was the X-ray diffraction parameter and 0.6 was the constant derived by Cave (1966). Three measurements were made for each sample.

Statistical analysis

Data analysis was conducted using SAS 8.0 (1999, SAS Institute Inc., USA) software. Three-way analysis of variance (ANOVA) was performed to evaluate the effects of vertical (height) and horizontal (layer) positions of culms as well as age on the properties of bamboo. A multiple comparison with least significance difference (LSD) method at $\alpha = 0.05$ was also carried out to show further differences between various levels of each variable.

RESULTS

Microfibril angle analysis

The typical intensity curves of moso bamboo are shown in Figure 2. The diffraction intensity of samples from outer layers of the culm was extremely strong compared with that from inner layers, showing an obvious decreasing tendency from the outer to the inner layers.

The mean MFA values of moso bamboo of different ages at different horizontal and vertical positions are listed in Table 1. The mean MFA ranged from 8.17° to 10.51° regardless of age or positions within the culm. The analysis of variance indicated that age, height and layer had significant effects on MFA (Table 2). Mean MFA values and their variation for each age class are shown in the box-and-whisker plot (Figure 3). The result of the multiple comparison test is also given as indicated by different letters in the plot. In general, MFA increased from year 0.5 to year 2, and then decreased at older ages. The LSD test showed that MFA of the 2-year-old bamboo was significantly different from that of years 0.5, 1 and 4, and MFA of the 6-year-old bamboo was the smallest.



Figure 2 Typical X-ray diffraction pattern obtained from different radial layers of bamboo culms

A	Height	MFA (°)*					
Age (year)		Layer 1	Layer 2	Layer 3	Layer 4	Layer 5	
0.5	Bottom	9.70 (0.16)	9.37 (0.27)	9.30 (0.48)	8.89 (0.54)	9.17 (0.30)	
	Middle	9.79 (0.23)	9.57 (0.21)	9.75 (0.46)	9.40 (0.19)	8.91 (0.23)	
	Тор	9.76 (0.30)	9.72 (0.27)	9.92 (0.32)	9.62 (0.30)	8.94 (0.56)	
1	Bottom	9.83 (0.32)	9.78 (0.28)	9.90 (0.52)	8.94 (0.28)	8.60 (0.59)	
	Middle	9.74 (0.38)	9.62 (0.35)	9.86 (0.52)	9.37 (0.46)	8.77 (0.44)	
	Тор	9.76 (0.10)	9.80 (0.40)	10.01 (0.47)	9.47 (0.32)	8.57 (0.40)	
2	Bottom	10.01 (0.45)	10.44 (0.88)	10.28 (0.53)	8.93 (0.63)	8.85 (0.60)	
	Middle	9.91 (0.41)	9.88 (0.30)	10.16 (0.61)	9.13 (0.52)	8.54 (0.47)	
	Тор	9.74 (0.20)	10.46 (0.86)	10.51 (0.37)	9.70 (0.68)	8.82 (0.66)	
4	Bottom	9.96 (0.19)	9.65 (0.26)	9.69 (0.37)	9.03 (0.36)	8.56 (0.21)	
	Middle	9.75 (0.21)	9.58 (0.43)	9.68 (0.18)	9.30 (0.45)	8.17 (0.20)	
	Тор	10.04 (0.43)	9.62 (0.15)	9.71 (0.25)	9.57 (0.26)	8.81 (0.32)	
6	Bottom	9.90 (0.18)	9.65 (0.21)	9.58 (0.43)	8.50 (0.35)	8.26 (0.68)	
	Middle	9.66 (0.11)	9.45 (0.17)	9.54 (0.38)	8.87 (0.26)	8.40 (0.24)	
	Тор	9.52 (0.17)	9.47 (0.21)	9.55 (0.22)	9.05 (0.18)	8.46 (0.41)	

 Table 1
 Mean microfibril angles (MFA) of moso bamboo at different positions

*Data are shown as means of five bamboo culms with standard deviations in parentheses.

 Table 2
 Analysis of variance table for MFA and air-dry density of moso bamboo

	Source of variation	DF	SS	MS	F value	$\Pr > F$
MFA	Age	4	9.6057	2.4014	13.58	< 0.0001
	Height	2	1.6773	0.8387	4.74	0.0093
	Layer	4	74.0434	18.5108	104.70	< 0.0001
Air-dry density	Age	4	2.4950	0.6237	163.77	< 0.0001
	Height	2	0.1449	0.0724	19.02	< 0.0001
	Layer	4	12.4535	3.1134	817.42	< 0.0001



Figure 3 Variation of microfibril angle (MFA) as a function of age for moso bamboo depicted as box-and-whisker plot (50% of the values inside the box; median, line in the box; arithmetic mean, rectangle in the box). Means with the same letter are not significantly different at $\alpha = 0.05$.

The vertical position of bamboo culms also had an effect on MFA (Figure 4). Although there was no difference in MFA between the bottom and the middle portions of the culms, the difference in MFA between the top portion and the lower portions was statistically significant. On the other hand, it was interesting to note that there was a difference in MFA across the culm wall (Figure 5). The MFA varied little from the outer layer to the middle layer, whereas the inner layers (layers 4 and 5) showed slightly lower MFA values than the rest of the layers.

Analysis of air-dry density

The mean air-dry density values of moso bamboo of different ages at different horizontal and vertical positions are listed in Table 3. The mean air-dry density varied considerably from 0.41 to 1.14 g cm⁻³. The ANOVA indicated that age, height and layer had extremely significant effects on air-dry density (Table 2). The mean air-dry density of bamboo culms increased significantly at young age and then levelled off at older ages (Figure 6). For example, the mean



Figure 4 Vertical variation of MFA with the culm height for moso bamboo. Means with the same letter are not significantly different at $\alpha = 0.05$.



Figure 5 Radial variation of MFA from the outer layer to the inner layer for moso bamboo. Means with the same letter are not significantly different at $\alpha = 0.05$.

Age (year)	Height	Density (g cm ⁻³)						
		Layer 1	Layer 2	Layer 3	Layer 4	Layer 5		
0.5	Bottom	1.02 (0.06)	0.66 (0.10)	0.48 (0.07)	0.41 (0.04)	0.43 (0.04)		
	Middle	1.04 (0.05)	0.62 (0.07)	0.48 (0.09)	0.44 (0.09)	0.46 (0.06)		
	Тор	1.07 (0.06)	0.63 (0.09)	0.50 (0.05)	0.47 (0.04)	0.50 (0.04)		
1	Bottom	0.99 (0.01)	0.64 (0.06)	0.50 (0.05)	0.46 (0.04)	0.53(0.09)		
	Middle	1.02 (0.00)	0.71 (0.06)	0.56 (0.04)	0.49 (0.03)	0.56 (0.07)		
	Тор	1.08 (0.05)	0.72 (0.04)	0.59 (0.04)	0.52 (0.04)	0.53 (0.03)		
2	Bottom	1.03 (0.05)	0.72 (0.04)	0.60 (0.06)	0.56 (0.04)	0.53(0.09)		
	Middle	1.06 (0.06)	0.73 (0.08)	0.58 (0.06)	0.55 (0.05)	0.54 (0.07)		
	Тор	1.07(0.05)	0.76 (0.09)	0.64 (0.07)	0.61 (0.04)	0.59 (0.05)		
4	Bottom	1.08 (0.04)	0.85 (0.02)	0.68 (0.03)	0.66 (0.05)	0.69 (0.06)		
	Middle	1.04 (0.11)	0.84 (0.07)	0.74 (0.04)	0.70 (0.06)	0.73 (0.04)		
	Тор	1.12 (0.03)	0.87 (0.07)	0.76 (0.06)	0.71 (0.06)	0.72 (0.03)		
6	Bottom	1.10 (0.02)	0.85 (0.01)	0.72 (0.01)	0.67 (0.02)	0.66 (0.03)		
	Middle	1.10 (0.03)	0.87 (0.02)	0.74 (0.06)	0.70 (0.05)	0.69 (0.03)		
	Тор	1.14 (0.04)	0.94 (0.10)	0.76 (0.05)	0.71 (0.05)	0.68 (0.06)		

 Table 3
 Mean air-dry density of moso bamboo at different positions

Data are shown as means of five bamboo culms with standard deviations in parentheses.



Figure 6 Variation of air-dry density as a function of age for moso bamboo. Means with the same letter are not significantly different at $\alpha = 0.05$.

density increased 33% from year 0.5 (0.61 g cm⁻³) to year 4 (0.81 g cm⁻³) and kept almost constant from then till year 6 (0.82 g cm⁻³). The LSD test showed there were statistically significant differences in the air-dry densities between the three young age classes, and their density values were significantly lower than those of the two

older age classes. In addition, it was observed that the bamboo culms of young ages, especially the year 0.5, showed large variation in air-dry density, and this variation appeared to become small with ageing (Figure 6), indicating that a more homogeneous structure was formed during the maturation process of bamboo culms. Height effect was also significant for the airdry density of the culms. The top portions had consistently higher density than the bottom portions for each horizontal layer group, regardless of age (Table 3). On average, the air-dry density of the top portions was 0.75 g cm⁻³, which was slightly higher than that of the bottom portion (Figure 7). On the other hand, the largest portion of variation in density was from the horizontal positions of the culms. The outer layers showed consistently higher density than the inner layers, irrespective of age or culm height. The mean density value of layer 1 was almost twice that of layer 5. The LSD test showed that there were significant differences in density between the outer three layers, whereas the difference between the inner two layers was not statistically significant (Figure 8).



Figure 7 Vertical variation of air-dry density with the culm height for moso bamboo. Means with the same letter are not significantly different at $\alpha = 0.05$.



Figure 8 Radial variation of air-dry density from the outer layer to the inner layer for moso bamboo. Means with the same letter are not significantly different at $\alpha = 0.05$.

DISCUSSION

Microfibril angle

MFA values obtained in the present study varied narrowly from 8.17° to 10.51° for moso bamboo, irrespective of age or positions within the culm, with a coefficient of variation of only 6.7%. This result supported earlier observations that MFAs varied little in moso bamboo from 8° to 10° (Wang 2001, Yu et al. 2007). Similar results were also reported in previous studies of other bamboo species such as Guadua amplexifolia, Gigantochloa apus and Bambusa pervariabilis with MFA values ranging from 6° to 11° (Xu 2008). It should be noted that although both parenchyma tissue and fibres in bamboo culm appear to contribute to the X-ray diffraction signal of the samples, the contribution of parenchyma cells may be negligible due to their thin-walled structure as well as randomly oriented microfibrils. Thus, the information obtained by X-ray diffraction is mainly derived from fibres and the MFA determined represents the mean fibril angle of the fibres within the sample measured.

As mentioned above, the fibre wall of bamboo consists of alternating narrow and broad lamellae, with the former showing fibril angle of 85–90° to the axis and the latter with fibril angle almost parallel to the axis. Thus, broad lamellae with small fibril angle may to a large extent determine properties of the entire fibre wall due to their great thickness and high proportion relative to narrow lamellae. This may partly account for the relatively small MFA observed in moso bamboo.

It is interesting to note that horizontal culm wall layers of bamboo had statiscally different MFA values (Table 2). The MFA showed a decreasing tendency from the outer layer to the inner layer, irrespective of age or culm height, although the magnitude of change in MFA was less than 2°. This radial variation pattern of MFA is significantly different from that of wood, which is characterised by a remarkable decrease in MFA from the pith outward for a number of years (juvenile wood) and then reaches a relatively constant value (mature wood) (Zobel & Van Buijtenen 1989). For some wood species, the change in MFA along the radial direction may exceed 30°. The relatively uniform distribution of MFA in bamboo implies that MFA may not be the dominant factor in determining the variation of physical and mechanical properties of bamboo culms, which is in contrast to the case in wood. From a practical point of view, the low variation of MFA in bamboo culm may be favourable for imparting consistency to properties of fibre-based products of bamboo such as paper, fibreboard and textile because mechanical properties of fibres are largely dependent on MFA.

Air-dry density

The air-dry density of moso bamboo varied considerably with age and also within the culm. The densities of culms are strongly correlated with their anatomical structure. The bamboo culm consists of about 52% parenchyma, 40% fibres and 10% conducting tissue and its density is largely determined by the content of thick-walled fibres, fibre diameter and cell wall thickness (Liese 1998). The present study showed that the mean air-dry density of bamboo culm increased 33% from year 0.5 to year 4, and then kept almost constant at older ages. The increase in density of the culm is mainly due to thickening of the cell wall during maturation of the culm, as clearly demonstrated by other studies (Murphy & Alvin 1997, Liese 1998, Lybeer et al. 2006). Our results also indicated that cell wall thickening occurred mostly in the first four years resulting in substantial increase in density but only slight changes during later years. This suggests that bamboo culm appears to evolve into a mature stage after four years of growth.

One of the most unique characteristics of bamboo is the large variation in density across the culm wall, with outer layers showing remarkably higher density than inner layers. This density variation within the culm may be attributed to the distribution pattern of fibres and parenchyma tissue of bamboo. It has been reported that across the culm wall, the percentage of fibres decreases greatly from the outer to the inner layers, while the parenchyma increases, resulting in about 50% of the fibres being located in the outer third of the culm wall (Liese 1998), which accounts for the considerable radial variation of density in bamboo culms. With respect to the variation of density along the culm height, top portions of culms showed generally higher density than the corresponding lower portions. This can be explained by the fact that the top portions possess relatively thinner culm wall with a higher frequency of vascular bundles which have a higher density than parenchyma tissue. The density is the most important single characteristic affecting the physical and mechanical properties of bamboo. In order to use bamboo materials efficiently, the specific horizontal layers of culms may be separated and utilised for specific purposes based on their density difference.

CONCLUSIONS

Mean MFA values of moso bamboo varied slightly from 8.17° to 10.51° , irrespective of age or position within the culm, with a coefficient of variation of only 6.7%. ANOVA indicated that age, height location as well as horizontal layer had significant effects on MFA, although the magnitude of change in MFA was small. MFA may not be a dominant factor in determining the variation in physical and mechanical properties of bamboo culms. The air-dry density of moso bamboo varied considerably with age and also within the culm. It increased considerably during the initial four years of growth and then kept almost constant at older ages. One of the most unique characteristics of bamboo is the large variation in density across the culm wall, with the outer layers showing remarkably higher density than the inner layers. The top portion of the culm showed generally higher density than the lower portions.

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