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Bamboo fiber reinforced thermoplastic molding made of steamed wood flour

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Abstract To improve the mechanical property of moldings made of steamed wood flour, layered wood moldings reinforced with steam-exploded bamboo fiber was prepared. Setting the bamboo fiber weight fractions at 25, 50, and 75%, and number of layers at three-, five-, and seven-layered wood moldings were prepared by compression molding. The results of tensile test showed that the tensile strength as well as Young's modulus increased along with the increase in the bamboo fiber fractions. Where the bamboo fiber content was 75%, the tensile strength became approximately 3.8 to 5.8 times greater, and the tensile Young's modulus became approximately 2.5 times greater when compared to moldings of 100% wood flour. This fact shows that bamboo fiber is effective to improve the mechanical property of wood moldings. In addition, the tensile strength also increased as the number of layers increased. This result suggested that interfacial shear stress was produced between the layers of bamboo fiber and wood flour.

Introduction

An effective use of wood biomass as sustainable carbon-neutral resource is essential for construction of a recycling society, and the development of usage techniques of wood biomass has been desired. As part of this development, wood–plastic composites (WPC) have been actively investigated recently [1–5]; however, an attempt to create moldings only of biomass materials without adding plastic has also been performed [6, 7]. In our previous report [8], we discussed the process of creating resin-like moldings from steamed Japanese beech flour without using petroleum-based adhesives. The beech flour steamed at a temperature of 180 °C or higher flowed under a high-temperature and high-pressure environment [9] and resin-like moldings with a density of 1.45 g/cm^3 were obtained by compression molding. These resin-like moldings were probably obtained because the steaming made the molecules of hemicellulose and lignin smaller, causing them to plasticize due to the reheating. Water absorption of these moldings after 24 h immersion in distilled water at 20 °C is about 5% [8], and this value is higher than that of thermoplastics but much smaller than standard value (20%) of tempered hardboard [10]. Moreover, these moldings have bending strength and bending Young's modulus with the same degree of polypropylene (PP) and acrylonitrile butadiene styrene (ABS); however, it has a low fracture toughness causing it to be quite fragile. In addition, the rigidity of this molding is nowhere near that of engineering plastics such as nylon and polycarbonate. In order to use this molding material as an alternative for such engineering plastics, it needs to be greatly reinforced.

Reinforced plastics combining glass fiber and mineral filler have been applied in a wide variety of products including automobile parts. However, these plastic products with mineral compounds have become an environmental problem because of the residue generated after incineration. As a solution for this issue, reinforced plastics using natural fibers, such as jute, bamboo, hemp, sisal

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hemp, and kenaf hemp, have been actively developed [11–15].

The purpose of this research is to improve the mechanical property of moldings made of steamed wood flour, and furthermore, to conduct material design for moldings. Focusing on this goal, we produced layered wood moldings reinforced with steam-exploded bamboo fiber and evaluated its mechanical characteristics.

Experiments

Materials

Japanese beech (*Fagus crenata* Blume) shaving was steamed in an autoclave (Yasujima SBK-450BS) at 200 °C for 20 min in the presence of water, the amount of which equal to the oven-dry weight of raw material. The steamed material was dried at room temperature and milled in a Wiley mill. Then milled flour was sieved with screens with a size of 90–250 µm. Steam-exploded bamboo fiber (Ban Co., Ltd.) was used as a reinforcing material. Each continuous bamboo fiber was cut to a length of 100 mm.

Tensile test of bamboo fiber

Tensile test on the bamboo fibers were conducted according to ISO 11566 to understand the mechanical characteristics of the bamboo fibers used as reinforcing material. First, both 35-mm ends of a 100-mm bamboo fiber was fixed on a mat board using an instant adhesive as shown in Fig. 1, and another mat board was attached on the top from above. To calculate the dimension of the cross-sectional area of the bamboo fiber, the diameters were first obtained by image capture of each individual fiber from two different directions using an optical microscope, and then the area was calculated by applying elliptic approximation as the following formula.

$$A = \frac{\pi ab}{4} \quad (1)$$

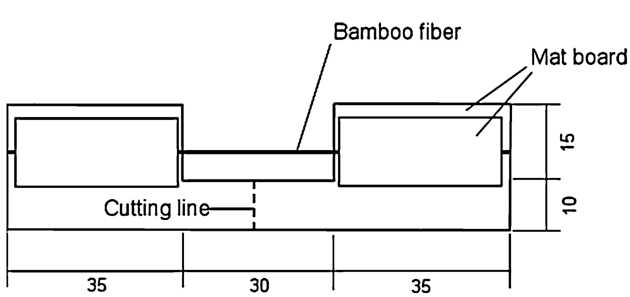

Fig. 1 Tensile test specimens of bamboo fiber, unit: mm

where *a* and *b* are the diameters of a fiber. Then, the specimen was attached in the tensile tester (Orientec, Tensilon RTC-1310A) and the mat boards were cut before the tensile test. The crosshead speed was 1 mm/min.

Preparation of layered wood molding

The bamboo fiber weight fraction was set at 25, 50, and 75%, and wood flour and bamboo fiber were layered alternately. The number of layers was set at three, five, and seven. Figure 2 shows the diagrams and calculated thicknesses after compression molding process.

First, prescribed amount of bamboo fiber was compressed at room temperature to make a preform. Next, wood flour with the adjusted moisture content of 3% and bamboo fiber preforms were layered alternately in the molding container, and were compressed at room temperature to create a layered preform. All bamboo fibers were aligned in the same direction, and wood flour was used for the outermost layers. Then the layered preform was heated and pressed at 150 °C and 30 MPa for 10 min to prepare a 100 × 100 × 4 mm layered molding. Two-layered moldings under each condition were made. For comparison, a molding with 100% steamed wood flour (0% of bamboo fiber) and a molding with 100% bamboo fiber (0% of wood flour) was made.

Tensile test of layered wood molding

From the layered moldings, a tensile test specimen with a parallel orientation direction of the bamboo fiber was obtained. Tapers were made on the specimen using the belt sander, and a strain gauge was attached on the central portion of the specimen. The tensile test was performed according to ISO 527-1 and 527-2. Figure 3 shows the shapes and dimensions of the specimen. The tensile test was performed at the test speed of 1 mm/min. Six specimens were tested for each sample.

Results and discussion

Tensile characteristics of bamboo fiber

Figure 4 shows the relation between the tensile strength and the cross-sectional area of the bamboo fiber. The cross-sectional area of most of the fibers ranged from 0.03 to 0.06 mm^2, a couple of these fibers were assumed to have bundled, and their areas ranged from 0.14 to 0.2 mm^2. These fibers were treated as one fiber and were composed of several thin fibers bundled together. Where fibers were ruptured, the rupture occurred at each individual point as

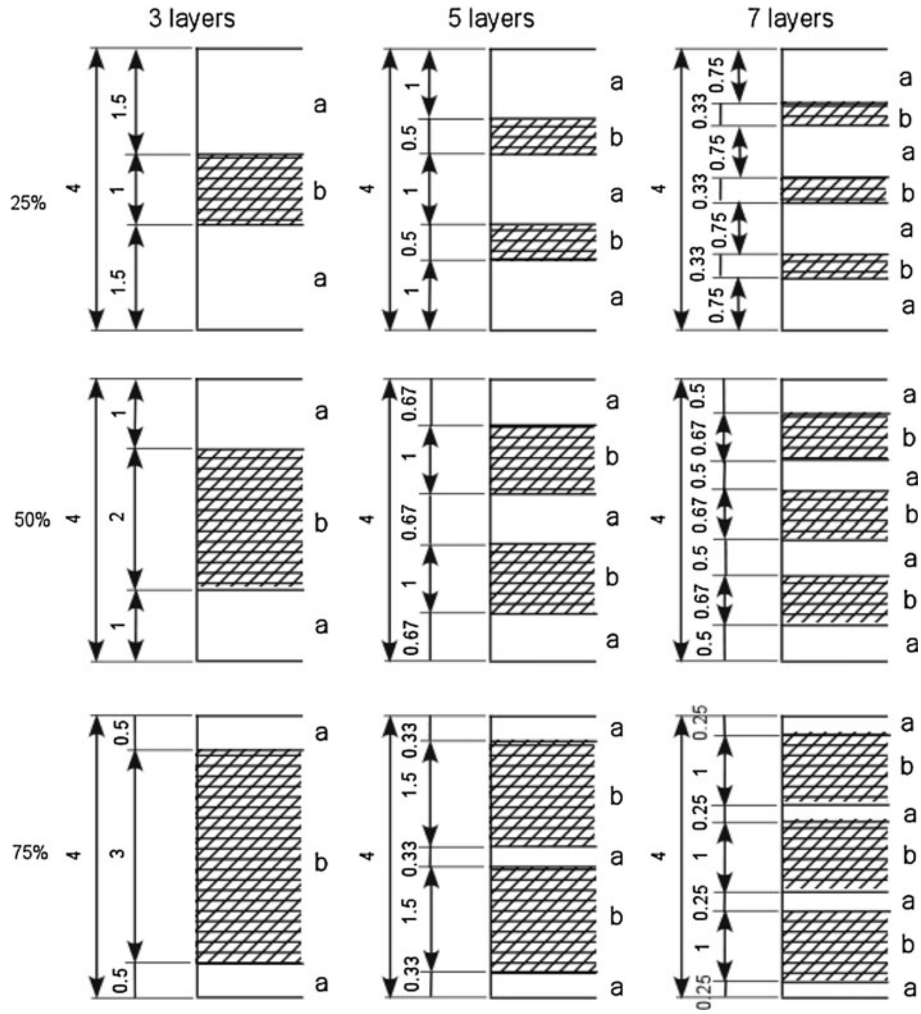

Fig. 2 Diagram and calculated thickness of layers in molding, unit: mm. **a** wood flour layer, **b** bamboo fiber layer

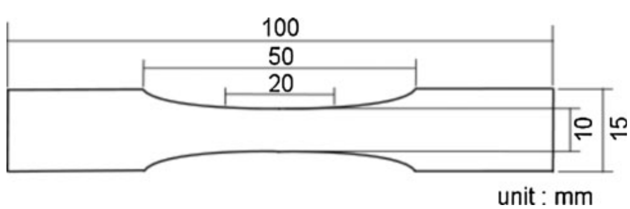

Fig. 3 Dimensions of the specimen for tensile test

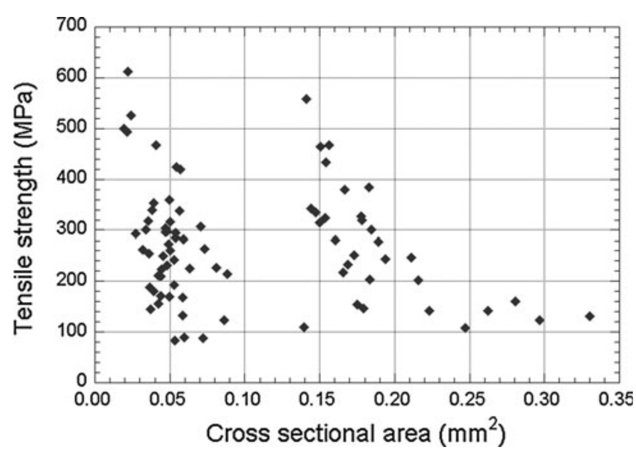

Fig. 4 Relation between tensile strength and cross-sectional area in bamboo fiber

shown in Fig. 5. Tensile strength significantly varied from 83 to 612 MPa. The following reasons can be considered as the causes for this significant variation in tensile strength: cross-sectional areas were overestimated because of the woody parts (parenchyma cells) attached to the fibers, and the degree of damage experienced during the steam-explosion process and the fiber sampling process differed [16]. To evaluate the tensile characteristics of the bamboo fiber used in this research and its variation, we applied the two-parameter Weibull distribution functions, which are frequently used as a distribution model of tensile fiber for brittle failures, and we also examined the calculation of the parameters of this distribution function and the adequateness of its distribution [17].

Fig. 5 Rupture point of bamboo fiber

The two-parameter Weibull distribution is based on the weakest link theory, and its cumulative distribution function F is expressed with the following formula.

$$F(\sigma) = 1 - \exp\left\{-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right\} = 1 - \exp\left\{-\frac{A}{A_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right\} \quad (2)$$

where m is the shape parameter, and σ_0 is the scale parameter. V and V_0 are the fiber's dimensions and average fiber's dimensions, respectively, while A and A_0 are the fiber's cross-sectional area and average fiber's in the cross-sectional area, respectively,. Where a natural logarithm is applied to both sides of formula (2), the following formula is then obtained.

$$\ln\ln\frac{1}{1-F(\sigma)} - \ln\left(\frac{A}{A_0}\right) = \mathrm{m}\ln\sigma - \mathrm{m}\ln\sigma_0 \quad (3)$$

In this formula, m and σ_0 can be obtained by conducting straight-line approximation after plotting the left side of the formula on the vertical axis, and plotting lnσ on the horizontal axis. The cumulative rupture probability, $F(\sigma)$, was obtained using the median rank method. In the median rank method, the ith fracture probability where n data is aligned in ascending sequence is defined as the following formula.

$$F_i = \frac{i - 0.3}{n + 0.4} \quad (4)$$

Figure 6 shows the Weibull plot of the tensile strength in the bamboo fiber that was obtained based on formulas (3) and (4). Two parallel sets of data were observed in the plot. They are derived from two groups of data in Fig. 4. The group of small cross-sectional area in Fig. 4 corresponds to upper line in Fig. 6 (the correlation coefficient was $R = 0.973$ by linear regression analysis), and the group of large cross-sectional area corresponds to lower line (same $R = 0.985$). However, all bamboo fiber regardless of size of cross-sectional area is used in this study, overall analyzed values are used in following analysis. When linear regression analysis was conducted on this plot, the correlation coefficient was $R = 0.887$. Table 1 shows the Weibull parameter obtained from the linear regression equation (slope = 3.11 and intercept = −60.4) based on formula (3), and the mechanical characteristics of the bamboo fiber obtained from the experimental results. Shape parameter m shows the same values as that of both bamboo fiber and kenaf hemp fiber that have been reported until now [18, 19], and they show lesser values and significantly vary as compared to industrial materials such as glass fiber [20].

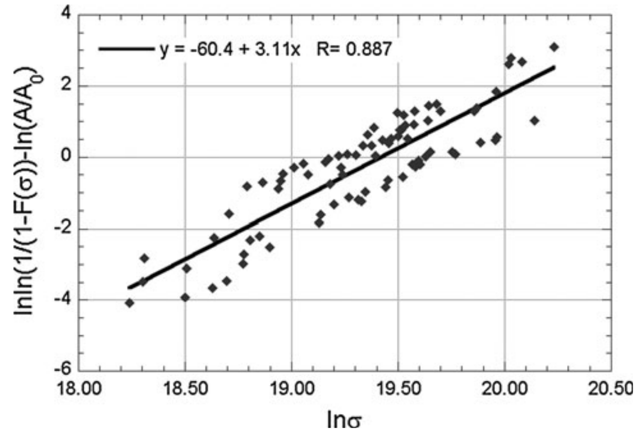

Fig. 6 Weibull plot in bamboo fiber

Table 1 Mechanical properties and Weibull parameters of bamboo fiber

Number of samples	80
Average cross-sectional area (mm^2)	0.099 ± 0.074
Average tensile strength (MPa)	274.7 ± 114.7
Average Young's modulus (GPa)	15.8 ± 4.2
Shape parameters m	3.112
Scale parameter σ_0 (MPa)	272

Tensile characteristics of layered molding

Figure 7 shows the stress–strain curves of the layered moldings of the tensile test for each bamboo fiber weight fraction. The results of the moldings of 100% wood flour and 100% bamboo fiber are also shown. The initial slope of the curve increased as the bamboo fiber fraction increased, coming close to the curve of the molding with 100% bamboo fiber. Table 2 shows the tensile test results. Tensile strength as well as tensile Young's modulus increased with the increase in the bamboo fiber fraction. The tensile strength of the seven-layered molding with 75% bamboo fiber became 5.8 times stronger than the molding with 100% wood flour. This result shows that the reinforcement

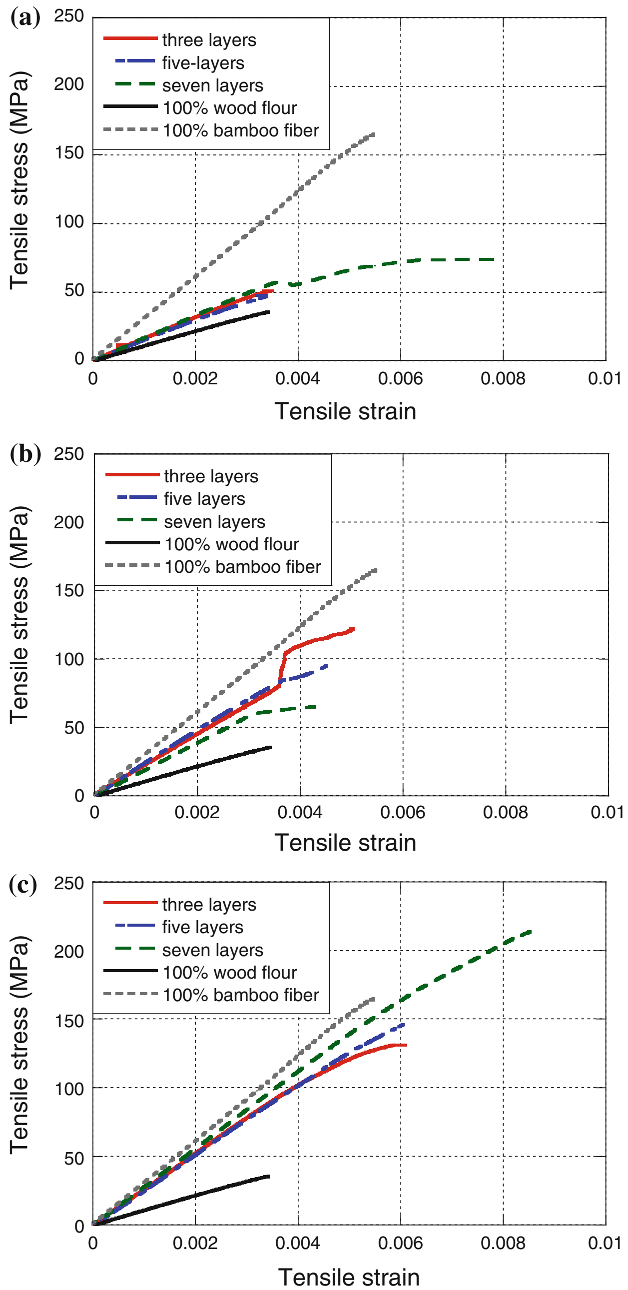

Fig. 7 Typical tensile stress–strain curves of moldings. Fiber weight fraction is **a** 25%, **b** 50%, **c** 75%

of moldings with bamboo fiber was effective. In addition, the tensile strength became approximately 1.5 times greater when compared to the tensile strength (135 MPa) [21] of the fiber direction of beech wood. There existed almost no difference due to layer construction differences in the tensile Young's modulus, resulting in low variations. The tensile Young's modulus of the molding with 100% bamboo fiber was approximately twice the average Young's modulus of the bamboo fiber (15.8 GPa, as shown in Table 1). From this result, the bamboo fibers are considered to be adhered each other, even though they were only bamboo fibers, enhancing the rigidity compared to the case of single fibers. On the other hand, it is likely that the tensile strength varied significantly, which was greatly affected by variation in the strength of the bamboo fiber itself. When focusing on the differences in layer construction, the tensile strength increased with the increase of the number of layers in the molding with 75% bamboo fiber. Statistical comparisons of the results were made using one-way analysis of variance (ANOVA). Significant differences ($P < 0.05$ and $P < 0.01$) between each molding were analyzed by Scheffe's F test. Analytical results are shown in Table 3 (Young's modulus) and Table 4 (tensile strength).

Figure 8 shows SEM microphotographs of specimens after tensile test. The specimen of the molding with 100% wood flour ruptured smoothly. On the other hand, as with the case of the fiber tensile test, thin fibers were cut at various portions in the molding with 100% bamboo fiber, which seemed to indicate that the fibers were weak in their adhesive property. From the ruptured surface of the five-layered molding, it was revealed that the border between the wood flour layer and the bamboo fiber layer was not clear and the wood flour penetrated into the diameter of half or one single bamboo fiber.

Analysis of the tensile characteristics of layered moldings

We tried to model the tensile characteristics of thermoplastic layered moldings made of bamboo fiber and wood flour by applying the experimental results obtained.

Young's modulus

It has been known that the rule of mixture, which can be expressed by the following formula, is generally applied to Young's modulus, where the reinforcement layer (bamboo fiber) and the basic material layer (wood flour layer) align parallel to load as seen in this experiment [20].

$$E_L = E_f V_f + E_m (1 - V_f) \quad (5)$$

where E_f and E_m are the elastic modulus of the fiber and wood flour layer, respectively, and V_f is the fiber volume fraction. It is right to thinking that fiber weight fraction is equal to fiber volume fraction because the density of the layered molding is independent of the fiber weight fraction as shown in Table 2. Figure 9 shows predicted Young's modulus based on the rule of mixture and the experimental values. As shown in this figure, the predictions show values closer to the experimental values, and it is likely that Young's modulus of this layered molding conforms to the rule of mixture.

Table 2 Mechanical properties of moldings

Content of bamboo fiber (%)	Number of layers	Density (g/cm^3)	Young's modulus in tension ± SD (GPa)	Tensile strength ± SD (MPa)
0	1	1.45	10.4 ± 0.7	37.5 ± 3.2
25	3	1.44	16.9 ± 1.8	62.4 ± 16.9
25	5	1.44	14.8 ± 2.4	44.7 ± 7.6
25	7	1.44	15.6 ± 1.1	66.9 ± 13.8
50	3	1.43	22.1 ± 2.1	81.6 ± 39.1
50	5	1.42	23.4 ± 2.3	121.3 ± 31.4
50	7	1.45	19.5 ± 0.9	61.1 ± 21.3
75	3	1.42	25.8 ± 1.8	112.6 ± 50.0
75	5	1.43	25.3 ± 1.4	142.8 ± 30.9
75	7	1.42	27.4 ± 1.0	217.0 ± 28.3
100	1	1.44	29.6 ± 3.9	158.5 ± 62.8

Table 3 Statistic analysis of Young's modulus

	Number of layers	100% wood flour	25%			50%			75%			100% bamboo fiber
			3	5	7	3	5	7	3	5	7	
100% wood flour			**	-	*	**	**	**	**	**	**	**
25%	3			-	-	*	**	-	**	**	**	**
	5				-	**	**	*	**	**	**	**
	7					**	**	-	**	**	**	**
50%	3						-	-	-	-	*	**
	5							-	-	-	-	**
	7								**	**	**	**
75%	3									-	-	-
	5										-	*
	7											-
100% bamboo fiber												

* $P < 0.05$, ** $P < 0.01$;
– no significant difference

Table 4 Statistic analysis of tensile strength

	Number of layers	100% wood flour	25%			50%			75%			100% bamboo fiber
			3	5	7	3	5	7	3	5	7	
100% wood flour			-	-	-	-	*	-	*	**	**	**
25%	3			-	-	-	-	-	-	*	**	*
	5				-	-	-	-	-	**	**	**
	7					-	-	-	-	-	**	*
50%	3						-	-	-	-	**	-
	5							-	-	-	**	-
	7								-	*	**	**
75%	3									-	*	-
	5										-	-
	7											-
100% bamboo fiber												

* $P < 0.05$, ** $P < 0.01$;
– no significant difference

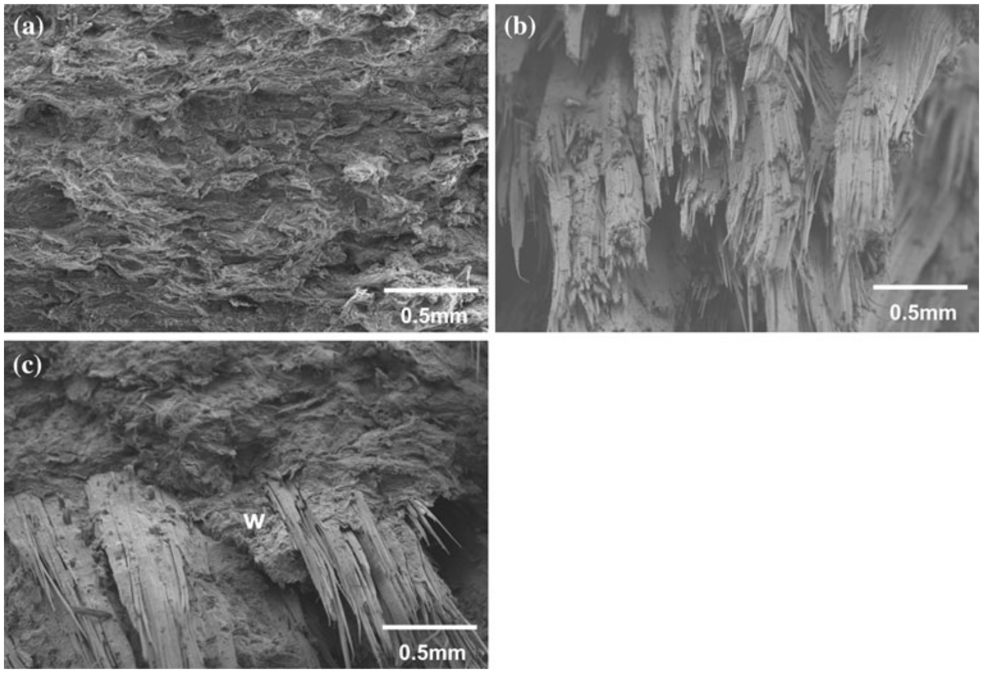

Fig. 8 SEM photomicrographs of specimens after tensile test. **a** 100% wood flour, **b** 100% bamboo fiber, **c** 50%-5 layers. *w* wood penetrate into bamboo fiber layer

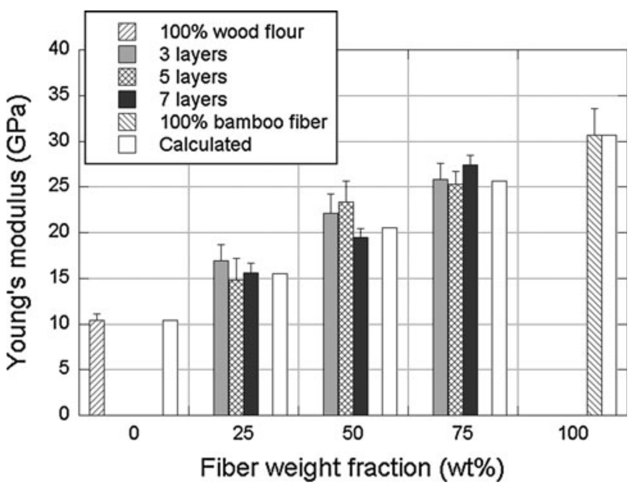

Fig. 9 Young's modulus of molding and prediction

Tensile strength

Next, we predicted the tensile strength. Where the strength distribution of each individual fiber follows the Weibull distribution, the breaking strength of the bundle of these individual fibers can be predicted by applying the following formula obtained by Coleman [22].

$$\frac{\sigma_b}{\overline{\sigma}} = \left(\frac{1}{me}\right)^{\frac{1}{m}} - \frac{1}{\Gamma(1+1/m)} \tag{6}$$

where σ_b is the breaking strength of a bundle of fibers, $\overline{\sigma}$ is the average strength of bamboo fiber, m is the shape parameter, e is the base natural logarithm, and Γ is the Gamma function. When σ_b is calculated by assigning m and $\overline{\sigma}$, which were obtained in the previous section, to the above formula, the breaking strength of the bundle of bamboo fibers used in this experiment is estimated as 152.9 MPa. This prediction is almost compatible with the average tensile strength, 158.5 MPa, of the molding with 100% bamboo fibers as shown in Table 2. Therefore, the molding process did not have any impact on the molding tensile strength.

Next, the tensile strength of the layered moldings was predicted. This tensile strength can be expressed by the following formula if the rule of mixture is applied.

$$\sigma_{uts} = \sigma_f V_f + \sigma_m (1 - V_f) \tag{7}$$

where σ_f and σ_m are the tensile strength of the fiber and the wood flour layer, respectively, and V_f is the fiber volume fraction. The tensile strength of the layered moldings with each fiber volume fraction was predicted by assigning σ_b (152.9 MPa), which was obtained from formula (6), to σ_f, and assigning the average tensile strength (37.5 MPa) of the molding of 100% wood flour shown in Table 2 to σ_m. Figure 10 shows predicted values and the experimental values. The experimental value of the tensile strength of the molding with 25% bamboo fiber showed almost the same value as the predicted value. The average tensile strength of the three-layered moldings showed values closer to the predicted values in each fiber volume fraction; however, a tendency was observed that when the

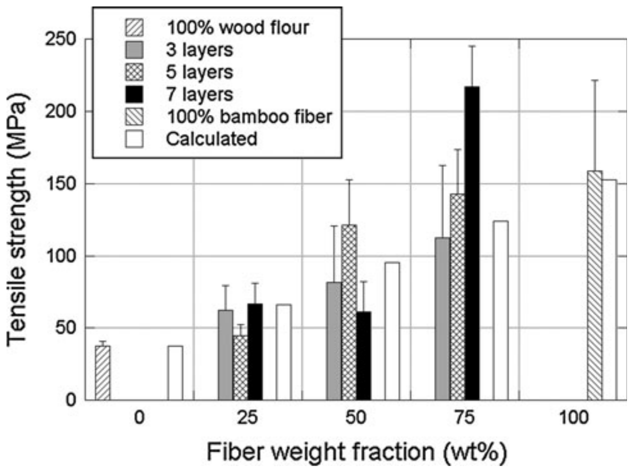

Fig. 10 Tensile strength of molding and prediction

percentage of bamboo fiber and the number of layers increased, the experimental values exceeded the predicted values. This result is probably due to the strength prediction made by applying formula (7) that does not consider the interactions between the wood flour layer and the bamboo fiber layer. Hence, we assumed that the mixture layer, in which the wood flour layer and bamboo fiber layer mix together is in between both these layers. Where the volume fraction of each of the genuine bamboo fiber layer, mixture layer, and genuine wood flour layer is V_f', V_{mix}', and V_m', respectively, and where the tensile strength of the mixture layer is σ_{mix}^*, formula (7) can be re-expressed as the following formula.

$$\sigma_{uts} = \sigma_f V_f' + \sigma_{mix}^* V_{mix} + \sigma_m V_m' \quad (8)$$

However, this formula follows the conditions below.

$$V_f' + V_{mix} + V_m' = 1 \quad (9)$$

Specifically, the tensile strength of the mixture layer, σ_{mix}^*, should be examined. In this respect, Curtin defined the critical fiber stress, σ_c, with formula (10), assuming that stress owned by the ruptured fiber can be similarly owned by the rest of the fibers when the rupture of the fiber is caused by a long-fiber reinforced composite. In addition, Curtin indicated that the composite strength, σ_{uts}', could be approximated by the following formula (11) [23].

$$\sigma_c = \left(\frac{\sigma_0^m \tau L_0}{r}\right)^{1/(m+1)} \quad (10)$$

$$\sigma_{uts}' = V_f \sigma_c \left(\frac{2}{m+2}\right)^{1/(m+1)} \left(\frac{m+1}{m+2}\right) + (1 - V_f)\sigma_y \quad (11)$$

where r is the fiber radius, τ is the fiber and matrix interfacial shear strength, σ_y is the tensile stress of the base material, and V_f is the fiber volume fraction. Where the tensile strength of the mixture layer of our layered molding, σ_{mix}^*, follows formula (10) and (11) shown by Curtin for obtaining the strength of the long-fiber reinforced composite, formula (8) is expressed as the following formula.

$$\sigma_{uts} = \sigma_f V_f' + \left(\frac{\sigma_0^m \tau L_0}{r}\right)^{1/(m+1)} \left(\frac{2}{m+2}\right)^{1/(m+1)} \times \left(\frac{m+1}{m+2}\right) V_{mix-f} + \sigma_m V_{mix-m} + \sigma_m V_m' \quad (12)$$

where V_{mix-f} and V_{mix-m} are the volume fraction of each of the bamboo fiber and wood flour in the mixture layer. Each volume fraction can be expressed as follows when organized.

$$V_f' + V_{mix-f} = V_f, \quad V_{mix-m} + V_m' = 1 - V_f \quad (13)$$

Therefore, formula (12) can be organized as follows.

$$\sigma_{uts} = \sigma_f (V_f - V_{mix-f}) + \left(\frac{\sigma_0^m \tau L_0}{r}\right)^{1/(m+1)} \left(\frac{2}{m+2}\right)^{1/(m+1)} \times \left(\frac{m+1}{m+2}\right) V_{mix-f} + \sigma_m (1 - V_f) \quad (14)$$

We need to learn the fiber and matrix interfacial shear strength, τ, in the mixture layer, to examine whether we should apply formula (14) to the results of this experiment; however, it is impossible for us to obtain this value immediately at this time. In other words, we are unable to directly verify formula (14). Therefore, in contrast, assuming that the tensile strength of our layered moldings follows formula (14), the estimation of the fiber and matrix interfacial shear strength, τ, was attempted. τ can be expressed based on formula (14) as follows.

$$\tau = \left(\frac{\sigma_{ult} - (\sigma_f (V_f - V_{mix-f}) + \sigma_m (1 - V_f))}{\left(\frac{2}{m+2}\right)^{1/(m+1)} \left(\frac{m+1}{m+2}\right) V_{mix-f}}\right)^{(m+1)} \cdot \frac{r}{\sigma_0^m L_0} \quad (15)$$

Figure 11 shows the estimated values of the interfacial shear stress where it is assumed that the thickness of the mixture layer ranged from 0 to 0.5 mm. τ did not converge into one value since the tensile strength of the layered moldings varied significantly. However, τ could be estimated as 10.9 MPa based on the equation of approximation curves, for example, where the mixture layer thickness was assumed to be 0.2 mm and the interfacial shear stress was assumed to be 5.1 MPa and 0.3 mm. These thicknesses show that there existed a situation where wood flour penetrated into the thickness of half of or one bamboo fiber, and these values can be probably regarded as valid judging from what was observed in Fig. 8. The mechanism that caused the interfacial shear stress estimated here is probably due to the following fact: the molecules of

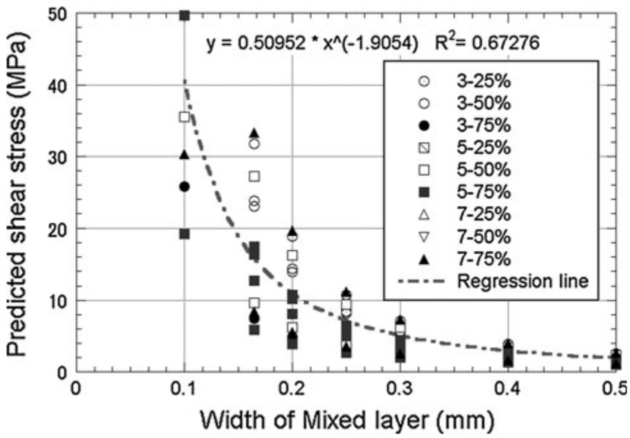

Fig. 11 Prediction of interfacial shear stress

hemicellulose and lignin within the wood flour were made smaller because of steaming, and they were plasticized by the compression molding process, resulting in adherence to the bamboo fiber. It is likely that the interfacial shear stress is the same with the matrix shear stress [24]. We can consider that it is possible to estimate the matrix shear stress by applying the method attempted above.

Conclusion

In this research, we made an attempt to improve the mechanical properties of wood moldings by utilizing bamboo fibers as reinforcing material. The results showed that where the bamboo fiber fraction was 75%, the tensile strength became approximately 3.8 to 5.8 times greater, and tensile Young's modulus became approximately 2.5 times greater than the moldings with 100% wood flour. This result confirmed that bamboo fiber is capable of improving the mechanical property of wood moldings. In addition, we tried to model the tensile characteristics of thermoplastic layered moldings made of bamboo fiber and wood flour by applying the experimental results obtained. This attempt showed that tensile Young's modulus follows the rule of mixture. Where it is assumed that Curtin's model could be applied in respect to the tensile strength, the interfacial shear stress between the bamboo fiber layer and the wood flour layer would be estimated as about 5 to 11 MPa when the mixture layer thickness was assumed to be 0.2–0.3. It was revealed that the wood flour penetrated into the diameter of half or one single bamboo fiber by SEM observation.

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