Microscopic Characterization of Modified Phenol-Formaldehyde Resin Penetration of Bamboo Surfaces and its Effect on Some Properties of Two-Ply Bamboo Bonding Interface

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The bonding interface between bamboo elements and adhesives is presumed to be significantly influenced by the degree of adhesive penetration into the porous network of interconnected cells of bamboo surfaces. In the study presented here, the average depth and effective depth of phenol-formaldehyde resin (PF) modified by different contents of lower-molecular weight (LMW) PF on bamboo surface were evaluated, making use of fluorescent microscopy characterization. The shear distribution at the bonding interface was measured by means of electronic speckle pattern interferometry (ESPI), along with tensile strength measurements, to determine the shear strain distribution on a macroscopic scale. This research combined macroscopic mechanical properties with microscopic interfacial mechanical properties, and it was found that PF modified with 10% LMW PF performed better than other modified PF. Moreover, it was assumed that the results of this study would influence the choice of bamboo-specific adhesives under different strain conditions.

Keywords: Lower-molecular weight modified phenol-formaldehyde resin; Bamboo; ESPI; Bonding interface

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INTRODUCTION

Fundamental research on bamboo adhesives and adhesion on bamboo surface have been characterized by indeterminacy because of poorly characterized initial and final conditions (Aydin 2004; Smith *et al.* 2002; Sonnenschein *et al.* 2005, 2009). Bamboo, with its fast growing rate, high strength, and stiffness, is one of the most suitable forest materials to be used for furniture and construction (Wang and Ren 2007). However, the interaction of a bamboo surface with an adhesive is inherently difficult to characterize. In addition to factors such as surface energy and wetting, surface roughness, and weak boundary layer formation (Kamke 2007), the diffusion of adhesive compounds into the bamboo cell wall is one factor that has a potential influence on adhesive bonding performance. While penetration of resin in the sense of filling of cell cavities - not cell walls - has received considerable attention (Gindl *et al.* 2004a,b), many studies, using a variety of analytical techniques, have examined the adhesive penetration and mechanical properties of the interface between wood and the adhesive (Modzel *et al.* 2011; Valla *et al.* 2011). However, there are few studies of resin penetration of bamboo surfaces and its effect on the bonding interface between bamboo and resin, other than phenol formaldehyde modified by PVA on bamboo (Guan *et al.* 2012, 2013), though adhesion and properties of low molecular weight phenol formaldehyde-treated plybamboo samples have been studied (Anwar *et al*. 2009, 2012).

Water-soluble phenol-formaldehyde resol resin is commonly used in the bamboo-based panels industry, such as recombined bamboo timber (Japan), laminated bamboo lumber, and bamboo mat plywood, and its utility for many bamboo-based applications is beyond question (Guan *et al.* 2005, 2006). However, questions have arisen regarding whether or not the formation of an interpenetrating network by PF penetration on bamboo surface is achieved when compared with that in the case of wood. This is because bamboo is intrinsically lacking of horizontal organization such as wood rays and so on. Also, bondline thickness and bondline strength formed by adhesive penetration have a great influence on a composite material's shear strength (Serrano and Gustafsson 1999; Tomblin *et al.* 2002). All of these unusual and notable factors affect the widespread use of PF in bamboo-based engineering materials.

Sufficient penetration of wood surfaces is considered important for good bond formation, but the relative importance between penetration into lumens and into cell walls is not normally discussed. Especially for bamboo structures, the low viscosity adhesive PF is normally better for the wetting and adhesion, but the adhesive can also be thin, leading to overpenetration into the bamboo, which produces a starved bondline that is the weak link (Frihart 2005). However, what is interesting is that the penetration into cell walls depends upon the molecular size of the adhesive components, and it is easier for a lower viscosity adhesive to penetrate into cell walls, thus changing a sharp bamboo-adhesive interaction into a more diffuse boundary layer, making the role of primary and secondary chemical bonds at the adhesive-bamboo interface less important (Sernek *et al.* 1999). That is to say, for lack of horizontal organization of bamboo, it is important to consider more about LMW resin penetration into cell walls instead of just cell lumens and to avoid adhesive overpenetration of the bamboo surface at the same time.

In this study, the intention was to manufacture two-ply bamboo panels with PF modified by different LMW PF contents, and then attempt to investigate the morphology of adhesive bondline on bamboo surface by the method of fluorescence microscopy. The micro-scale strain distribution and strain concentration in the vicinity of the bonding interface of two-ply bamboo panels was also tested by means of ESPI, as well as the bonding line shear strength, to suggest a possible bonding interface model for the interaction of modified PF with the bamboo matrix.

EXPERIMENTAL

Sample Preparation of Bamboo Bonding Interface

The standard normal PF resin and LMW PF were prepared according to formulations already reported by manufacturers in the laboratory. The number-average molecular weight of the two resins were tested and found to be 2036 and 810, respectively, with different dispersion coefficients of 1.9578 and 1.1183, respectively. Then, according to different proportions (10:0.5, 10:1, and 10:2), these two resins were mixed together.

Modified adhesives were used to glue 2-ply Moso bamboo (*Phyllostachys pubescens*, 4 years old from Zhejiang bamboo factory, China) panels parallel to each other (each ply is 5 mm). The specimens were all cured for 15 min in a press at 2 MPa and at an ambient temperature of 140 °C. After curing, specimens were maintained in a condition room at 65% RH and 20 °C for 1 week until constant weight was attained.

Sample Sections and Fluorescent Microscopy

To gain a better understanding of the morphology of adhesive penetration on the bamboo surface, the bonding interface was stained by fluorescent dye (Guan *et al.* 2012). A cross section across the area of interest on each sample was created using a Reichert-Jung Ultracut E microtome equipped with a sapphire knife at room temperature after the sample was softened by soaking. The cross section was then dehydrated using graded ethanol (30%, 50%, 75%, 95%, and 100%), and then 0.5% fluorescent dye Toluidine Blue O was added dropwise for 30 min. Each sample was then washed in distilled water twice, glycerin was added, and the sample was covered for observation. Fluorescent microscopic observations were made, and images were recorded digitally. Penetration observations of the bamboo surface, such as depth, were studied and analyzed by Image J software.

ESPI Measurement of Bamboo Bonding Interface

To monitor shear displacement on the surface of the two lap joint specimens, shear testing corresponding to DIN EN 302-1-2004 was performed on a universal testing machine equipped with TS-S1-1XP ESPI system (Guan *et al.* 2012). The fundamental principles of the ESPI technique were explained in detail in a previous paper (Muller *et al.* 2005). With the optical set-up used here, the size of the field of view (FOV) observed with ESPI was 44×35 mm^2, and the working distance between camera and specimen was about 300 mm. Specimens were pre-loaded to 50 N and then strained in 14 steps of 5 N. The shear testing was conducted twice in two directions X and Y, since this kind of ESPI caught the deformation only from one direction, so each deformation of 2-ply bamboo should be controlled in its elastic stage. At each displacement step, a interference fringe image of the observed field of view was taken. For in-plane measurements the recorded intensity $I_{(X1,\,X2)}$ could be written as Eq. 1,

$$I_{(X1,X2)} = I_{R(X1,X2)} - I_{D(X1,X2)} = 2\sqrt{I_{r(x1,x2)} I_{o(x1,x2)}} COS\Delta\Phi_{(x1,x2)} \qquad (1)$$

where $I_{R(X1,X2)}$ and $I_{D(X1,X2)}$ are the intensity of the speckled images before and after deformation, $I_{r(x1,x2)}$ and $I_{o(x1,x2)}$ are the intensity of the object and reference beam, and $\Phi_{(x1,x2)}$ is the phase difference due to displacement of the sample surface.

A series of steps can be summed to measure high displacements and deformations, the strain components, axial strain ε_{11}, transversal strain ε_{22}, and shear strain ε_{12}, could be calculated from deformation field according to standard mechanical Eq. 2.

$$\varepsilon_{11} = \frac{\Delta u_1}{\Delta x_1} \quad \varepsilon_{22} = \frac{\Delta u_2}{\Delta x_2} \quad \varepsilon_{12} = \frac{1}{2}(\varepsilon_{11} + \varepsilon_{22}) = \frac{1}{2}\left[\frac{\Delta u_1}{\Delta x_1} + \frac{\Delta u_2}{\Delta x_2}\right] \qquad (2)$$

The displacement maps were computed by summing up information from all 14 displacement steps. Each resin with 5 specimens were tested at least five times until all the specimens showed similar results, and then only one specimen for calculation was chosen.

Shear Strength Testing

Shear strength testing was carried out to measure the bonding strength of modified adhesive at the bamboo surface. Shear specimens were manufactured experimentally in accordance with DIN EN 302-1-2004 with a total length of 150 mm, a width of 20 mm, and a thickness of 10 mm (each ply is 5 mm). Incisions were made by using a circular saw to detect the notches towards the glue line, allowing for a tested bond length of 10 mm, as suggested by DIN EN 302-1-2004. Shear testing was done on a universal testing machine applying the load at a speed of 2.5 mm/min. A sufficient number of specimens to get 10 valid numbers were tested in dry conditions referring to A1 and tested immediately after an obligatory 7 days in standard atmosphere [20/65] and wet conditions, respectively; this permitted a bamboo failure rate of nearly 30%. Wet conditions referred to A4: 1) 6 h soaking in boiling water; 2) 2 h soaking in water at (20 $\pm$5) ^{o}C; and 3) Samples tested in the wet state.

RESULTS AND DISCUSSION

Fluorescent Characterization of Bamboo Bonding Interface

In some previous papers (Kamke 2007; Modzel *et al.* 2011), it has been determined that the fluorescence of wood materials in a thin section of phenol-formaldehyde bondlines could be suppressed by an 0.5% aqueous solution of Toluidine Blue O to yield good color contrast. The interface is commonly where bamboo cells and resins exist together, while the bondline is the area where two parts of the matrix are glued by resins.

Figure 1 shows different fluorescent pictures of the bamboo bonding interface with PF modified by different content of LMW PF. The parts of the bonding interface where bamboo parenchyma were mainly located were chosen in order to avoid higher-strength vascular bundles squeezing resins unnecessarily.

Bitmaps of bamboo bonding interface could be seen in the left side of the fluorescent pictures (Fig. 1), and the adhesive effective penetration (EP) and average penetration (AP) could also be calculated from these bitmaps according to standard Eqs. 3 and 4 (Ma 2009),

$$EP = \frac{\sum_{i=1}^{n} A_i}{x_o} \tag{3}$$

$$AP = \frac{\sum_{i=1}^{5} y_i}{5} \tag{4}$$

where *EP* is the effective penetration (µm), *AP* is the average penetration (µm), *n* represented the resin spot numbers, A_i is the “i”th area of penetration (µm^2), x_o represents the glueline length (420 to 480 µm), and y_i is the farthest penetration distance (µm). The results of penetration depth are shown in Table 1. In the bamboo bonding interface modified by PF with LMW PF, there was no obvious variation trend in adhesive AP, but there was a slight downtrend in adhesive EP when the proportion of LMW PF was increased. Micromorphology of the glueline appeared to change more irregularly with the increase of LMW PF ratio, as shown in Fig. 1, and the resin distribution looked especially chaotic at 20% of LMW PF.

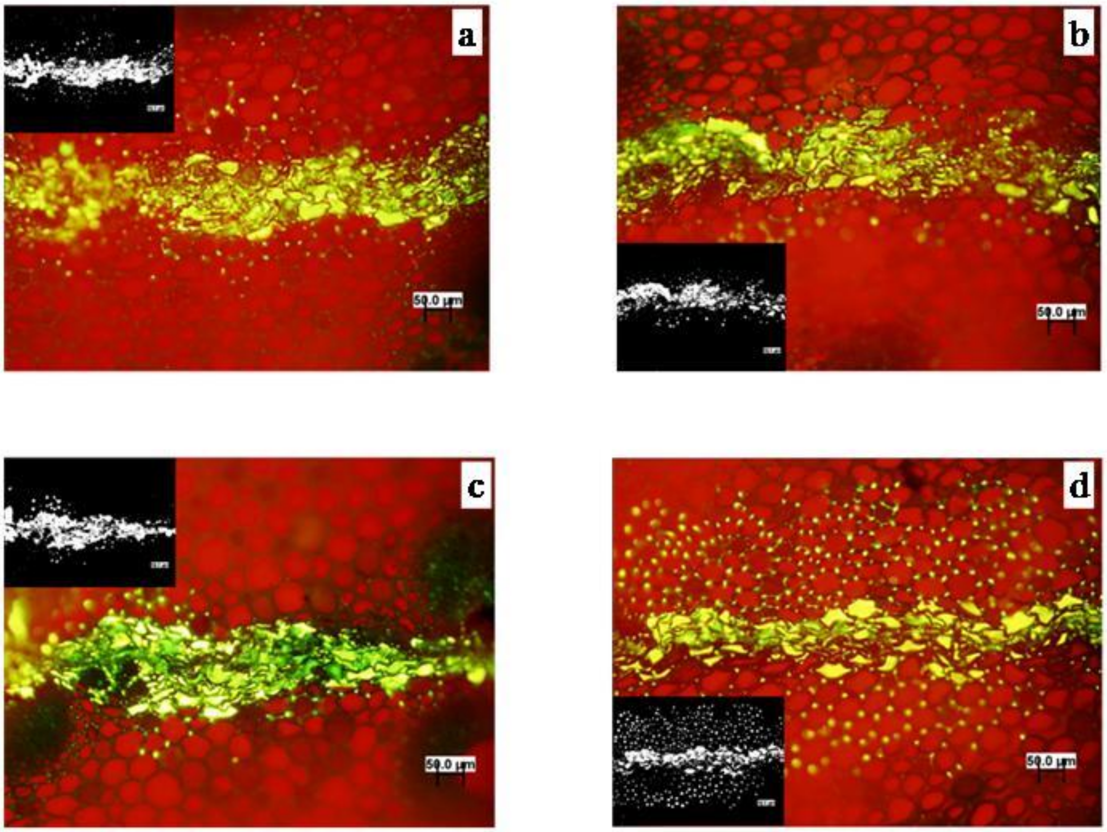

Fig. 1. Fluorescent photomicrograph of the bamboo/PF bonding interface with bitmap in the left corner: (a) bamboo/PF without modification; (b) bamboo/PF modified by 5% LMW PF; (c) bamboo/PF modified by 10% LMW PF; (d) bamboo/PF modified by 20% LMW PF

Table 1. AP and EP of Modified PF with LMW PF in Bamboo Bonding Interface

Materials	Proportion of LMW (%)	AP			EP		
		Average Value (μm)	Standard Deviation (μm)	Variable Coefficient (%)	Average Value (μm)	Standard Deviation (μm)	Variable Coefficient (%)
Two-ply bamboo	0	164.14	41.95	25.56	83.23	9.97	12.12
	5	144.67	31.03	21.45	77.39	15.04	19.43
	10	154.38	19.05	12.34	77.12	14.66	16.27
	20	142.95	36.38	25.45	59.68	13.28	22.26

In many previous wood bonding interface studies, adhesive effective penetration depth tended to increase in hot pressing when the viscosity of adhesive was reduced (White 1997), but the opposite tendency existed in the bamboo bonding interface. It was assumed that, in the adhesive coating process of the bamboo surface, linear, small, low-molecular weight molecules preferred to enter into bamboo surface cell walls without the guidance of wood tray, and in addition, were obstructed by the aspirated pits. This would lead to the average bonding interface thickness decreasing.

Moreover, on the bamboo surface, the variation tendency of AP was more significant than that of EP, which meant that part of LMW PF was subjected to an extraordinary load by hot steam pressure (Brady and Kamke 1988) and reached almost the same distance on the bamboo surface, except for the rest of the parts, which entered into cell walls.

Shear Strain Distribution of Bamboo Bonding Interface

As can be seen from Figs. 2 and 3, two-dimensional strain distribution around the bonding interfaces and shear strain along the bondline were measured by ESPI.

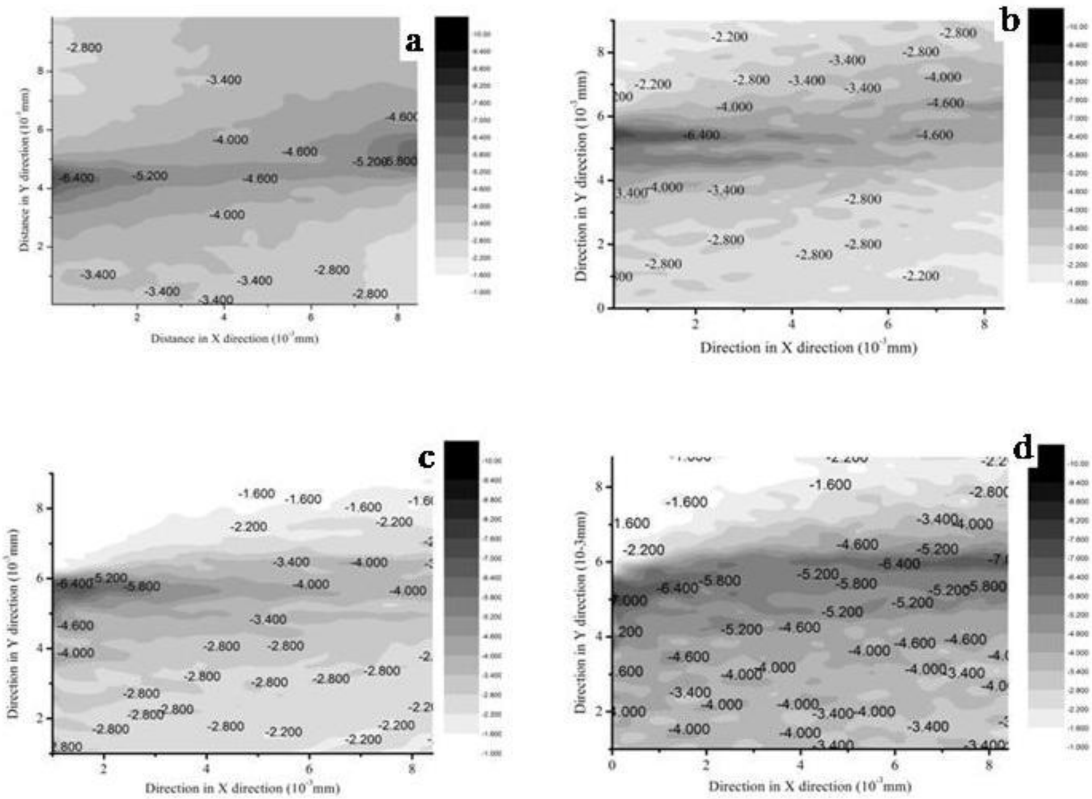

Fig. 2. Strain distribution of the bamboo/PF bonding interface with observed fields of view: (a) bamboo/PF without modification; (b) bamboo/PF modified by 5% LMW PF; (c) bamboo/PF modified by 10% LMW PF; (d) bamboo/PF modified by 20% LMW PF

For all modified PF, the ESPI measurements of single lap-joint samples showed that the normal and shear strains along the bondline were comparably low in the middle of the overlapping area but increased steeply at both ends of the glued area. These increased values of the normal and shear strains were localized in the thin bondline itself and in an area not extending further than 1 mm from the bondline.

In the region of the bonding interface, strains from 4.6×10^{-3} to 6.4×10^{-3} mm were observed for both the PF sample and modified PF with 5% LMW PF sample, and compared with the strain from 4.0×10^{-3} to 6.4×10^{-3} mm for modified PF with 10% LMW PF sample, and strain from 5.8×10^{-3} to 7.0×10^{-3} mm for modified PF with 20% LMW PF sample.

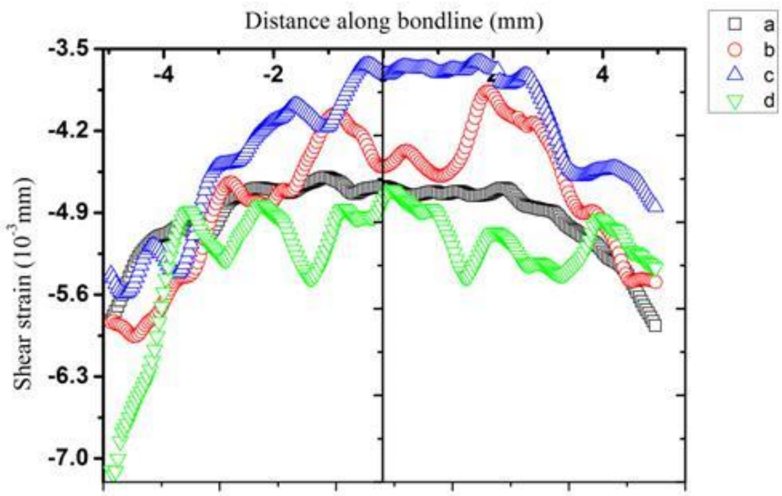

Fig. 3. Values of strain along the bondline with different bamboo/PF bonding interface: (a) bamboo/PF without modification; (b) bamboo/PF modified by 5% LMW PF; (c) bamboo/PF modified by 10% LMW PF; (d) bamboo/PF modified by 20% LMW PF

In some previous studies, it was noted that LMW resin could penetrate cell walls and change a sharp wood-adhesive interaction into a more diffuse boundary layer. The micro-channels penetration into bamboo might also serve as a nano-mechanical interlock and cause the formation of interpenetrating polymer chains, which would effectively improve its physical properties (Sernek *et al.* 1999). In the bamboo bonding interface, which lacks transversal organizations, it was more possible for LMW PF to penetrate into cell walls on the bamboo surface in hot pressing and thus stabilize cell walls and reduce interfacial stress concentration. However, the crosslinking degree and polymer chains of LMW PF were not as good as those of a normal PF sample (Schmidt and Frazier 1988), as the apparent chaos in the bonding interfacial regions increased with increasing percentage of LMW PF. According to Gindl's research (Gindl and Müller 2006; Gindl *et al.* 2005), for *in situ* polymerized adhesives, strain distribution along the bondline was related to the material's MOE, which meant that the strengthening of cell walls might influence the strain distribution in the bonding interface. In the work presented here, 5% LMW PF sample was not enough to sufficiently fill cell walls, while 20% LMW PF sample was too excessive to fill cell walls, which then led to a reduction in bondline thickness and a weakening in mechanical interlock of interfacial bondline. Moreover, additional LMW PF mixed in the bondline caused strain distribution around the bondline regions to become chaotic and the added break of strain transmission would lead to slippage of the bamboo bonding interface. As a result, the 10% LMW PF sample performed best, as the strain value in the bonding interface was smaller and the distribution of the chaotic region was better when compared with other samples.

Shear Strength of Two-Ply Bamboo Panels

Clear differences in dry shear strength and wet shear strength were observed in specimens glued by normal PF with different content of LMW PF. As depicted in Fig. 4, with the percentage of LMW PF increasing, dry shear strength reached a maximum of

14.08 MPa with a LMW PF content of 10%, whereas the minimum dry shear strength was 8.03 MPa with a LMW PF content of 20%. With regard to wet shear strength, a regular liner variation was observed. With the percentage of LMW PF increasing, wet shear strength continued declining from the maximum of 10.00 MPa to a minimum of 7.11 MPa.

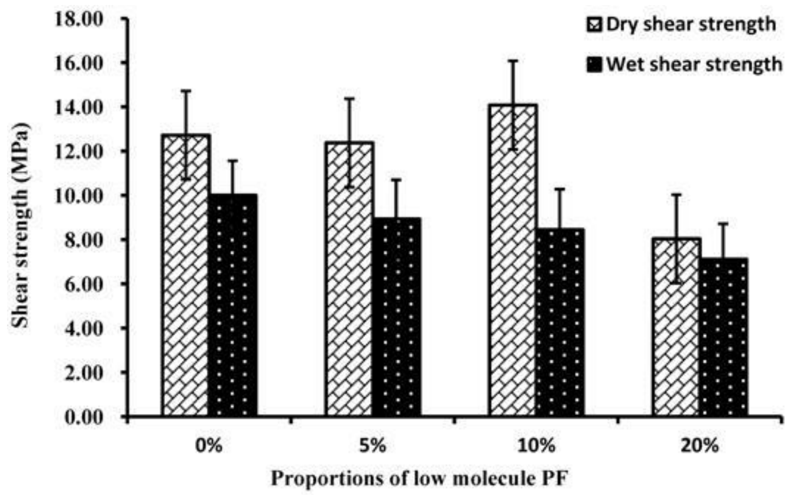

Fig. 4. Shear strength of bamboo/PF bonding interface with LMW PF in various proportions

It has been discussed previously that, just on bamboo surfaces, moderate LMW PF penetrating into cell walls could effectively increase bamboo bonding strength to some extent. As fluorescent characterization and ESPI measurement have shown before, not only did 10% LMW PF addition meet the requirement of mechanical interlock of bamboo bonding interface penetration depth, but also the LMW PF could stabilize the cell walls to form a bridge and change its mechanical properties. As a consequence, it was expected that the tendency of dry shear strength was very similar to that of ESPI measurements.

As moisture levels change, cell walls expand or contract, and absorbed water might also disrupt hydrogen bonding between the wood and adhesive (Frihart 2007). It could be seen that wet shear strength of bamboo bonding interface declined with the percentage of LMW PF increasing. According to the adhesive formulation and GPC testing, the extensive crosslinking degree of LMW PF was not better than that of normal PF in hot pressing. In addition, the needless formaldehyde and small molecules like CH_2O and H_2O produced in the solidification process (Gabilondo *et al.* 2011) would gain more energy under wet conditions and then continued striking into the low-density parts of crosslinked network, which was also the interspace of molecule chain entanglements (Serrano and Gustafsson 1999). Such effects possibly led to strain concentration in bamboo bonding interface, which might be the main reason why the wet shear strength of bamboo sample was reduced so fast.

CONCLUSIONS

1. In summary, this article has presented results on measurements of the average depth and effective depth of modified PF penetration in the bamboo bonding interface using

fluorescence microscopy. Combined with strain distribution by ESPI along the bondline and shear strength testing, significant differences were exhibited among the samples glued by general phenol-formaldehyde (PF) and those glued with different low molecular weight (LMW) PF contents.

2. The results showed that not only did 10% LMW PF addition meet the requirement of bondline thickness for mechanical interlocks needed on the bamboo surface, but the penetration of adhesive into cell walls also changed its mechanical properties. This was demonstrated by ESPI measurement and shear strength.
3. Wet shear strength in particular declined significantly with increasing percentage of LMW PF.

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