

Article

The Optimization of Thermo-Mechanical Densification to Improve the Water Resistance of Outdoor Bamboo Scrimber

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Abstract: The water resistance of bamboo scrimber used in outdoor environments greatly affects its applications and lifecycle. Physical and chemical studies have been conducted to investigate the influence of the hot-pressing temperature during thermo-mechanical densification on the water resistance of outdoor bamboo scrimber. Investigated parameters included the failure mode of surfaces, the vertical density profile, and the change of chemical components, which provides theoretical support for optimizing bamboo scrimber for outdoor applications. Here, the vertical density profiles of bamboo scrimber were measured using an X-ray density profiler, and the response of cells and bonding interfaces of bamboo scrimber to water absorption were recorded by using extended depth-of-field 3D microscopy and field emission scanning electron microscopy (FE-SEM). The composition was evaluated by X-ray diffraction (XRD), Fourier-transform infrared (FT-IR) spectroscopy, and X-ray photoelectron spectroscopy (XPS) to investigate the effect of temperature on water resistance during thermo-mechanical densification. The water resistance of bamboo scrimber significantly improved as the temperature increased from 140 °C to 170 °C. The spring-back from the compressive deformation of cells and cracks was the main failure mode, and showed a negative correlation upon increasing the temperature. The moderate increase in cellulose crystallinity, the increase in the polymerization degree of the PF resin, and the thermal degradation of hemicelluloses explained the failure behavior of the bamboo scrimber at the molecular level.

Keywords: outdoor bamboo scrimber; thermo-mechanical densification; water resistance; hot-pressing temperature; cell deformation; cracks; chemical components



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1. Introduction

Bamboo is a low-cost, abundant natural material that grows quickly and can be harvested every 3–5 years. Its strength-to-weight ratio is greater than that of structural steel, and it is widely used in indoor and outdoor applications [1–3]. Bamboo and wood have similar microstructures and chemical compositions, but bamboo has better strength and toughness and better environmental and economic advantages [4,5]. Recently, tremendous efforts have been devoted to processing bamboo into sustainable, eco-friendly, cost-effective, and high-performance composite materials for the construction industry [6].

Bamboo scrimber is a novel bamboo fiber-reinforced composite designed to have more consistent mechanical and physical properties than conventional bamboo. It is widely used as flooring in outdoor applications, structural materials for bridges, and traffic guardrails. Bamboo scrimber was developed at the beginning of the 1980s and has been popularized and applied over the last 20 years. Three major factors during the manufacturing of bamboo scrimber are its density [7], resin content [8], and hot-pressing temperature [9]. The defibering degree and thickness of the veneer [10], mat formation [11], and the type and molecular weight of the resin [12] are also important. There has been much research into the influence of processing parameters on the various properties (dimensional stability, mechanical properties, bonding properties, etc.) of bamboo scrimber [7,12–15]. The density

and resin content have been the most studied, whereas the temperature has received less attention. Many studies have been accomplished by our research group, in which the temperature was controlled within the range of 140–150 °C. Therefore, this article is a supplementary study to this previous research [8,12–15]. Scrimber made from different species (*Populus* spp. [9,10,16,17]) and other types of engineering bamboo products has been researched to determine the influence of processing parameters on its properties. Bamboo strand lumber [18] and glulam [19] have received particular attention. Studies have revealed that water resistance is positively correlated with the resin content, and the mechanical properties are strongly correlated with density.

Bamboo is a heterogeneous cellular material [20] that is different from softwood and hardwood. Hot-pressing combines heat treatment and densification [21] and can be used to pretreat raw bamboo-based composites. The range of temperatures has been summarized in previous review articles [22–24], and generally ranges from 100 °C to 240 °C. This temperature range includes three stages: thermal treatment (<150 °C), thermal modification (150–240 °C), and thermal degradation (>240 °C). This covers the range of hot-pressing temperatures of composites. Therefore, the influence of various temperatures on the changes in chemical components (lignin, cellulose, and hemicelluloses) and mechanical properties of cell walls can be referenced [25]. Previous studies [26–30] have shown that increasing the treatment temperature promoted the decomposition of hemicelluloses and cellulose and increased the relative content of lignin. The hygroscopicity was decreased because the hydrophilic functional groups in hemicelluloses decomposed. The nano-mechanical properties of bamboo cell walls can be affected by cellulose's crystallinity and chemical composition.

Thermo-mechanical (TM) densification is an eco-friendly method to improve the mechanical properties of wood/bamboo [31]. It uses mechanical compression to reduce the void volume inside and between cells and impregnates pores with liquid chemicals [32,33]. The densification of bamboo-based composites increases their density and improves their mechanical strength [34], and the quality of the final product is affected by processing parameters such as temperature, pressure, and densification degree [35]. Compression and densification in the radial direction and softening during heating allow the cells to be densified and prevent fracture during compression [36,37]. The preset temperature must be higher than the glass transition temperature (T_g) of bamboo, to allow it to be reshaped by an external load. The thermal decomposition of bamboo's chemical components is particularly notable above 160 °C, and superior dimensional stability can be obtained by increasing the preset temperature to reduce the hygroscopicity by decreasing the number of free hydroxyl groups [35,38].

Previous studies [39,40] showed that water absorption and swelling are key factors for ensuring the dimensional stability and overall performance of wood/bamboo-based composites, including the mechanical properties, aging resistance, antiseptic properties, and antimycotic ability. The hydrolysis of polymers or a reduction in adhesion between the resin and fiber matrix results in weak interfacial interactions for carrying a load and resisting mildew and insect infestations when a composite absorbs moisture [41].

Although the hot-pressing temperature plays a crucial role in the production and utilization of bamboo scrimber, there have been few related studies. According to the above situation, this article explored the failure mechanism of bamboo scrimber with various hot-pressing temperatures from 140 °C to 170 °C by performing water resistance tests according to GB/T 40247-2021 and GB/T 17657-2013. The failure mode of the bamboo scrimber was imaged by an extended depth-of-field 3D microscope and a field emission scanning electron microscope (FE-SEM) to understand cell deformation and cracking. In addition, the vertical density profile of the bamboo scrimber was measured by an X-ray density profiler, and changes in the cellulose crystallinity degree and chemical compositions of bamboo scrimber with the temperature were investigated using X-ray diffraction (XRD), Fourier-transform infrared (FT-IR) spectroscopy, and X-ray photoelectron spectroscopy (XPS). The multi-scale

studies revealed the mechanism by which the hot-pressing temperature affected the water resistance of bamboo scrimber.

2. Materials

2.1. Raw Materials

Four-year-old Moso bamboo (*Phyllostachys pubescens* Mazel) was obtained from Anji, Zhejiang Province, China. The phenol-formaldehyde (PF) resin (trade name: PF16L510) was supplied by Guangzhou Dynea Chemical Industry Company. Its viscosity was (25 °C) 67.5 cps. The solids content (3 h/120 °C) was 53.5%, and the pH (25 °C) was 9.71.

2.2. Preparation of Bamboo Scrimber

The hot-pressing temperature was chosen as the optimized control factor, and bamboo scrimber was prepared at preset hot-pressing temperatures of 140 °C, 150 °C, 160 °C, and 170 °C. The bamboo scrimber with a density of 1.20 g/cm³ and the manufacturing process is shown in Figure 1.

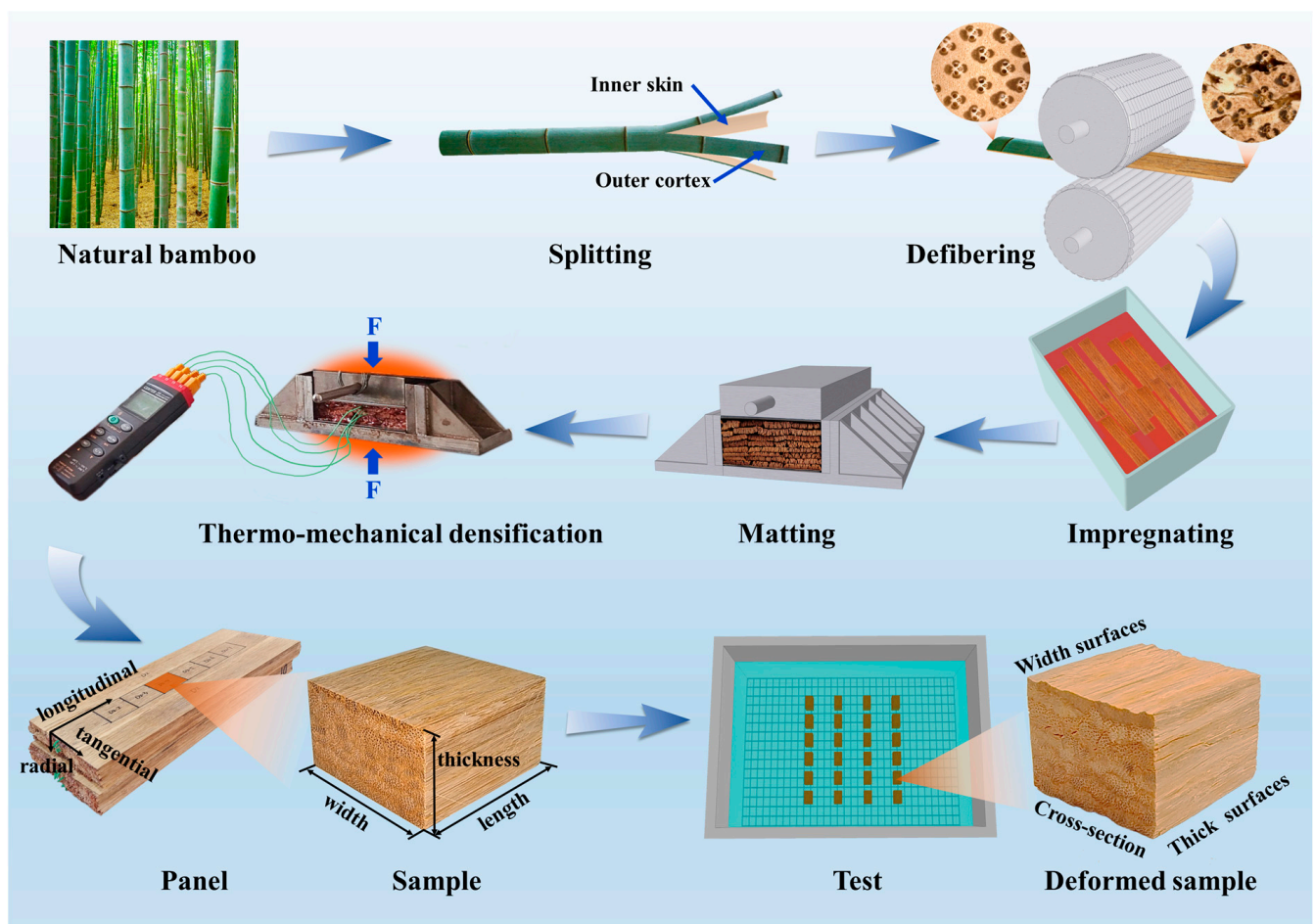


Figure 1. Schematic illustration of outdoor bamboo scrimber preparation and experiment.

The bamboo was sawn into bamboo tubes and split longitudinally into four quarter-circular bamboo tubes. The bamboo tubes were pushed into a fluffer to form a net-structured bundle with a width of 50–70 mm and thickness of 6–8 mm, respectively. The moisture content of the dried bundles was 7.6%. The net-structured bamboo bundles were sawn into small segments with a length of 280 mm and infused into the diluted PF resin solution for 5 min at ambient temperature to attain a resin content of 15%. The impregnated bamboo bundles were air-dried to a moisture content of 12%. A mat of bamboo

scrimber was hand-formed with 8 layers. Bamboo fibers were oriented along the length of the member in a compression mold. The orientation of the inner skin and outer cortex of bamboo bundles was controlled symmetrically along the central layer of the mat and horizontally parallel to the press platens, and the mat thickness was about 53 mm. The temperatures from the surface layer to the inner layer were measured by a thermocouple thermometer (CENTER-309, CENTER, Taiwan, China) to ensure they reached the preset temperatures. The bamboo scrimber was produced with dimensions of 280 mm (length) $\times$ 120 mm (width) $\times$ 19 mm (thickness). The boards were stored in a laboratory at 25–30 °C and at 60%–65% relative humidity for 14 days until a constant weight was attained. The final moisture content of specimens was about 5%.

3. Methods

3.1. Water Resistance

The lab-made boards with a stable moisture content were machined into test specimens, with six replicate samples in each group. Referring to GB/T 40247-2021 and GB/T 17657-2013, the air-dried density, thickness swelling rate (TS), width swelling rate (WS), and water absorption rate (WA) were tested. The dimensions of the specimens were 30 mm (length) $\times$ 30 mm (width) $\times$ 18 mm (thickness). The specimens were divided into three groups and tested using three sets of parameters: test A (fully impregnated in water at 20 °C for 24 h), test B (impregnated in water at 63 °C for 24 h), and test C (28 h cycle test; fully impregnated in water boiled at 100 °C for 4 h, 20 h oven-dried at 63 °C and then fully impregnated in water boiled at 100 °C for 4 h). The TS, WS, and WA were calculated based on the thickness, width, and quality, respectively.

3.2. Failure Mode

The cross-section and width surfaces of specimens of water resistance tests were sanded with abrasive paper of 3000 mesh and 1000 mesh, respectively. Surface topography and roughness were characterized by extended depth-of-field 3D microscopy (EDF 3D microscope, VHX-6000, Keyence, Japan). Then, the shapes of vascular bundles, ground tissue cells, and cracks that emerged during the various tests were observed. Samples with dimensions of 5 mm (*L*) $\times$ 5 mm (*T*) $\times$ 5 mm (*R*) were prepared using a sliding microtome to obtain smooth surfaces. The samples were coated with gold and observed with a field emission scanning electron microscope (FE-SEM, S-4800; Hitachi, Japan) operated at 10.00 kV.

3.3. Physical Property Analysis

The vertical density profiles of the bamboo scrimber specimens at various hot-pressing temperatures were determined by using an X-ray density profiler (DENSE-LAB X, Electronic Wood Systems GmbH, Hameln, Germany) with a step of 20 μ m. The samples had dimensions of 30 mm (*L*) $\times$ 30 mm (*T*) $\times$ 18 mm (*R*).

3.4. Chemical Component Analysis

The samples were prepared as slices with dimensions of 30 mm (*L*) $\times$ 10 mm (*T*) $\times$ 0.15 mm (*R*). The crystal structure and crystallinity of cellulose were determined by X-ray diffraction (XRD) analysis (D8 ADVANCE, Bruker, Germany). The samples were made into powder by a grinder and screened with 200 mesh. The surface chemical groups of samples were recorded by FTIR spectroscopy (Nicolet IS10, Thermo Scientific, Waltham, MA, USA). The spectra were measured in the range of 500–4000 cm^{-1} . The resolution of the spectrometer was 4 cm^{-1} , the SNR was 50,000:1, and 32 scans were used. The samples were cut into small chips with average dimensions of 4 mm (*L*) $\times$ 4 mm (*T*) $\times$ 0.15 mm (*R*) by using a sliding microtome and scissors. X-ray photoelectron spectroscopy (XPS; ESCALAB 250, Thermo Scientific, Waltham, MA, USA) was used to analyze surface chemistry changes using a monochromatic Al $K\alpha$ ($h\nu = 1486.6$ eV) X-ray source with a power of 225 W. Survey scans were collected over a binding energy range of 0–1100 eV.

4. Results and Discussion

4.1. Water Resistance

The air-dried densities of bamboo scrimber samples for water resistance tests are displayed in Table 1. Variations in WA, WS, and TS with hot-pressing temperatures from 140 °C to 170 °C were influenced by the test method. The most obvious influence was observed for test C (28 h cycle test). Changes in the WA were not prominent in tests A and B and fluctuated from 5.38% to 5.90% and from 13.42% to 14.56%, respectively. In contrast, the WA showed the opposite trend for test C and decreased by 49.32% as the hot-pressing temperature was raised from 140 °C to 170 °C (Figure 2a). The WS varied within a small range for tests A and B. In general, WS for test C showed a downward trend as the hot-pressing temperature increased and reached the minimum when the hot-pressing temperature was 170 °C. WS showed the same trend as that of the WA, and decreased by 65.52% from 140 °C to 170 °C for test C (Figure 2b). The TS was one of the important parameters for water resistance and was affected by the location of the measurement. Comparing Figure 2c with Figure 2d shows that the E-TS was greater than C-TS during the same test. There was a consistent trend between the TS of the center point and edge from Figure 2c,d. TS decreased as the temperatures increased from 140 °C to 170 °C for different tests, in agreement with a previous study [42]. C-TS decreased by 23.76% for test A, 20.45% for test B, and 61.94% for test C. E-TS decreased by 29.06% for test B, and 57.84% for test C. The analysis of variance (ANOVA) of the hot-pressing temperature on the water resistance of bamboo scrimber is shown in Table 2, which shows that the influence of hot-pressing temperature on the water resistance of bamboo scrimber had significant differences at $p < 0.001$ for test C. All of the performance indicators for the water resistance responded strongly to the hot-pressing temperature for test C (28 h cycle test), but displayed only a weak correlation for tests A and B. Moreover, the temperature of the test water had a significant effect on the water resistance compared with 20 °C and 63 °C, which was almost 2.3 to 8.4 times higher than that of other variables. Increasing the hot-pressing temperature improved the water resistance of bamboo scrimber, which is correlated with the concentrated stress release efficiency and adhesive failure degree [43]. Consequently, the 28 h cycle test is the most appropriate method for characterizing the water resistance of outdoor bamboo scrimber. Thermo-mechanical densification results in various mechanical and chemical changes in wood or bamboo-based composites [44].

Table 1. The average values and standard deviation of the density of samples. Values in parentheses are the standard deviation.

Test Method	Hot-Pressing Temperature/°C			
	140	150	160	170
A	1.18 (0.02)	1.20 (0.03)	1.19 (0.02)	1.17 (0.01)
B	1.18 (0.02)	1.20 (0.02)	1.19 (0.01)	1.18 (0.02)
C	1.18 (0.02)	1.20 (0.02)	1.19 (0.01)	1.18 (0.03)

4.2. Failure Mode Determined by EDF 3D Microscopy and FE-SEM

The images and data in this section were recorded from the specimens of test C. The EDF 3D microscopy images of the failure mode of bamboo scrimber after hot-pressing at temperatures from 140 °C to 170 °C for test A and test B are shown in Figures S1–S8.

4.2.1. Cross-Section

The deformation and cracking of bamboo scrimber samples during tests were related to the morphology, matting, and compression direction of the bamboo bundles. The details of the matting and orientation of cells are shown in Figure 3a, where the blue lines are the bonding interfaces of each layer. The radical deformation in the cross-section, parallel to the compression direction of bamboo scrimber, was the prominent part of three dimensions

(radial, tangential, and longitudinal). It mainly contributed to the resilience of cell walls to resist cracking failure at bonding interfaces.

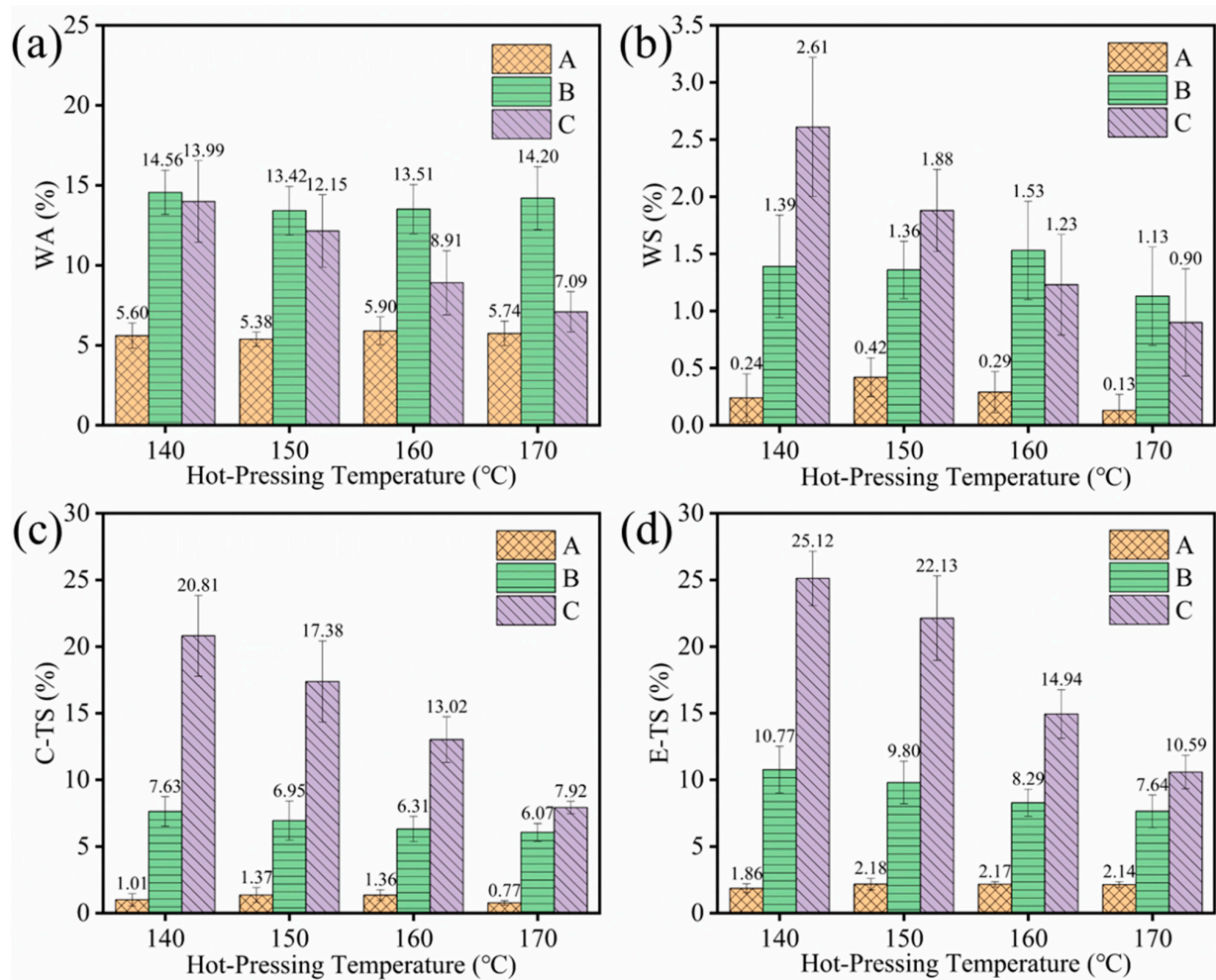


Figure 2. Water resistance of bamboo scrimber samples with different hot-pressing temperatures during three tests: (a) water absorption rate (WA), (b) width swelling rate (WS), (c) thickness swelling of the center point (C-TS), and (d) thickness swelling of the edge point (E-TS).

Table 2. Analysis of variance (ANOVA) of the hot-pressing temperature on the water resistance of bamboo scrimber.

Test Method	Dependent Variable	Independent Variable	Quadratic Sum	Degree of Freedom	Mean Square	F	p
A	WA	Hot-pressing temperature	0.889	3	0.296	0.553	0.652 n.s.
	WS		0.257	3	0.086	2.751	0.070 n.s.
	C-TS		1.517	3	0.506	2.906	0.060 n.s.
	E-TS		0.416	3	0.139	1.465	0.254 n.s.
B	WA	Hot-pressing temperature	5.365	3	1.788	0.681	0.574 n.s.
	WS		0.497	3	0.166	1.064	0.387 n.s.
	C-TS		8.854	3	2.951	2.477	0.091 n.s.
	E-TS		36.413	3	12.138	5.966	0.004 **
C	WA	Hot-pressing temperature	174.356	3	58.119	13.540	0.000 ***
	WS		10.285	3	3.428	11.613	0.000 ***
	C-TS		559.720	3	186.57	34.445	0.000 ***
	E-TS		790.851	3	263.617	53.294	0.000 ***

Notes: n.s.: Not significant, **: Significant difference at $p < 0.01$, ***: Significant difference at $p < 0.001$.

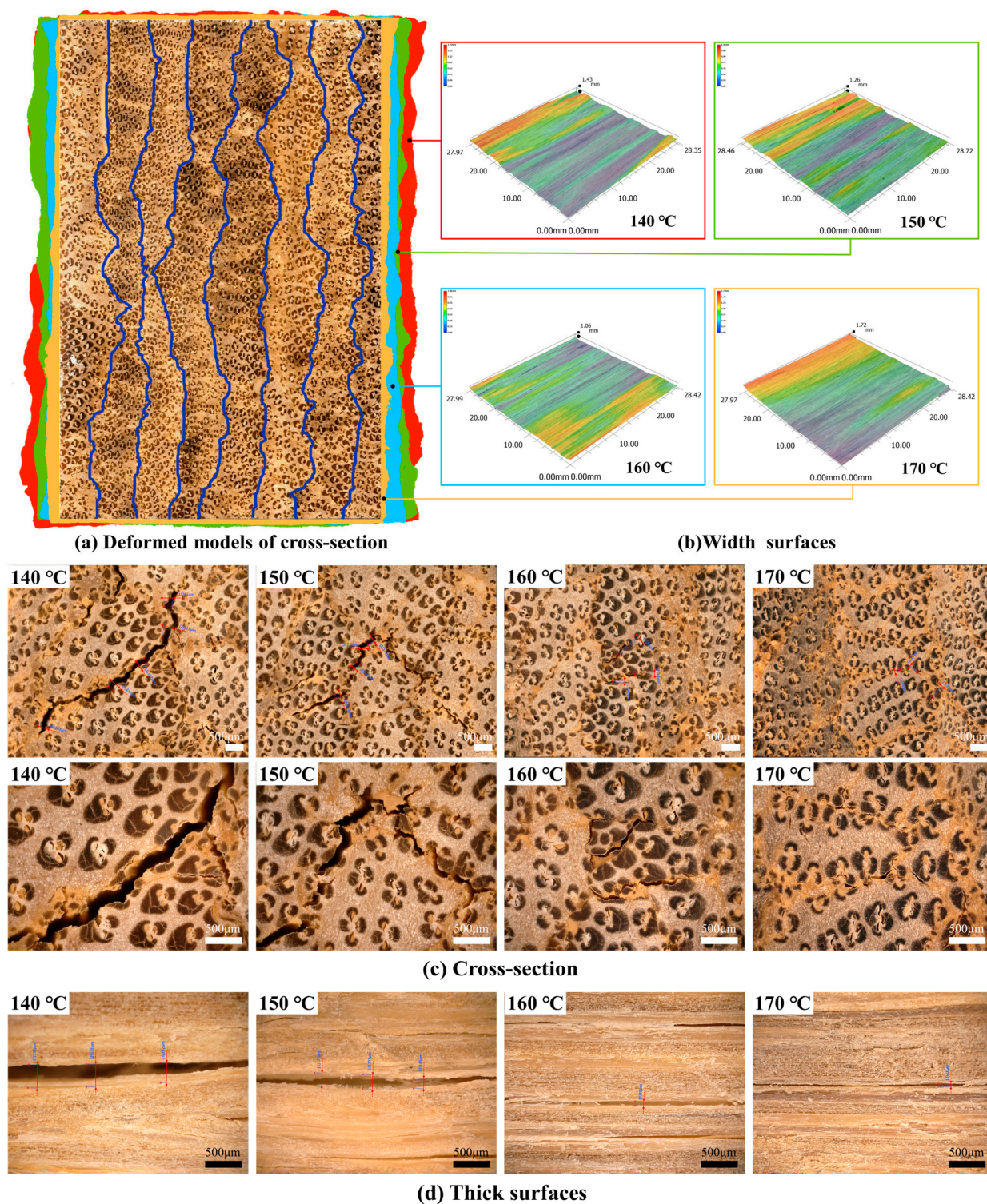


Figure 3. Surfaces of three dimensions: (a) deformed models of the cross-section, (b) width surfaces of bamboo scrimber with hot-pressing temperatures from 140 °C to 170 °C after 28 h cycle test, and typical cracks of (c) cross-sections and (d) thick surfaces of bamboo scrimber samples with various hot-pressing temperatures after a 28 h cycle test.

Changes in cell walls were observed at the cross-section of samples in Figure 4a,b, which showed cell walls of vascular bundles dramatically rebounded, especially at the metaxylem vessels, phloem, and the exterior region of the sclerenchyma sheath. The ground tissue cell walls swelled in Figure 5a,b. This phenomenon was explained by Tero

Mäkinen [45] using four-dimensional in situ tomography in which they showed that the thinner wood cell wall experienced much greater compression. Figure 4c,d show the cross-sections of natural bamboo and bamboo bundles after defibering and clarify the location and extension of cracks. Initially, the cell walls swelled in opposition to the thermo-mechanical densification direction. Compressive stress was released during the 28 h hydrothermal treatment cycle, and then imperceptible cracks appeared in vascular bundles due to the uniform stress release and failure of tiny bonding interfaces. Fine cracks, combined with a defective sclerenchyma sheath and radius deformation of the metaxylem vessels and phloem, emerged at vascular bundles in the early stages of the tests. They extended into radial cracks of bundles because defibering gradually formed macroscopic cracks during the tests [46]. Typical cracks with different lengths and widths at hot-pressing temperatures from 140 °C to 170 °C are shown in Figure 3c. The maximum width of cracks decreased successively from 263 mm, 133 mm, 60 mm, to 41 mm. The crack length was closely related to the thickness of bundles and the extension at weak bonding interfaces. Macroscopic cracks were mainly found in the bundles of each layer and were parallel to the thickness direction. Overall, the cracks visibly decreased as the hot-pressing temperature increased.

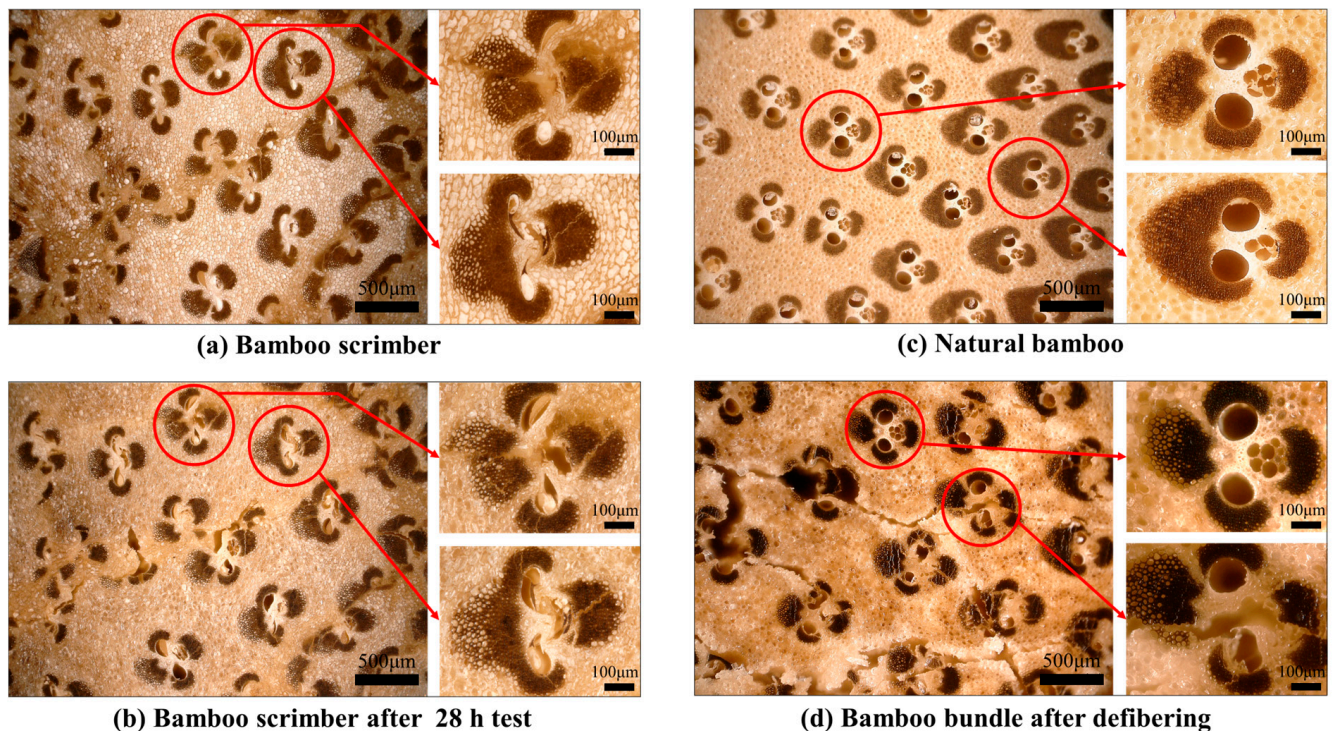


Figure 4. Deformation of cells in the cross-section of bamboo and bamboo scrimber at different stages: (a) bamboo scrimber, (b) bamboo scrimber after 28 h test, (c) natural bamboo, (d) bamboo bundle after defibering.

4.2.2. Width Surfaces

The width surfaces parallel to the width and longitudinal directions of samples are shown in Figure 1. The surface properties of bamboo scrimber were represented by the arithmetic mean of the profile (R_a) and the maximum height of the profile (R_z), as measured by an EDF 3D microscope. Table 3 demonstrates that the roughness of the samples was negatively correlated as the temperature was raised from 140 °C to 160 °C. There was an increase at 170 °C due to differences in swelling between the edge and central parts of samples. Representatively, the outline of cross-sections and changes in the width surfaces of samples at different hot-pressing temperatures for the 28 h cycle test are displayed by image models in Figure 3a,b. The swelling of the cross-section due to water absorption

corresponded to a decrease in the TS as the hot-pressing temperature increased. The deformation of the width surface morphology decreased at higher temperatures within the range of 140 °C to 170 °C, showing that increasing the hot-pressing temperature improved the water resistance of the width surface.

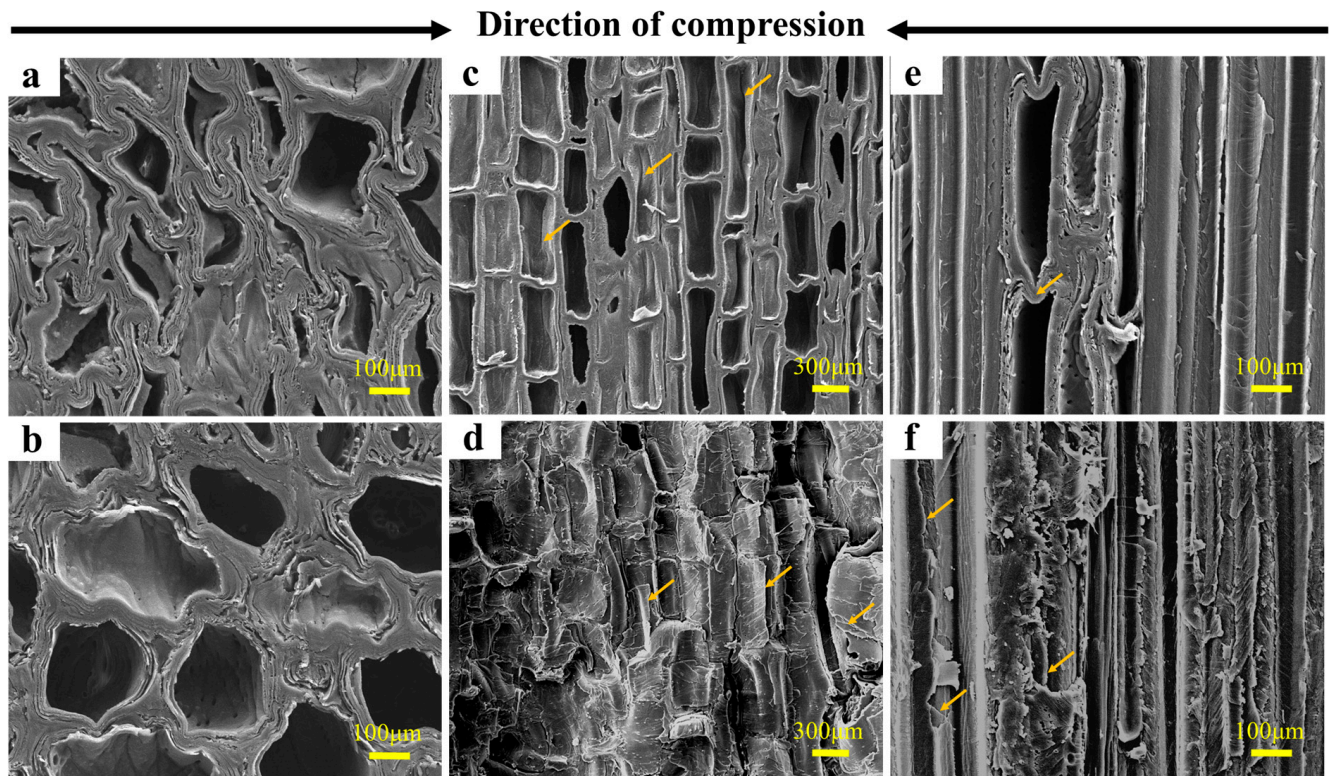


Figure 5. The deformation and cracks of cross-section and radial section of samples before and after the 28 h cycle test: (a,b) parenchyma cells in the cross-section, (c,d) parenchyma cells of ground tissue in the radial section, and (e,f) fibrocytes of vascular bundle in the radial section.

Table 3. The roughness parameters of samples treated in different situations with various hot-pressing temperatures.

Sample	Roughness Parameter	Hot-Pressing Temperature/°C			
		140	150	160	170
Untreated	R_a (mm)	0.06	0.06	0.06	0.06
	R_z (mm)	0.39	0.42	0.35	0.43
28 h cycle	R_a (mm)	0.26	0.20	0.13	0.29
	R_z (mm)	1.44	1.27	1.07	1.73

4.2.3. Thick Surfaces

Thick surfaces parallel to the radial and longitudinal directions of panels (Figure 1) split at the bonding interfaces between bundles of adjacent layers, as displayed in Figure 3d. The failure mode was usually macroscopic delamination. The maximum width of cracks decreased from 335 mm, 207 mm, 65 mm, to 45 mm as the hot-press temperature increased from 140 °C to 170 °C. Figure 5c,d display the different parenchyma cells between the unbroken ground tissue before the test and the position of cracking after the test. The differences between the sclerenchyma sheath in the intact vascular bundles before the test and the position of cracking after tests are displayed in Figure 5e,f. Significantly, Figure 5c,e show that the crinkles of cell walls caused by thermo-mechanical densification vanished. The microscopic failure mode of cracks in the radial section involved the stratification and destruction of the multilayer structure of fiber walls, as shown in Figure 5d,f.

4.3. Vertical Density Profile

Density profile analysis is a general method for measuring density distributions that directly determines the properties of wood or bamboo-based composites. Existing studies [47,48] revealed that the preset temperature greatly affects the vertical density profile (VDP) of wood-based composites, and the temperature controls the shape of the density profile. The recorded hot-pressing temperature of the inner layer of bamboo scrimber during thermo-mechanical densification is shown in Figure 6a. In contrast, Figure 6b,c illustrate that the VDP of bamboo scrimber showed a weaker relationship with the temperatures of thermo-mechanical densification and was more influenced by the mat formation of bamboo bundles. The density profile variations throughout the thickness coincided with the differences from the outer cortex to the inner skin of bamboo bundles shown in Figure 6c. There was a gradual decrease due to the distribution of vascular bundles, which was similar to the density difference between earlywood and latewood. The yellow lines in Figure 6c indicate the bonding interfaces of each layer. The density profile showed that the portion with a lower density occupied a larger percentage of ground tissue composed of parenchyma cells. This can promote water absorption because there are more microchannels and a higher specific surface area for water molecules. The portion with a higher density accounted for a larger percentage of vascular bundles, which are the weak points at which cracks may form because of crushed sclerenchyma sheaths and deformed metaxylem vessels of vascular bundles made during defibering and hot-pressing.

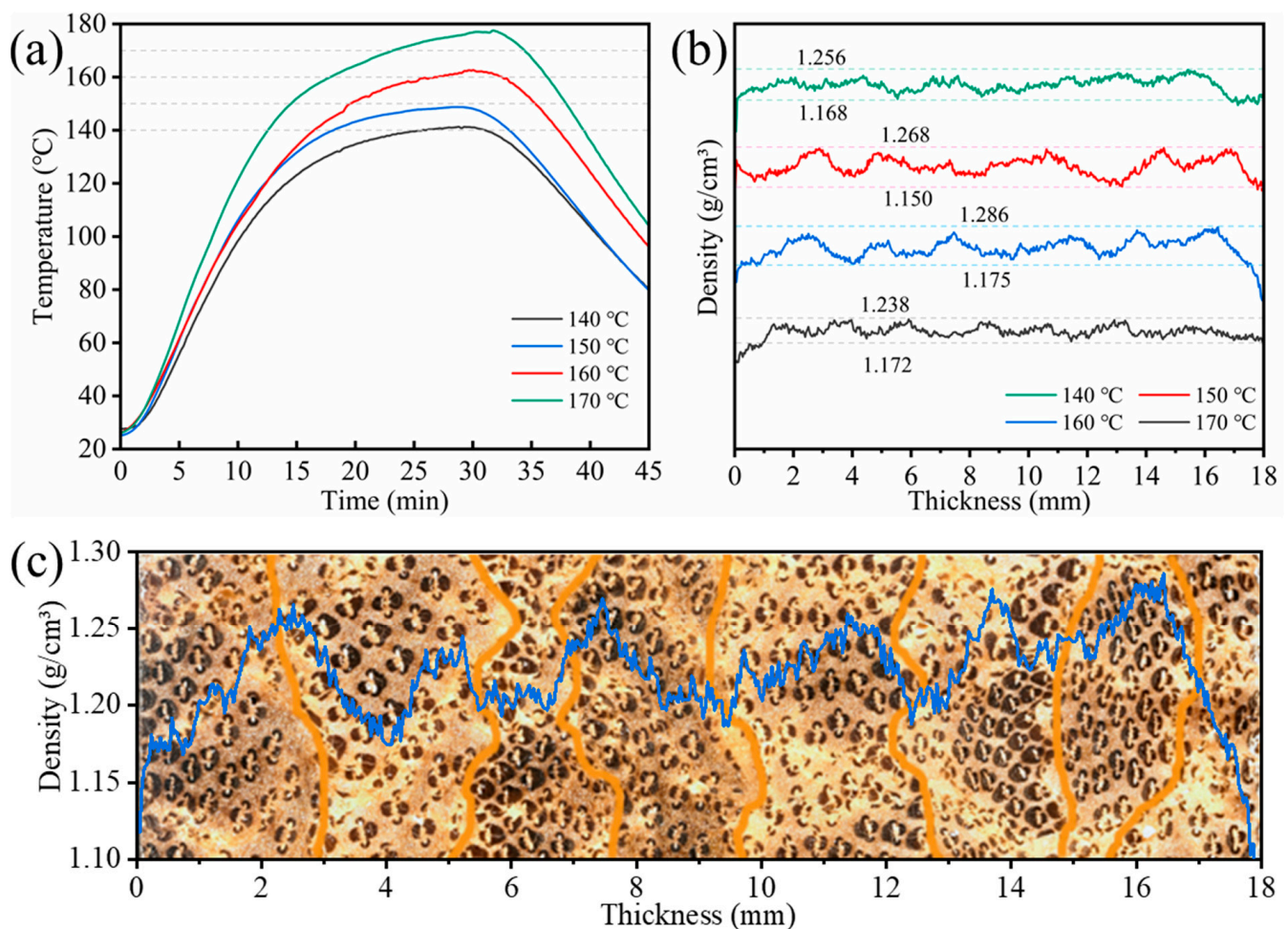


Figure 6. (a) Time–temperature curves of the inner layer during thermo-mechanical densification. (b) Density profiles of bamboo scrimber at different temperatures. (c) Schematic of the vertical density profile variations during mat formation of bamboo scrimber.

4.4. Analysis of the Crystalline Structure of Cellulose by XRD and FT-IR

The crystallinity of cellulose is an important factor in determining the physical and mechanical properties of bamboo scrimber. Here, they were measured by XRD and FT-IR. A higher crystallinity of cellulose can improve the water resistance of outdoor bamboo scrimber due to fewer hydroxyl groups in the amorphous region. The relative crystallinity index (*CrI*) of samples was calculated by using the formula $CrI (\%) = (I_{002} - I_{am})/I_{002}$ (Table 4), where I_{002} and I_{am} are the intensity of the crystalline portion at $2\theta = 22^\circ$ and the amorphous portion at $2\theta = 18^\circ$ in Figure 7a, respectively.

Table 4. The crystalline structure of cellulose in samples at hot-pressing temperatures of 140 °C, 150 °C, 160 °C, and 170 °C.

Test Method	Crystalline Structure	Hot-Pressing Temperature/°C			
		140	150	160	170
XRD	<i>CrI</i> (%)	62.72	62.95	63.63	63.73
FT-IR	H_{1428}/H_{897}	0.965	0.951	0.976	0.979
	H_{1376}/H_{2900}	1.002	0.995	1.000	1.002

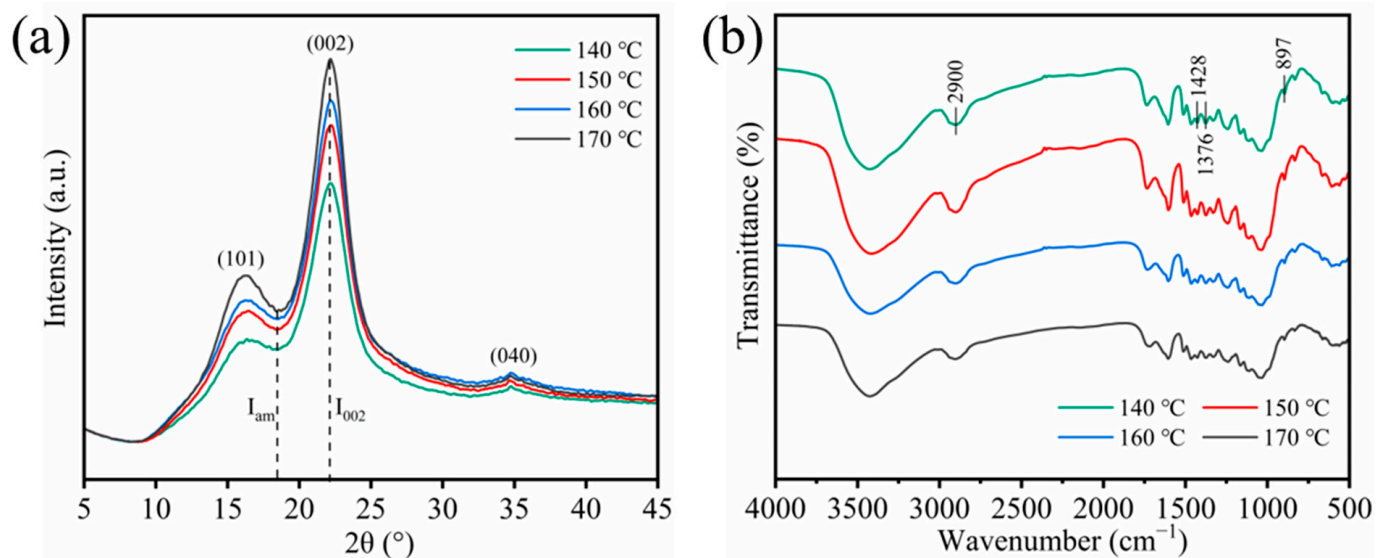


Figure 7. (a) The XRD patterns of bamboo scrimber slices. (b) FT-IR spectra of bamboo scrimber powder.

The FT-IR spectra were used to observe the surface chemical components of samples. Figure 7b shows the transmittance spectra of samples. There was no substantial difference in their peaks at different hot-pressing temperatures due to similar functional groups in all samples. In previous research [30,49–51], the absorption peak at 2900 cm^{-1} is primarily due to the C–H stretching vibration of the methyl and methylene groups of cellulose. The CH_2 scissor motion in cellulose appeared at 1428 cm^{-1} . The C–H bending vibration of cellulose was identified at 1376 cm^{-1} , and C–H deformation in cellulose appeared at 897 cm^{-1} . These characteristic peaks of cellulose were used to determine the crystalline percentage of cellulose. The IR crystallinity ratios H_{1428}/H_{897} [52] and H_{1376}/H_{2900} [53,54] are the infrared peak height ratios used to characterize the crystalline structure of cellulose (Table 4). These different peak heights were measured in the absorbance spectra of bamboo scrimber samples. The absorbance was calculated by using the Beer–Lambert law, $A = \lg(1/T)$. The results of the *CrI* measured and calculated in three ways showed the same variation trends and demonstrated that hot-pressing temperatures from 140 °C to 170 °C had a slight positive effect on the ordered degree of molecular chains in bamboo scrimber. The decomposition of amorphous regions in cellulose may have contributed to

this phenomenon, in agreement with a previous study [26] on the crystalline structure of cellulose during heat treatment.

4.5. Surface Chemical Component Analysis by XPS

Quantitative changes in the surface chemical components of samples at different hot-pressing temperatures were characterized using XPS. Carbon and oxygen in cellulose, hemicelluloses, lignin, and extractives of bamboo and PF resin were the most prominent elements in the XPS spectra of bamboo scrimber. The XPS spectra revealed that the peaks of C and O for bamboo scrimber appeared at 285–290 eV and 531–534 eV, similar to those of wood [55]. Detailed chemical bond analysis of C and O was made by Gaussian fitting of the C 1s and O 1s regions. The C 1s and O 1s spectra were deconvoluted into four and three components (C1–C4, O1–O3) to understand the functional groups reported in previous studies [30,49,55]. C1 (284.7 eV) was assigned to carbon atoms bonded only with saturated carbon or hydrogen atoms, C–C and C–H, mainly originating from lignin, fatty acids, and other extractives such as phenylpropane. C2 (286.5 eV) was assigned to carbon atoms bonded with one oxygen atom, C–O, which mainly originated from cellulose and hemicelluloses. C3 (288 eV) was assigned to carbon atoms bonded to a carbonyl or two non-carbonyl oxygen atoms, C=O and O–C–O, and mainly originated from hemicelluloses and lignin. C4 (289.1 eV) was assigned to carbon atoms bonded to one carbonyl and one non-carbonyl oxygen atom O–C=O and mainly originated from hemicelluloses and extractives. O1 (531.5 eV) was assigned to oxygen atoms in benzyl aryl ether and diaryl ether groups of lignin. O2 (533 eV) was assigned to oxygen atoms in aliphatic hydroxyl groups, carbonyl groups (including aldehydes and ketones), lactones, and aliphatic ethers (C–O–C) of cellulose, hemicelluloses, fatty acids, and other extractives. O3 (534.2 eV) was assigned to oxygen atoms in phenolic hydroxyl groups, aliphatic aromatic ether excluding methoxyl, methoxyl groups, and carboxylic acids in hemicelluloses and lignin.

Upon increasing the hot-pressing temperature (Figure 8), each O/C, C2, C3, and O2 component gradually decreased, while C1, C4, and O1 components increased. This illustrated that the thermal degradation of hemicelluloses and cellulose formed acetic acid and increased the relative content of lignin. These results were confirmed in previous research [26,28].

Temperature was the dominant factor during the curing of the PF resin [9]. The hydrophobic surfaces and interfacial interlocks comprising PF resin and cell walls during the thermal curing prevented the hydroxyl groups of bamboo scrimber from interacting with water molecules to improve the water resistance [56,57]. C and O atoms of the PF resin were located in the benzene ring, methylene, hydroxymethyl groups, methylene ether bonds, and phenolic hydroxyl groups. Chemical reactions (a) and (b) occurred during the thermal curing of the PF resin below 170 °C, in Figure 9, which improved the polymerization degree and formed methylene and methylene ether bonds. The methylene ether bonds readily decomposed into methylene and released formaldehyde above 160 °C, as shown by chemical reaction (c) in Figure 9. The reduction in the methylene ether bond, which was categorized as O2, and the increase in the methylene content, which was categorized as C1, during the reaction was consistent with variations in the relative amount of O2 and C1 as the hot-pressing temperature rose from 140 °C to 170 °C.

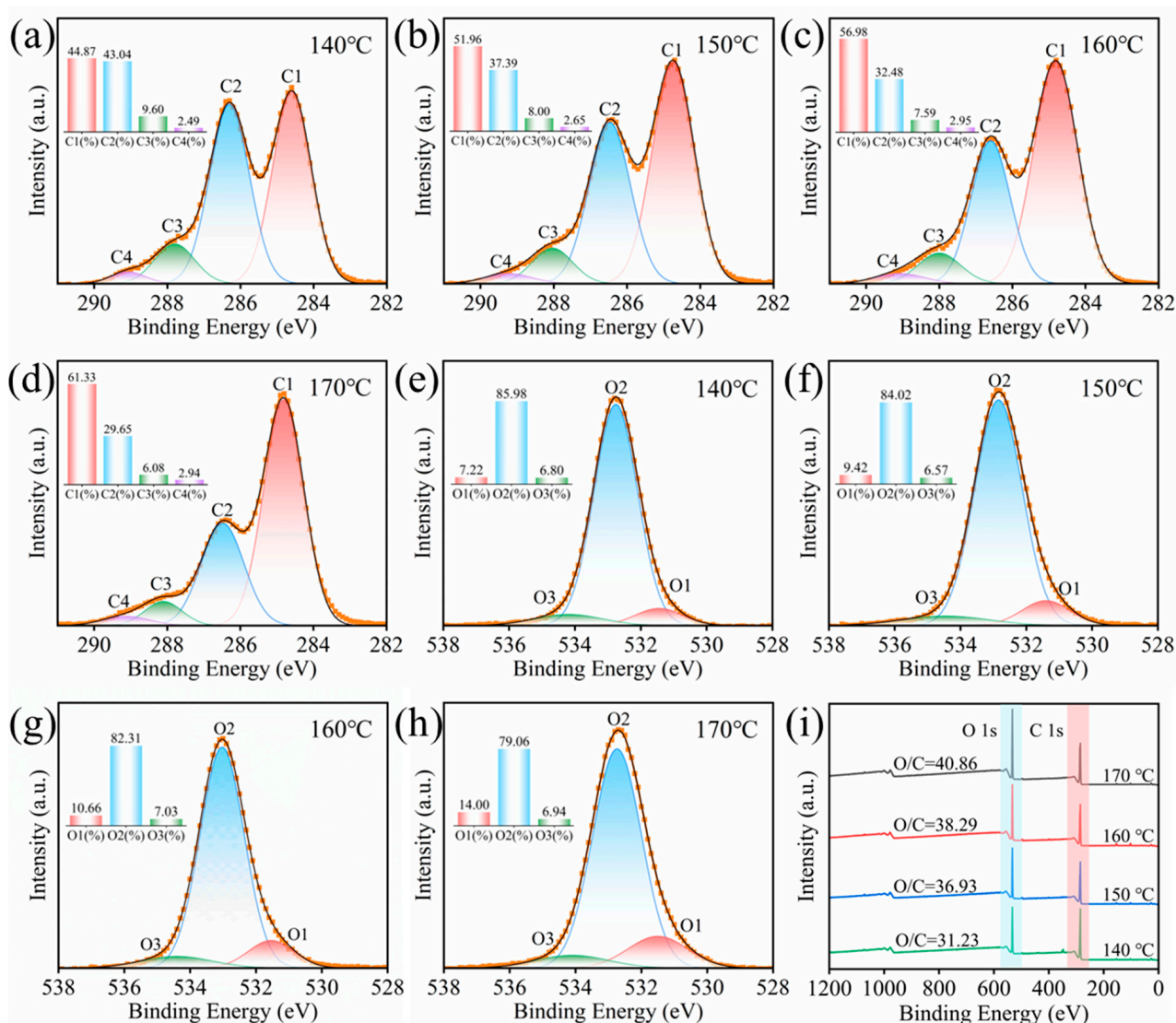


Figure 8. XPS spectra of bamboo scrimber at different hot-pressing temperatures: (a) C 1s-140 °C, (b) C 1s-150 °C, (c) C 1s-160 °C, (d) C 1s-170 °C, (e) O 1s-140 °C, (f) O 1s-150 °C, (g) O 1s-160 °C, (h) O 1s-170 °C; (i) XPS spectra of bamboo scrimber at hot-pressing temperatures of 140 °C, 150 °C, 160 °C, and 170 °C.

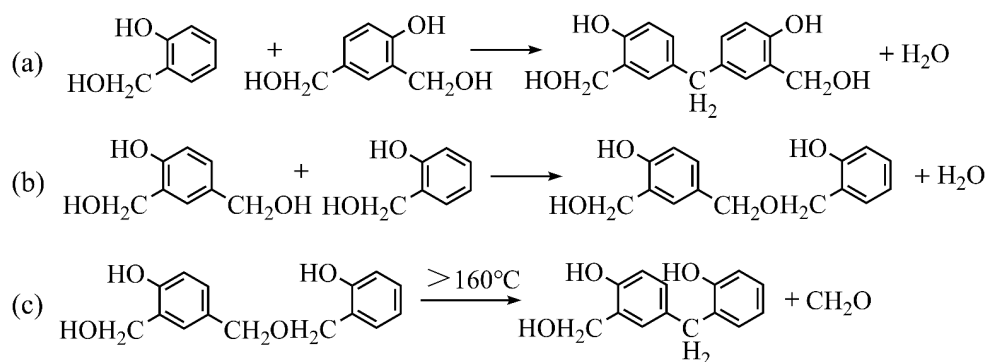


Figure 9. Chemical reactions occurring during thermal curing of the PF resin.

5. Conclusions

Multi-scale studies were conducted to optimize the water resistance of outdoor bamboo scrimber by improving the hot-pressing temperature during thermo-mechanical densification. The results showed that the hot-pressing temperature greatly influenced the water resistance from 140 °C to 170 °C. The TS and WS significantly decreased by 59.3% and 75.4% under the 28 h cycle test, respectively. There was a negative correlation between the hot-pressing temperature and TS and WS, but the relationship with WA was affected by the test method. The 28 h cycle test was appropriate for measuring the water resistance of outdoor bamboo scrimber. The deformation of cells and the position of cracks followed the same patterns at different hot-pressing temperatures. However, the deformation degree of cells and the size and quantity of cracks were negatively correlated with the temperature from 140 °C to 170 °C. These differences were reflected in the surface properties in three dimensions of bamboo scrimber, especially at the width surface, which will be exposed during applications. The destruction of bamboo scrimber involved the rebound deformation of cell walls and the failure of the bonding interface due to cracking. Cracks in bamboo bundles made during defibering and hot-pressing were the weak points of the bamboo scrimber, especially vascular bundles. Most small cracks appeared at crushed sclerenchyma sheaths and deformed metaxylem vessels of vascular bundles. Macroscopic cracks continuously grew at weak parts, such as small cracks or failed adhesive interfaces. The preset temperature had no obvious effect on the vertical density profile of the bamboo scrimber. Changes at the molecular level, including a moderate growth in cellulose crystallinity, an increase in the polymerization degree of PF resin, and the thermal degradation of hemicelluloses at higher temperatures, improved the water resistance and dimensional stability of outdoor bamboo scrimber. These results show that the water resistance can be improved by simply raising the hot-pressing temperature of bamboo scrimber, which can help extend its service life.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/f14040749/s1>, Figure S1: Images of bamboo scrimber with hot-pressing temperature at 140 °C for test A by EDF 3D microscope: (a) before the test, (b) after the test; Figure S2: Images of bamboo scrimber with hot-pressing temperature at 150 °C for test A by EDF 3D microscope: (a) before the test, (b) after the test; Figure S3: Images of bamboo scrimber with hot-pressing temperature at 160 °C for test A by EDF 3D microscope: (a) before the test, (b) after the test; Figure S4: Images of bamboo scrimber with hot-pressing temperature at 170 °C for test A by EDF 3D microscope: (a) before the test, (b) after the test; Figure S5: Images of bamboo scrimber with hot-pressing temperature at 140 °C for test B by EDF 3D microscope: (a) before the test, (b) after the test; Figure S6: Images of bamboo scrimber with hot-pressing temperature at 150 °C for test B by EDF 3D microscope: (a) before the test, (b) after the test; Figure S7: Images of bamboo scrimber with hot-pressing temperature at 160 °C for test B by EDF 3D microscope: (a) before the test, (b) after the test; Figure S8: Images of bamboo scrimber with hot-pressing temperature at 170 °C for test B by EDF 3D microscope: (a) before the test, (b) after the test.

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