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ORIGINAL ARTICLE

Mechanics and pyrolysis analyses of rotation welding with pretreated wood dowels

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Abstract This study examined the mechanics and pyrolysis analyses of rotation welding with treated dowels. Test results indicated that welding specimens with dowels immersed in $CuCl_2$ solution exhibited higher pullout resistance than untreated specimens. Scanning electron microscopy was used to observe and analyze the welding interface. Wood dowels immersed in $CuCl_2$ solution provided more flowing molten polymer to obtain better connection than untreated wood dowels. Based on the chemical analyses of X-ray photoelectron spectroscopy, and thermogravimetric analyses, the hydrolysis of cellulose and hemicellulose was detected after immersion of the dowels in a $CuCl_2$ solution. Pyrolysis of cellulose, hemicellulose and lignin occurred during the welding process. The hydrolysis of cellulose and hemicellulose may promote the pyrolysis and efficient connection of wood components during the welding process.

Keywords Rotation welding · Treated dowels · Hydrolysis · Pyrolysis

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Introduction

Welding technology is extensively applied in the fields of metals and plastics. This environmentally friendly way of connecting materials creates a new welding interface layer with the production of friction heating. During the generation of friction, thermoplastic materials are softened and fused and can be solidified once the friction is stopped. The main components of wood are cellulose, hemicellulose and lignin as a type of natural polymer material. In general, when wood is heated, the cellulose remains relatively stable, the hemicellulose generates thermal pyrolysis, and the lignin becomes softened [1, 2].

Four types welding methods are typically used: linear welding, orbital friction welding, circular welding, and rotation welding. These methods differ in velocity and force. In linear and orbital friction welding, the velocity and frictional force is a sinusoidal wave caused by linear oscillatory and elliptical movement, respectively, during welding. For circular and rotation welding, the velocity and frictional force are constant and caused by a circular and rotation movement [3]. Linear welding and rotation welding have been well studied. Linear welding occurs with high-speed linear motion between the surfaces of two wood blocks. In this method, the welding pressure, amplitude, frequency, holding pressure, and time are the main parameters affecting welding efficiency. In contrast, wood dowel

welding with lignin softening and degradation is caused by the increase in temperature resulting from the friction generated by the high-speed rotation between the wood dowel and the substrate hole [4–7].

The theoretical temperature may reach 456.65 K during wood dowel welding in 0.9–1.2 s due to friction [8]. Then, the temperature reduces to 413.15 K without additional interface materials. The well-distributed molten polymer generated by the increased temperature affects the water resistance. The wood dowel welding joints perform well with increased water resistance. No obvious delamination appeared after 8 months in a humid environment because of the special wood dowel welding connection [9].

The interface materials are composed primarily of the wood dowels [10] and the welding interface is influenced by the rotation and insertion speeds. A good wood-dowel welding interface requires a fine balance between rotation and insertion speeds and these may differ for different materials. While for European beech (*Fagus sylvatica*) the optimum rotation speed of the dowels was of 1200 to 1600 rpm using dowels of 10 mm in diameter [11], the ideal speed for Norway spruce substrate was 1000 rpm with beech dowels of 12 mm in diameter [12]. If the rotation speed is excessive, it will cause charring in the interface. At high rotation speed, a constant insertion speed may induce better welding properties than an accelerated speed [13]. A constant insertion speed of 25 mm/s gave the best results for sugar maple and 16.7 mm/s was the best for yellow birch [14], with sugar maple showing better overall welding properties.

According to several studies, welding properties could be significantly improved using smaller pre-drilled holes, dry wood dowels and increasing the contact area with non-vertical insertion methods. The smaller pre-drilled holes may increase the lateral pressure between the wood dowels and the pre-drilled holes. The wood dowels may provide more materials for the interface. Dry wood dowels after welding may absorb water from the environment. Wood dowels in a state of inflation may increase the bonding strength [11, 14–17]. It was found that native extractives of some special wood species could improve the water resistance [18–20]. Placencia and Pizzi compared welded joints with and without additives and found that welded joints with natural additives (rosin and lignin) showed improved water resistance [21, 22]. Water extracts of welded beech and welded spruce were analyzed after hydrolysis of welded materials and amounts of lignin, mono-oligosaccharides, acetic acid, vanillin, furfural, 5 hydroxymethylfurfural, and syringaldehyde were measured [23].

Chemical changes occurred in the wood components including the depolymerization and pyrolysis of cellulose, hemicellulose and lignin during welding. According to the research of Karl-Christian, acid-catalyzed cleavage of carbohydrates and the formation of formaldehyde, furfural, and other aldehydes occurred with the elevated temperature. In addition, lignin was cleaved and began auto-condensation [24]. With the changes in the wood components, the free phenolic hydroxyl groups increased and formed the hydrogen bonds with the cellulose to strengthen the stability of the interface [25, 26], and the hydroxyl benzene propane unit decreased [27]. Water vapor and carbon dioxide were the primary components of the volatile gas in the welding process [28–30]. The wood dowel welding joints showed improved pullout resistance [31, 32]. The pullout resistance property of the wood dowel welding joints is equivalent to that of wood dowel joints with adhesives, and better than that of nailed joints [33]. Wood dowel welding may also be used to enhance the mortise and tenon interference fit connection [34]. Novel full-scale multi-layered timber beams with welded-through wood dowels obtained considerable bending strength [35].

Citric acid was used as waterproofing additive in butt joints linear wood welding [36]. In the preliminary study of wood dowel welding, wood dowels pretreated with acid solution were considered. Several preliminary tests with dowels immersed in different acid solution including $CuCl_2$, $FeCl_3$, HCl solution, and chemical copper plating were carried out. The effect of immersion time of 1, 3, 5, and 7 days was tested. According to the test results, dowels immersed in $CuCl_2$ solution for 7 days showed the best pullout resistance. $CuCl_2$ solution is slightly acidic because of the hydrolysis of Cu^{2+}. The softened surface of the wood dowels caused by the acid corrosion promoted the formation of molten materials. This study aimed to analyze the influence of wood dowels immersed in $CuCl_2$ solution for 7 days on welding performance. The chemical changes were detected using X-ray Photoelectron Spectroscopy (XPS), and Thermogravimetric (TG) analyses. These results revealed significant changes of functional groups and wood components [33, 37, 38].

Materials and methods

Materials

Wood dowels were made of birch (*Betula* spp.), and were 12 mm in diameter and 100 mm in length. Dowels were pre-conditioned at 336.15 K to obtain a 2% moisture content. The temperature of 336.15 K was determined from preliminary experiments because the wood dowels could achieve the desired moisture content (MC) over 2 days almost without warping and cracking. Wood dowels with a 2% moisture content were divided into two categories including a control group without pretreatment (group A) and a pretreatment group (group B). Thirty wood dowels of

Group B were immersed in 0.1 mol/L $CuCl_2$ solution with the volume of 500 ml for 7 days at 293.15 K and 60% relative humidity (RH). During this immersing process, wood dowels and solution were stirred every 12 h. After 7 days wood dowels were dried to 2% moisture content for wood dowel welding.

Larch (*Larix gmelinii*) slats that were 40 mm (Tangential, T) × 60 mm (Radial, R) × 1000 mm (Longitudinal, L) were used as the substrate. Slats were pre-conditioned at 293.15 K and 60% RH, until equilibrium moisture content was achieved.

Manufacturer of specimen

Wood substrates were pre-drilled with holes 8.5 mm in diameter using a drilling machine (Proxxon TBH Typ 28 124). Next, the wood dowels were welded into the pre-drilled holes in the substrates 30 mm to create bonded joints with a high-speed rotation at 1000 rpm. The insert part of the dowel was transferred to a conical shape (Fig. 1) because of the different abrasion level during the welding progress. The rotation of the wood dowel stopped when the fusion and bonding was achieved in approximately 2–4 s [4]. After welding, wood slats were cut into 10 parts evenly in the length direction so that every welded dowel was 40 mm (T) × 60 mm (R) × 100 mm (L) in size. The specimens were conditioned at 293.15 K and 60% RH for 7 days before the tests were conducted.

Pullout test

The pullout resistance of the specimens was tested using universal testing equipment (WDW-300E) that pulled the welding wood dowels out of the substrate at a speed of 2 mm/min [11]. The specimens were fixed by clamping the dowel into the jaw of the fixed beam and the substrate block was fixed to the mobile beam with a metal framework.

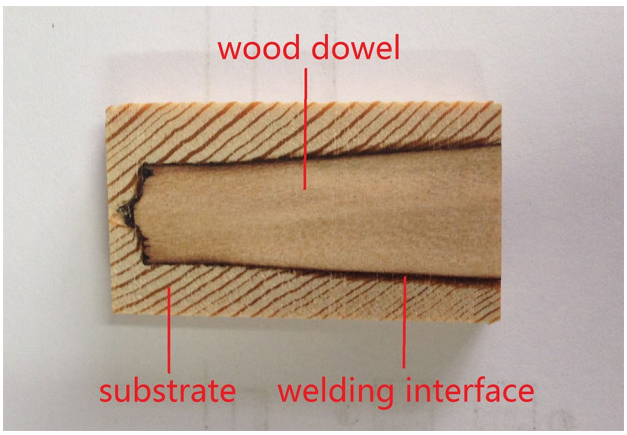


Fig. 1 The conical shape of the welding interface

SEM

The samples of welding interface were conditioned to oven dry. The size of the samples was 2 mm (T) × 2 mm (R) × 10 mm (L). The welding interface showed inhomogeneous distribution for all the specimens of group A. So the samples of group A were selected from the insufficiency and agglomerate regions. While for group B, the welding interface showed homogeneous distribution. So the samples of group B were selected from the welding interface randomly. Scanning electron microscopy (SEM) micrographs of the surfaces were obtained after metallizing with gold–palladium by a Hitachi S-3400N II microscope [14].

XPS

XPS was used to analyze the surface elements of the untreated wood dowel, wood dowel immersed in $CuCl_2$, welding interface, and welding interface using the $CuCl_2$ immersed dowel. Three samples which exhibited average pullout resistance with the mechanical test for each group were selected from the relative 30 specimens. The sample of wood dowel and welding interface were from the same specimen and the $CuCl_2$ immersed wood dowel and welding interface similarly were from the same specimen. The surface samples of the untreated and treated wood dowels were cut transversally from the non-welded part of the wood dowels and the surface samples of the welding interfaces were cut from the welded part of the wood dowels. XPS analyses obtained with a spectrometer (ESCALAB 250Xi Thermo Fisher) were used to provide quantitative data to verify the elementary composition of the sample surfaces. The angle of the emitted photoelectrons was 30° to the surface normal, the source type was Al K Alpha, and the analyser mode was a pass energy 100.0 eV with an energy step size of 1.000 eV and a total number of energy steps of 1361. The binding energy scale was corrected with reference to the 284.6 eV C–C bond and a XPSpeck41 was applied for the curve-fitting of the C1s spectra [37].

TG

Four samples were prepared for the TG test including the untreated wood dowel, wood dowel after immersion in $CuCl_2$, the welding interface, and the welding interface produced with the dowel treated with immersion in $CuCl_2$. Each sample was prepared by scraping and mixing the powders from the surface of the 30 tested specimens. Especially for welding interface, the powders were collected by scraping the black welding materials. All the powders of

each sample were stirred and blended uniformly. And then the powders were dried to 0% moisture content in 373.15 K before TG test [25, 28]. The TG analyses were performed using 10 mg powders for each test. The programmed heating pyrolysis of wood dowel and welding interface was carried out in a NETZSCH STA 449F3 simultaneous thermal analyzer. The samples in the crucible of TG were heated from 323 to 973 K at a heating rate of 10 K min^{-1}. The purified nitrogen was used as carrier gas to provide an inert atmosphere [39, 40].

Results and discussion

30 replicate welding specimens were tested for each group. The pullout resistance results are summarized in Table 1. The mean values of the welding samples using untreated wood dowels, group A, and the samples with treated wood dowels, group B, were 4027 and 4988 N, respectively.

Surface morphology of the welding interface

From the pullout resistance test, no matter group A and B, dowels of all specimens were pulled out along the welding interface without wood fracture. All the 30 replicate welding specimens of group A showed the same surface morphology. The end 3–5 mm region of the wood dowels appeared blank regions (Fig. 2a) primarily because of the decreased diameter of the end region caused by the excessive friction generated during initial insertion. For the2 30 replicate specimens of group B, 25 specimens showed performance as illustrated in Fig. 2b, with homogenous distribution of the molten polymer on the interface. The other 5 specimens appeared more like the untreated samples as shown in Fig. 2a.

The properties of the surface of the welding interface were observed and analyzed by SEM. The welding interface materials of group A showed inferior fluidity, leading to incompletely covered interweaved fibers (Fig. 2c) resulting in insufficiency (Fig. 2d) and agglomerate regions (Fig. 2e). Figure 2f showed the SEM results of the materials at the interface between the substrate hole and the wood dowel of group B. The interface materials were well distributed. This phenomenon indicated that the molten polymer flowed along the welding interface with good fluidity during the welding process.

As seen from Fig. 2g, the interweaved fibers formed a skeleton structure in the welding interface. Then, the molten polymer, such as the lignin, covered the interweaved fibers to form an integrated connection between the wood dowel and substrate with considerable strength [4].

Table 1 Results of the pullout resistance of 30 mm depth predrilled holes in a single substrate

Test group	A	B
Mean value (*N*)	4027	4988
Minimum value (*N*)	2351	3855
Maximum value (*N*)	5307	6301
Standard deviation (*N*)	641	748

Group A meant the untreated group; group B meant the treated group

XPS analyses

The XPS analyses of wood dowel and welding interface was performed because the materials of the welding interface were mostly generated from the wood dowel [10]. During the XPS test, Cu and Cl were not identified, because few of them existed in the samples. So in the XPS test, the element of C, O, and N were detected. But according to the pyrolysis–gas chromatography-mass spectrometry (PY-GC-MS) test, little amount of Cl existed in the treated wood dowel and welding interface. The content of elements and the oxygen/carbon (O/C) ratio for the wood dowels and the welding interface were shown in Table 2. The quantitative analyses were performed by peak fitting for the C1s categories. Deconvolution for the four types of carbon bonds was performed (Table 2) for the peaks from the wood dowel and welding interface and were determined as C1 class (C–C/C–H bonds, 284.6 eV), C2 class (C–O bonds, 286.5 eV), C3 class (C=O/O–C–O bonds, 287.9 eV) and C4 class (O=C–O bonds, 289.2 eV) [25].

Factor A with two levels was defined as untreatment and treatment. Factor B with two levels was defined as wood dowel and welding interface. To analyze the statistical significance of the two factors, the analysis of variance was carried out. No significant two factor interactions existed between the two factors. So in the analysis of variance, no interaction statistical significance test was done. For the results of the elements of C, and O, the ratio of O/C, C3, and C4, the factor B showed the high significant difference. For the results of C1, and C2, both the factor A and B showed the high significant difference. All the test results meet the requirements of homogeneity of variance. So the mean values were used to be analyzed below.

Treated wood dowel vs. untreated wood dowel

A 7.17% increase in the O/C ratio was identified for the treated wood dowels versus the untreated wood dowels (Table 2) because the cellulose and hemicellulose were hydrolyzed in the acid solution due to the $CuCl_2$ and the rupture of the glycosidic bond. During hydrolysis, the long chain of cellulose was broken into short ones due to chain scission and the hydrolysis of the compounds for

hemicellulose resulted in the formation of hexose and pentose [41]. The same phenomenon was found in the PY-GC-MS test, deacetylate occurred during the immersing process. According to these reactions, the portion of C1 and C3 decreased 5.66 and 18.26%, respectively, and the portion of C2 increased 21.52%.

Wood dowel vs. welding interface for both treated and untreated samples

Variation of O/C indicated that the main reactions occurred during the wood welding process between the welding interface and the wood dowels. From Table 2, a 35.29 and 28.94% increase in the O/C ratio were identified and calculated for the welding interface versus the wood dowels of group A and group B, respectively. During the rotation welding, high pressure assured the wood dowel would weld into the substrate hole quickly and successfully. Molten polymer that was rich in oxygen-containing groups formed from the amorphous cellulose, hemicellulose, and lignin that were depolymerized with the rapidly increasing temperature [42, 43]. Because of the high speed of rotation, these polymers were mixed well together. Once the welding process stopped, the molten polymer solidified. The new generated chemical materials with oxygen-containing groups caused the O/C increase [28].

The variation of C1 to C4 was primarily due to the depolymerization of the wood components, hydrolysis under acidic conditions and pyrolysis during the welding process. In general, the proportion of the C1 and C4 categories decreased and the proportion of the C2 and C3 increased in the welding interface versus the wood dowel.

The chemical bound water was dehydrated with a small quantity of the long cellulose broken chain for the temperature range from 420 to 510 K [34], which resulted in the increase of C3 [41]. Meanwhile, the pyrolysis of cellulose was observed by the Fourier Transform Infrared Spectroscopy (FTIR) analyses, which caused the decrease of C1. The presence of the peak at 1420 cm^{-1} represented the CH_2 bending band of cellulose. For the treated welding interface, the intensity of the 1420 cm^{-1} peak decreased by the pyrolysis of cellulose fragments. For the pyrolysis of hemicellulose, furfural and other compounds were generated during the process, which resulted in the increase of C2, C3, and the decrease of C1. The formation of free formaldehyde from side chain scission and oxidation in the welding interface may result in the decrease in C1 [37]. Free phenolic hydroxyl groups were formed from ruptured ether bonds that caused the increase in C2, as observed in the 1230 cm^{-1} band by FTIR. The increase in C3 indicated that oxidation products were formed during the welding process and the heat treatment process.

Treated welding interface vs. untreated welding interface

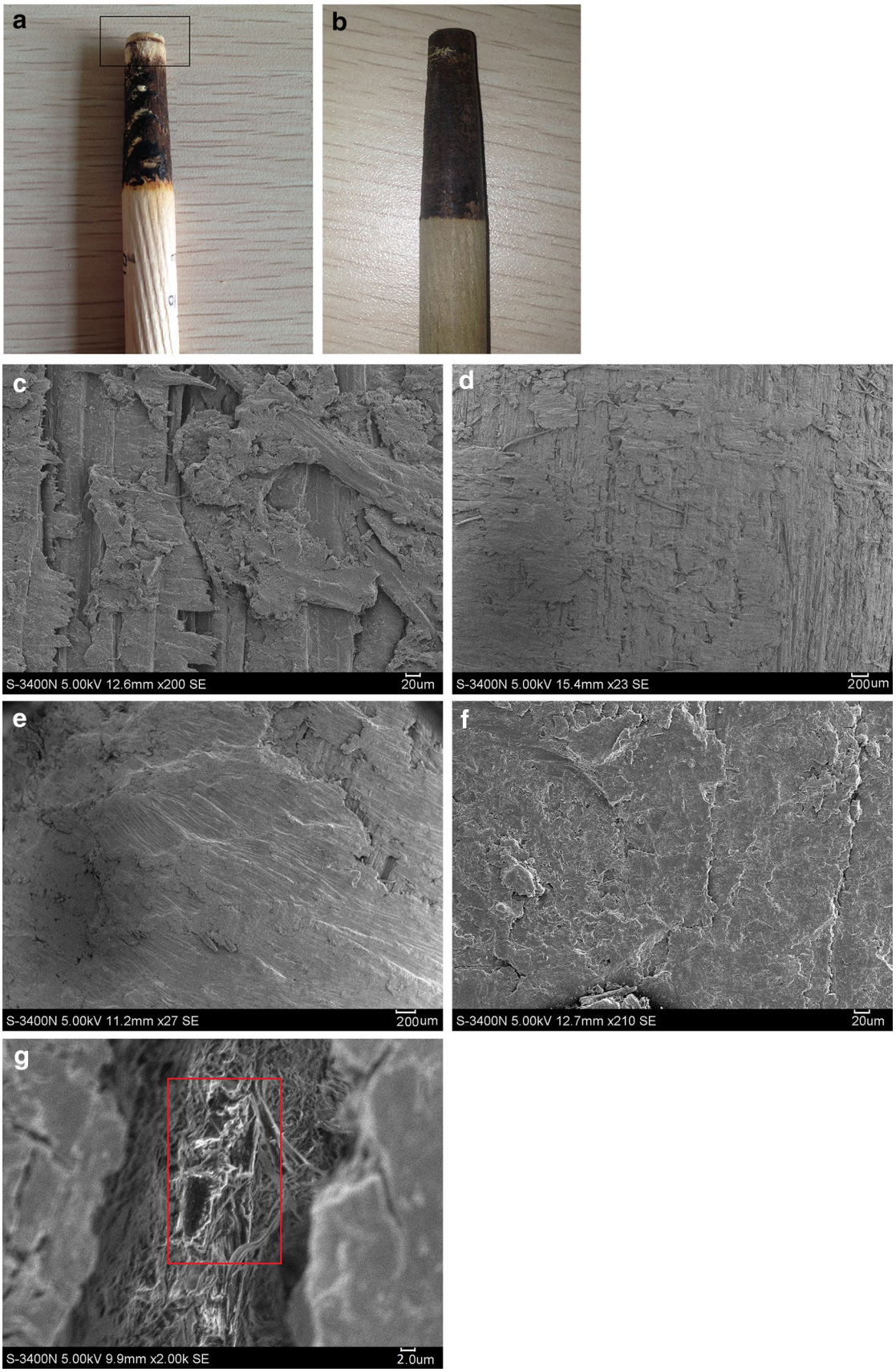
There were differences in the C1–C3 data for the untreated (group A) and treated (group B) wood dowels and welding interfaces. For group A, the portion of C1 decreased 12.91% and the portion of C2 and C3 increased 24.07 and 50.75%, respectively. For group B, the portion of C1 decreased 20.82% and the portion of C2 and C3 increased 30.34 and 77.29%, respectively. From the data analyses, difference existed in the rotation welding process. Similar reactions occurred in the welding interface during the rotation welding process, but the portions of C1-C3 of wood dowel and wood dowel immersed in $CuCl_2$ were different. So the untreated and treated welding interfaces differed in the degree and rate of pyrolysis reactions. More wood components in the treated welding interface were pyrolyzed to form furfural and other compounds during welding, causing a bigger increase of C2 and decrease of C1 and C3.

TG/Derivative thermogravimetric analyses

TG analyses

From the TG curves in Fig. 3a, the pyrolysis course of the welding interface was similar to that of wood dowel except for the final weight loss, which was lower for the welding interface. This phenomenon could be caused by the pyrolysis of cellulose and hemicellulose during the welding process, resulting in a higher relative content of lignin in welding interface than in the wood dowel. Based on this, the welding interface produced more coke than wood dowel during the TG testing process.

At the same time, the influence of immersion was studied. The TG curves were different in the temperature range of 500–650 K for the untreated and treated wood dowels. The weight loss rate of the treated wood dowel was higher than the untreated wood dowel. The hydrolysis of cellulose and hemicellulose in acid solution promoted the breaking of long chains and the formation of polysaccharide. The products from these changes may be more easily pyrolyzed. The final weight loss of the treated welding interface was slightly higher than the untreated welding interface. The pyrolysis and molten of lignin may be the principal factor, especially the pyrolysis of the aromatic ring [44]. This phenomenon was verified by FTIR analyses, peaks at 1508 and 1595 cm^{-1} represent the aromatic ring skeleton, which showed different changes for the untreated and treated welding interfaces. For untreated welding interface, the intensity of the two peaks increased due to the thermal condensation of the lignin, with the peak of 1460 cm^{-1} corresponding to the formation of CH_2 bridges between lignin fragments [37]. For treated welding interface, the pyrolysis of aromatic ring skeleton was identified


a
b
c
S-3400N 5.00kV 12.6mm x200 SE
20um
d
S-3400N 5.00kV 15.4mm x23 SE
200um
e
S-3400N 5.00kV 11.2mm x27 SE
200um
f
S-3400N 5.00kV 12.7mm x210 SE
20um
g
S-3400N 5.00kV 9.9mm x2.00k SE
2.0um

◂**Fig. 2** Surface morphology of the welding interface: **a** blank regions in the end 3–5 mm region, **b** homogenous distribution of the molten polymer, **c** incompletely covered interweaved fibers, **d** insufficiency regions, **e** agglomerate regions, **f** the interface materials well distributed, **g** a skeleton structure in the welding interface

after the welding process with the decrease of the peaks at 1508 and 1595 cm^{-1}. According to these analyses, it could be inferred that the fluidity of lignin from the treated welding interface was better than the untreated welding interface. Because of the better fluidity, the pullout resistance of the treated welding interface was higher than that of the untreated welding interface.

Derivative thermogravimetric analyses

The pyrolysis of cellulose and hemicellulose during welding process was revealed by the Derivative thermogravimetric analyses (DTG) curves (Fig. 3b). The pyrolysis rate of the welding interface was obviously lower than that of wood dowel in the temperature range of 500–600 K. This may be due to the pyrolysis of hemicellulose during the welding process. Additionally, the pyrolysis rate of the welding interface was lower than that of the wood dowel around 635 K likely due to the pyrolysis of cellulose during the welding process.

As a consideration of hydrolysis, the DTG curves of untreated and treated samples showed significant differences. The curves for the treated and untreated wood dowels differed in the temperature range of 500–650 K. Similar to the TG analyses, the hydrolysis of cellulose and hemicellulose likely caused these differences. The curves of welding interfaces showed differences in the temperature of fastest pyrolysis rate: 635 K for the untreated and 590 K for the treated one. The hydrolysis of cellulose and hemicellulose occurred during immersing. Long chains of cellulose were broken into short chains, and hemicellulose was decomposed into polysaccharide. The products from these two reactions were thermal decomposed more easily than the materials of welding interface [45]. The same phenomenon was found in the PY-GC-MS test, low molecular compounds and glucose of treated samples were generated more quickly than that of untreated samples. Thus, the temperature of fastest pyrolysis rate was different for the two kinds of welding interfaces.

Conclusions

Rotation welding enabled jointing between dowels and substrate with considerable strength. The dowels that were treated by immersion in a $CuCl_2$ solution showed improved welding properties compared with the untreated dowels. The end region of the wood dowels from the non-treatment group was not welded, but for the pretreatment group, the molten polymer on the interface was homogeneously distributed. XPS analyses showed that the hydrolysis of cellulose and hemicellulose occurred during the $CuCl_2$ immersion process, resulting in an increase in C2 and a decrease in C1. Heat degradation of wood components occurred during the welding process, resulting in an increase in C2 and C3 and a decrease in C1. The hydrolysis of cellulose and hemicellulose could promote the pyrolysis and molten of wood components. From TG/DTG analyses, the pyrolysis

Table 2 Surface composition of the wood dowel and welding interface determined by XPS

Test group	Samples	Elements (%)			Ratio (%)
		C	O	N	O/C
Group A	Wood dowel	76.52 (1.24)	22.92 (1.30)	0.56 (0.07)	29.98 (2.19)
	Welding interface	70.79 (1.58)	28.69 (1.37)	0.52 (0.22)	40.56 (2.83)
Group B ($CuCl_2$)	Wood dowel	75.31 (2.75)	24.11 (2.82)	0.58 (0.17)	32.13 (4.95)
	Welding interface	70.43 (3.07)	29.05 (3.11)	0.52 (0.04)	41.43 (6.21)
Test group	Samples	Carbon C1s components (%)			
		C1	C2	C3	C4
Group A	Wood dowel	65.23 (2.97)	25.14 (2.80)	6.68 (1.87)	2.95 (1.69)
	Welding interface	56.81 (5.02)	31.19 (6.19)	10.07 (2.26)	1.93 (1.81)
Group B ($CuCl_2$)	Wood dowel	61.54 (2.12)	30.55 (3.88)	5.46 (2.50)	2.45 (1.54)
	Welding interface	48.73 (3.14)	39.82 (4.36)	9.68 (2.20)	1.77 (1.02)

1, wood dowel and welding interface of group A meant untreated samples; 2, wood dowel and welding interface of group B ($CuCl_2$) meant treated samples; 3, elements (%) of C, O, and N meant the amount of these elements in the samples, respectively; 4, C1: C–C/C–H bonds, 284.6 eV; C2: C–O bonds, 286.5 eV; C3: C=O/O–C–O bonds, 287.9 eV; C4: O=C–O bonds, 289.2 eV; 5, the ratio of O/C meant the ratio of the amount of O and C; 6, test results were the mean value of three samples; 7, values in parentheses were standard deviation

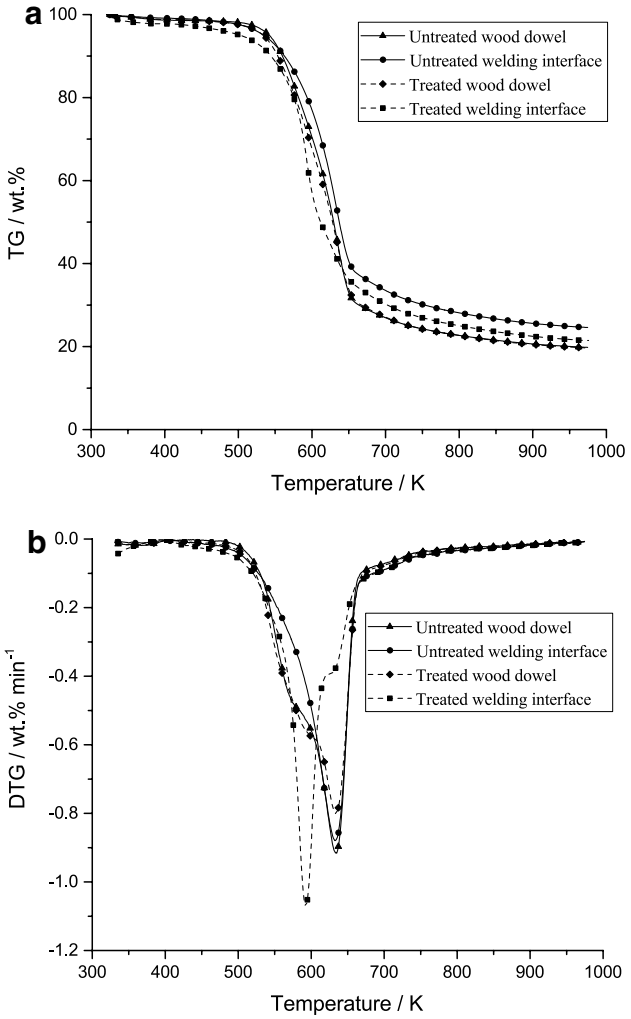


Fig. 3 The TG and DTG graphs of the treated and untreated wood dowels and welding interfaces: **a** TG, **b** DTG

degree of hemicellulose was higher than that of cellulose and lignin. The hydrolysis of cellulose and hemicellulose occurred during immersion and hydrolysis reduced the pyrolysis temperature of cellulose and hemicellulose.

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Author contributions Ying Gao and Songlin Yi designed the experiment, Xudong Zhu and Chun Ni performed the experiment and analyzed data; Jirong Zhang and Xiangya Luo completed pullout resistance test; Xudong Zhu prepared and edited the manuscript, and Ying Gao reviewed and directed the manuscript.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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