Effect of Cold Plasma Treatment on FRP Surface: The Behavior of FRP/Wood Interfacial Adhesion

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Abstract—In this work, FRP panel surface was treated by 12 plasma techniques formed by a pairwise combination of four power consumptions (300W, 400W, 500W, and 600W) and three transmission velocity (2.5m/min, 5.0m/min, and 7.5m/min) respectively, and then adhere to timbers. According to delamination test dates, 7 of 12 specimen groups meet the requirement. Shear strength at dry state of composite – FRP/wood –was improved. Mode G_{IIC} of 4ENF for the composite fracture toughness test was inferred in this paper. Variance analysis results demonstrated that power and velocity interaction has significant influence on delamination properties of FRP-wood interface, and power has significant influence on shear strength and fracture toughness. Considering strength and toughness synthetically, the optimized process of plasma treatment on FRP surface could be concluded, which power consumption was 300W, transmission speed was 5.0m/min. With this process, the properties of the composite interface are as following: the average shear strength at dry state was 9.46MPa, 6.49MPa at wet state, the G_{IIC} was 0.94kJ/m^2, and the average delamination ratio was 1% and 3% after soaked in normal and boiling water, which are all meet the request of standard.

Keywords-G-FRP; plasma treatment; glulam; interfacial adhesion; delamination properties; shear strength; fracture toughness

I. INTRODUCTION

Fiber Reinforced Plastic (FRP) can meet the demands of modern engineering structures with characteristic of large span, heavy load, light, enduring, etc. due to its outstanding properties such as high strength to weight ratio, corrosion resistance and anti-fatigue, etc. [1].

In FRP reinforced Wood composite: the FRP can effectively improve glulam mechanical strength especially flexural properties [2]. Dagher found the flexural strength with FRP/glulam composite could be increased by 56% at most when low and medium wood were used and beams reinforced with FRP on the tension side in the experimental study [3]. Bending stiffness and strength of composites were increased by 18% and 26%, respectively, when FRP reinforcement ratio is 3% [4]. Besides, pre-stress FRP sheets as external reinforcement of wood members can obtain more than 24% increasing of bending properties compare to not [5]. However, a number of studies have shown that delamination in the end position of composites is ubiquitous when FRP sheets pre-stressed [6–10].

It is important to note an effective interface cohesion is required in an excellent composite to obtain better stress transfer and crack-resistant [11]. According to FRP/Wood composite structure, five possible failure modes of FRP/wood interface are described as follows: failure of wood, interface between wood and adhesive, adhesive, interface between adhesive and FRP and FRP. In fact, it happens with more than one failure modes as usually. But since untreated FRP surface exhibit very low wettability, stress concentrates at interface between FRP and adhesive of FRP/wood composite which under loading [12]. Oporto studied to increase wettability of FRP surface with pre-treatment of cold plasma, and the results shown that shear strength of interface between FRP and WPC (Wood plastic composite) was improved to 200% [13]. What is more, Glass-FRP surface – treated by cold plasma – contact angle decreased from 98C to 19C, which means a higher surface energy obtained, and the same result shown through test analysis of AFM and XPS [14].

Interface fracture is a typical failure mode of composite system, and the main reason for development of fracture is delamination – the sliding crack [15]. Four – point bend end – notched flexure specimen (4ENF) were used appropriately for fracture toughness test of composite due to their three aspects of advantages: designed easily; crack propagation stability under loading; just measured critical load since the critical energy release rate of 4ENF is not affected by the influence of crack length [16]. The aim of our investigation is to evaluate bonding properties of FRP/wood – pretreated with cold plasma on FRP surface – composite through various test analysis include delamination properties, shear strength and fracture toughness synthetically, and obtain the optimal process finally.

II. EXPERIMENTS

A. Materials and Equipment

FRP: Glass fiber reinforced plastic with size of 1060 mm × 51 mm × 20 mm.

Wood: Camphor pine with density of 0.5 g/cm^3, moisture rate of 0.8% and size of 1060 mm × 51 mm × 20 mm provided by Dalian Shuanghua Factory (China).

Adhesive: One component polyurethane – PURBOND HB S709 – provided by Dalian Shuanghua Factory (China).

Number		P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
Cold Plasma treatment	Power/w	300	300	300	400	400	400	500	500	500	600	600	600
	Speed (m/min)	2.5	5.0	7.5	2.5	5.0	7.5	2.5	5.0	7.5	2.5	5.0	7.5

Figure 1. Technological parameters of cold plasma treatment on FRP surface.

Number		P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
Specimens soaked in water with temperature of 10C to 20C													
Delamination ratio (%)	Average	12	1	1	0	30	0	3	11	0	7	4	3
	max value	87	14	21	0	100	0	24	59	9	52	33	35
Specimens soaked in boil water													
Delamination ratio (%)	Average	1	3	4	3	20	1	12	10	1	5	4	2
	max value	26	33	42	39	78	7	94	62	14	55	44	18

Figure 2. Results of delamination test.

Plasma machine and universal mechanical testing machine provided by Beijing Forestry University (China).

B. Cold plasma Treatment

Firstly, the FRP surface was treated by cold plasma with variable quantities of power and speed of plasma machine respectively, and the technological parameters are shown in Figure 1.

Secondly, in proper order of wood after FRP after wood composites were glued with adhesive quantity of 180g/m². Then pressed the composites and made sure the temperature was 20°C, the relative humidity was 65% and time for press lasted 175min at least. Finally, composites were laid for 10 hours at temperature of 20°C after released.

III. RESULTS AND DISCUSSION

A. Delamination Properties

According to GB/T 26899 (China national standard for structural glulam), each composite were cut into 12 specimens with size of 75mm×50mm and each specimen's quality obtained. Half specimens soaked in water (10–20°C) for 24 h and the rest of specimens soaked in water (10–20°C) for 1h followed by 4h in boil water, and then all of them transferred into constant temperature

(70°C) drying box. The specimens were taken out until their qualities as 1.0–1.1 times as initial. At last, delamination ratio obtained through measurement and calculation as Figures 2 and 3 show.

The standard of delamination ratio is equal or lower than 5%, and 7 of 12 groups meet the requirement. The data of delamination appears regularly, so variance analysis demanded:
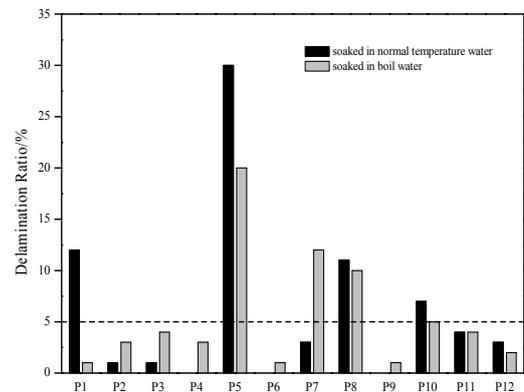

Figure 3. Delamination ratio.

Test method	Soaked in normal water			Soaked in boil water		
Source of variation	Power/W	Speed/M/Min	Interaction	Power/W	Speed/M/Min	Interaction
F ratio	2.50	14.50	13.39	2.74	7.29	4.16
F standard value	2.76	3.15	2.25	2.76	3.15	2.25
Significant	★	★★	★★		★	★

Note: α=0.05.

Figure 4. Variance analysis of delamination test date.

According to Figure 4, factors of speed and interaction influenced the delamination ratio significantly especially on specimens soaked in normal water as the table shows. Besides, wood density, angle of laminated texture and resin infiltration of wood are important reasons for delamination according to observe the failure specimens.

B. Shear Strength

According to GB/T 26899 (China national standard for structural glulam), each composite was cut into 12 specimens with exposed interface between wood and FRP as appropriate size as standard demand and each specimen's quality obtained. Half specimens soaked in water (10–20°C) for 24 h and then shear strength tested immediately at wet state according to GB 5005, and the others' shear strength tested at dry state. At last, shear strength date, wood failure ratio and retention of shear strength at wet state obtained through measurement and calculation as Figures 5 and 6 show.

Number	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
Specimens tested at dry state												
Shear strength/MPa	9.43	9.46	7.10	7.75	8.16	7.19	8.82	7.75	7.62	10.45	8.79	8.68
Wood failure ratio/%	75	71	93	84	62	86	93	80	94	83	59	90
Specimens tested at wet state												
Shear strength/MPa	5.58	6.49	5.66	4.96	4.55	6.16	5.13	4.57	5.02	5.68	5.83	6.27
Wood failure ratio/%	37	31	74	75	35	67	48	33	55	31	41	69
Strength retention/%	59.17	68.59	79.77	63.96	55.76	85.68	58.08	59.02	65.89	54.37	66.26	72.24

Figure 5. Results of shear strength test.
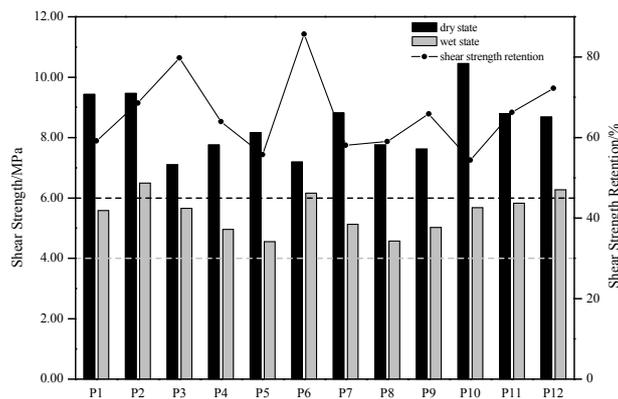
Figure 6. Shear strength dates analysis.

Test method	Tested by dry state			Tested by wet state		
Source of variation	Power/W	Speed/M/Min	Interaction	Power/W	Speed/M/Min	Interaction
F ratio	6.44	9.39	1.86	6.21	1.96	2.34
F standard value	2.76	3.15	2.25	2.76	3.15	2.25
Significant	★	★		★		★

Note: α=0.05

Figure 7. Variance analysis of shear strength test date.

The standard of shear strength is equal or more than 6MPa and 4MPa for dry state and wet state test, respectively. All groups meet the requirement. The maximum value of dry state shear strength appeared when plasma machine's power is 600W and speed is 2.5 m/min at group P10. Dates of shear strength retention and dry state

shear strength move in opposite directions and correlation coefficient $R=-0.60$. Remarkable variation expansion coefficient between wood and FRP could be the main reason – the stronger cohesion of interface, the more stress gathered during soaking for that. According to Figure 7, variance analysis shows that factor of power influenced both shear strength by dry and wet state test. And factors of speed and interaction influenced dry and wet state test, respectively.

C. Fracture Toughness

1) Reduction for 4ENF Test Model

Criterion for energy of crack propagation originated by Griffith's theory, then it developed by Iwrin–Kies in 1945 [17]:

$$G_c = \frac{P_c^2}{2b} \frac{\partial C}{\partial a} \tag{1}$$

In which, P_c is critical load, b is width, C is flexibility, a is crack length and G_c is critical value of shear modulus.

And according to Timoshenko's theory:

$$\triangle_L = \frac{PS_L^2}{D}\left[\frac{-RS_L^2}{12L} + \frac{RS_L}{3} - \frac{2L}{3} + (\frac{2-R}{2})a \times (1 - \frac{a}{2L} + \frac{a^2}{12L^2})\right] + \frac{PS_L}{D}\left[(1-\frac{2L-S_L}{2L})(\frac{RS_L^2}{12}) - \frac{2(2L-S_L)}{3} + \frac{(2L-S_L)^2}{2} - \frac{(2L-S_L)^3}{12} + (1-\frac{2L-S_L}{2L})(2-R)(\frac{a^2}{4} - \frac{a^3}{12L})\right]$$

$$\triangle_R = \frac{PS_L}{D}\left\{\frac{(2L-S_L)^2}{2} - \frac{(2L-S_L)^3}{12} + \frac{RS_L^2}{12} - \frac{2(2L-S_L)L}{3} - \frac{RS_R S_L^2}{24L} + \frac{(2-R)a^2}{4}\left[1 - \frac{2L-S_L}{2L} - \frac{a}{3L} + \frac{(2L-S_L)a}{6L^2}\right]\right\} + \frac{P}{D}\left\{(1-\frac{2L-S_L}{2L})\left[\frac{(2-R)a^3}{6} - \frac{2(2L-S_L)^2 L}{3}\right]\right\}$$

In which, D is bending stiffness and R is ratio of bending stiffness with uncrack to crack part.

Because $C = \frac{\delta}{P} = -\frac{\triangle_L + \triangle_R}{2(P_L + P_R)}$ and $P_L = P_R = P/2$, in

which P is total load, P_L is left load, P_R is right load, $\triangle_L$ is beam deformation at left and $\triangle_R$ is beam deformation at right.

So, $D = EI = E\frac{b(2h)^3}{12} = \frac{2Ewh^3}{3}$, and since

$R = \frac{EI}{EI_d} = E\frac{b(2h)^3}{12} \Big/ E\frac{wh^3}{12} = 8$ for homogeneous

material, in which, E is modulus of specimen in length direction.

According to Eq. (1),

$$G = \frac{9P^2}{16Eb^2 h^3}\left[S_L + a(1 - \frac{S_L}{2L} - \frac{S_R}{2L})\right]^2 \quad , \quad \text{and}$$

$S_L + S_R = 2L$, finally

$$G_{II}^{4ENF} = \frac{9P^2 S_L^2}{16E_1 b^2 h^3} \tag{2}$$

2) Specimen of 4ENF Size Design and Test

The composite was made by proper order of wood after FRP after wood, and the interface of composites for testing should located at the middle of the height direction, so the bending stiffness should be equal between the both side of that interface. According to this basic theory, the size of 4ENF was designed as follows: height of wood above/below FRP is 20mm/18.58 and of FRP is 1.4mm, width and length is 40mm and 960mm, span at outside/inside is 800mm/400mm, and prefabricated length of crake is 400 mm [18, 19]. Speed of loading during test is 2mm/min. Based on mathematical expression of (2) and critical load from test, fracture toughness result calculated as shown in Figure 8:

Variance analysis of fracture toughness is shown in Figure 9.

Number	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
Fracture toughness(KJ/m2)	0.95	0.94	1.28	0.83	0.85	0.73	0.55	0.60	0.55	0.51	0.53	0.56

Figure 8. Fracture toughness.

Test	Fracture toughness		
Source of variation	Power/W	Speed/M/Min	Interaction
F ratio	7.15	0.23	0.51
F standard value	3.01	3.40	2.51
Significant	★		

Note: α=0.05.

Figure 9. Variance analysis of fracture toughness.

Obviously, factor of power influenced the fracture toughness significantly. Besides, the groups of P10, P11 and P12 which are pretreated by 600W power plasma showed lower fracture toughness.The maximum value of fracture toughness appeared when plasma machine's power is 300W and speed is 7.5 m/min at group P3.

IV. CONCLUSIONS

Based on mode GIIC of 4ENF for the composite fracture toughness test, delamination properties test and shear strength test, the results showed that power and velocity interaction has significant influence on delamination properties of FRP-wood interface, and power has significant influence on shear strength and fracture toughness. Considering strength and toughness synthetically, the optimized process of plasma treatment on FRP surface could be concluded, which power consumption was 300W, transmission speed was 5.0 m/min. With this process, the properties of the composite interface are as following: the average shear strength at dry state was 9.46 MPa, 6.49 MPa at wet state, the GIIC was 0.94 kJ/m^2, and the average delamination ratio was 1% and 3% after soaked in normal and boiling water, which are all meet the request of standard.

V. ACKNOWLEDGMENT

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