

## Influence of plasma treatment on properties of ramie fiber and the reinforced composites

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### ABSTRACT

In this research, 9 series of ramie fibers were treated under low-temperature plasma with diverse output powers and treatment times. By analysis of the surface energy and adhesion power with epoxy resin, 3 groups as well as control group were chosen as reinforced fibers of composites. The influences of these parameters on the ramie fiber and its composites such as topography and mechanical properties were tested by scanning electron microscopy (SEM), atomic force microscopy (AFM), tensile property and fragmentation test of single-fiber composites. Contact angle and surface free energy results indicated that with the increased treatment times and output powers, surface energy and adhesion work with epoxy resin improved. Compared with the untreated fibers, surface energy and adhesion work with epoxy resin grew 124.5 and 59.1% after 3 min-200 w treatment. SEM and AFM showed low temperature plasma treatment etched the surface of ramie fiber to enhance the coherence between fiber and resin, consequently fiber was not easy to pull-out. After 3 min-200 w treatment, tensile strength of ramie fiber was 253.8 MPa, it had about 30.5% more than that of untreated fiber reinforced composite. Interface shear stress was complicated which was affected by properties of fiber, resin and interface. Fragmentation test showed biggest interface shear stress achieved 17.2 MPa, which represented a 54.0% increase over untreated fiber reinforced composites.

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## Introduction

Natural fiber reinforced composites, with good mechanical property and benefits for environment protection and resource saving, has been inevitably a trend to draw much attentions [1–3]. In the field of natural fiber reinforced composites, ramie fiber reinforced composite promises a great future [4,5]. Ramie fiber is a kind of bast fiber with long length, high crystallinity and large elastic modulus. Comparing with glass fiber and aramid fiber, the density of ramie fiber is smaller. Therefore, ramie is deemed to be one of the preferred reinforcing fibers of the composite. However, based on previous research, the outer pectin impurity covering of ramie fibers could greatly impact on the adhesion with epoxy resin and the transfer shear stress [6,7], resulting in degraded the performance of composites. In order

to achieve a good performance of ramie fiber reinforced composites, the modification of ramie fiber has become the focus in research.

A great many approaches could be used for surface treatment with different concerns and effects [8,9]. Alkali treatment is one of the most common methods for surface treatment, and could remove impurities of ramie fibers and increase fibrillation, improving the surface and mechanical properties of ramie fibers [10,11]. However, alkali treatment may cause damage to fiber properties, and reaction time, alkali solution concentration, and treatment temperature should be strictly controlled. Besides, chemicals used for alkali treatment may be blamed for environment protection. In order to reduce the impact of fibers properties caused by surface treatment, low-temperature plasma treatment was adopted for ramie fiber surface treatment.

The plasma can be generated by radio frequency discharge of gas, which involves tons of energy particles [12,13]. These energy particles can cause a series of physical and chemical reactions on the surface of material so that the surface structure of the material and its properties would change greatly [14]. Comparing with alkali treatment, plasma treatment has advantages such as less damage to materials, easy control, and obvious effect [15,16]. What is more, plasma treatment is pollution free, which belongs to the techniques of dry process without contamination of the environment.

Borooj et al. authenticated that the concentration of reactive functional groups on the fiber surface was increased after the plasma treatment, as well the surface roughness, which improved the interfacial adhesion between carbon fibers and epoxy resin [12]. Li et al. found that ramie fabrics were plasma treated with ethanol pretreatment to enhance mechanical properties, and proper selection of treatment voltage was critical for plasma treatment [15]. Nevertheless, up to present, only relatively few papers have been reported concerning influence of plasma treatment on properties of ramie fiber and the reinforced composites under different process parameters. Therefore, in this research, ramie fibers were treated by low-temperature plasma with different process parameters, based on contact angle measurement, scanning electron microscopy (SEM), atomic force microscopy (AFM), and mechanical property tests, and the properties of ramie fiber and the reinforced composites were also evaluated.

## Experimental

### Materials

Ramie fibers were purchased from Hunan Shangke Co. Ltd., China. The average diameter of a single fiber is 37.4  $\mu\text{m}$  with a fineness of 10.8 dtex. E51 epoxy resin was provided by Changshu Jiafa Co. Ltd., China. Before the experiment, E51 epoxy resin was mixed with 2101CN- iminazole at a ratio of 10:1(wt/wt).

### Fiber preparation and low-temperature plasma treatment

Before plasma treatment, ramie fibers were soaked in acetone for 15 min to remove residues thoroughly, and dried in a vacuum oven at 70 °C for 20 min. The fibers were then divided equally into 10 groups, and a group without any treatment was tested as control. The plasma treatment was carried out by using microwave low-temperature plasma device (PR3, Beijing Chuangshi Co. Ltd., China). The plasma was generated by

capacitance coupling glow discharge at 40 Pa. Ramie fibers with length of 25 cm were put in the glass container and placed in the device. 9 groups of ramie fibers were treated with different output powers (100, 150, and 200 w) for different periods (1, 2, and 3 min) with frequency 13.56 MHz, respectively. After the application, samples were placed into hermetic bags.

**Wettability measurement**

According to Young’s Equations (A1)–(A4) [17], ramie fiber surface energy and adhesion power with epoxy resin can be counted directly by contact angles [18–20]. The sessile drop technique was applied on a surface tensiometer (Kruss100, Germany) to measure the static contact angle, and the measured contact angle of each sample is the average value of 5 measurements.

$$\gamma_{l1}(1 + \cos\theta) = 2 \sqrt{\gamma_s^p \gamma_{l1}^p} + 2 \sqrt{\gamma_s^d \gamma_{l1}^d} \tag{A1}$$

$$\gamma_{l2}(1 + \cos\theta) = 2 \sqrt{\gamma_s^p \gamma_{l2}^p} + 2 \sqrt{\gamma_s^d \gamma_{l2}^d} \tag{A2}$$

$$\gamma_T = \gamma_s^p + \gamma_s^d \tag{A3}$$

$$W_a = \frac{4\gamma_s^d \gamma_l^d}{\gamma_s^d + \gamma_l^d} + \frac{4\gamma_s^p \gamma_l^p}{\gamma_s^p + \gamma_l^p} \tag{A4}$$

where  $\gamma_l$ (41.5 mJ/m<sup>2</sup>) is the surface free energy of epoxy resin,  $\gamma_{l1}$ (72.8 mJ/m<sup>2</sup>) is the surface free energy of distilled water, and  $\gamma_{l2}$ (48.0 mJ/m<sup>2</sup>) is the surface free energy of ethylene glycol;  $\gamma_l^d$ (34.3 mJ/m<sup>2</sup>) is the dispersion component of epoxy resin,  $\gamma_{l1}^d$ (21.8 mJ/m<sup>2</sup>) is the dispersion component of distilled water,  $\gamma_{l2}^d$ (29.0 mJ/m<sup>2</sup>) is the dispersion component of ethylene glycol;  $\gamma_l^p$ (10.8 mJ/m<sup>2</sup>) is the polar component of epoxy resin,  $\gamma_{l1}^p$ (51.0 mJ/m<sup>2</sup>) is the polar component of distilled water,  $\gamma_{l2}^p$ (19.0 mJ/m<sup>2</sup>) is the polar component of ethylene glycol;  $W_a$  is adhesion power between fiber and epoxy resin [21].

**Manufacture of composites**

By analysis of the surface energy and adhesion work with epoxy resin, 3 groups with obviously diverse plasma treatment conditions and control group were chosen as reinforced fibers of composites (indicated in Table 2). 1 g fiber of 4 groups were placed in the metal mold for heating and high-pressure curing. Initially cured at 55 °C 1 h and post-cured at 100 °C for 3 h, the size of composite was set with dimension 180 m × 6 mm × 2 mm and volume fraction of fiber was 30%.

### AFM and SEM measurements

The surface microscopy of the fiber was observed by AFM (CSPM5500, Benyuan Co. Ltd., China), and the scan size was (5  $\mu\text{m}$   $\times$  5  $\mu\text{m}$ ). Fracture surface of composites were performed using SEM (TM1000, Hitachi Co. Ltd., Japan). All the samples were gold coated prior to being tested.

### Tensile test

In order to analyze the damage of fibers by different treatments, a fiber tensile testing machine (YG001A, Hongda Co. Ltd., China) was employed to measure the tensile strength of fiber at a gauge length of 30 mm and across-head speed of 20 mm/min. At least 40 fibers in each kind of the sample were prepared to measure.

Composite tensile test proceeded on the electronic strength tester (Instron 3369, USA) which had 5kN load cell. 5 samples of each group were tested at a cross head speed of 2 mm/min.

### Single-fiber composite fragmentation test

At present, single-fiber composite fragmentation test is a reasonable tool to test interfacial shear stress [22–24]. A single fiber was attached into an I-shaped mould, and cured at 100 °C for 4 h after poured epoxy resin into the mould. The dimension of the composite were 10 mm in width, 3 mm in thickness, and gauge length were 20 mm. Samples were strained by a mini tester at a strain rate of 1 mm/min. As shown in Figure 1, with the increase of strain, a growing number fragments occurred at random location within the fiber. The experiment was finished until the fiber cannot break within the limiting region at some strain level [25,26]. The fiber fragments were observed and counted by a computer connected with a polarizing microscope (FM-12, Fuyouma Co. Ltd., China). The interfacial shear strength  $\tau$  is calculated by formula (5).

$$\tau = \frac{\sigma_f d}{2L_c} \quad (\text{A5})$$

Where  $d$  is the fiber diameter;  $\sigma_f$  is the fiber strength  $L_c$  is the critical length, and can be calculated by formula (A6)

$$L_c = \frac{4}{3}\bar{l} \quad (\text{A6})$$

where  $\bar{l}$  is the average fragment length of the samples.

## Results and discussion

### Wettability of fiber

Contact angle of solid surfaces is governed by both chemical composition and surface topographic structure. The value of contact angle is related to the performance of surface energy and adhesion power with epoxy resin. After low temperature plasma treatment,

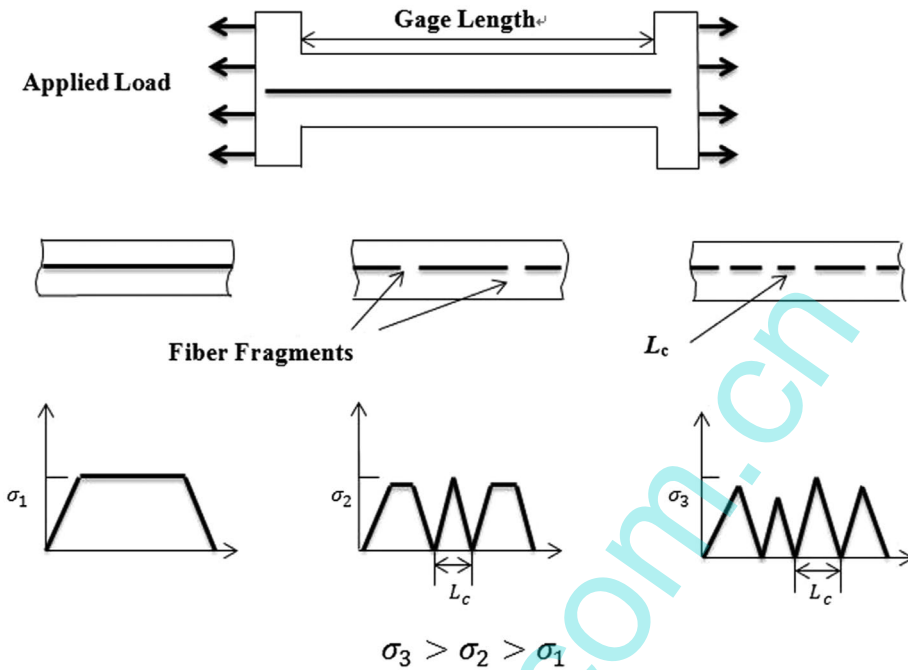


Figure 1. Schematic fracture process of single-fiber composite and the distribution of strength.

Table 1. Contact angle and surface energy of plasma-treated ramie fibers and adhesion work with epoxy resin.

Entry	Treatment condition		Contact angle/(°)		Surface tension/(mJ/m <sup>2</sup> )	Adhesion work/(mJ/m <sup>2</sup> )
	Irradiation time/(min)	Output power/(w)	Water	Ethylene glycol		
1	–	–	83.9 ± 6.2	71.9 ± 5.8	22.3	59.1
2	1	100	65.8 ± 5.5	54.4 ± 5.7	37.4	80.7
3	1	150	60.9 ± 6.1	47.3 ± 5.3	40.8	85.5
4	1	200	57.0 ± 6.3	46.0 ± 5.9	45.2	88.8
5	2	100	56.9 ± 6.1	43.1 ± 6.3	45.5	89.5
6	2	150	54.8 ± 5.4	42.1 ± 5.1	46.6	90.6
7	2	200	52.1 ± 5.2	39.6 ± 5.5	49.4	92.7
8	3	100	51.3 ± 4.9	37.6 ± 5.6	49.7	93.3
9	3	150	50.9 ± 5.5	36.3 ± 4.7	49.8	93.7
10	3	200	50.5 ± 4.5	34.9 ± 5.0	50.0	94.1

contact angle and surface energy of ramie fibers and adhesion power with epoxy resin improved significantly as collected in Table 1. The contact angle decreased and surface energy adhesion power with epoxy resin increased with increasing power at a fixed time.

The untreated fiber showed low polarity and poor wettability (surface energy is 22.3 mJ/m<sup>2</sup>, and adhesion power is 59.1 mJ/m<sup>2</sup>) because of pectin and impurities covering of fibers. After 1 min and 100 w plasma treatment, the pectin and impurities were separated effectively and the fine fiber with irregular striations was revealed, which exhibited improved surface energy 67.8% (37.4 mJ/m<sup>2</sup>) and adhesion power 36.5% (80.7 mJ/m<sup>2</sup>) with respect to the untreated values. In the meanwhile, the contact angle with distilled water and ethylene glycol decreased 21.5 and 24.4%, respectively. The wettability was enhanced with the

increased output power and plasma exposure time. Compared with 1 min-100 w treatment, the higher surface energy and adhesion power were obtained at 2 min-150 w, surface energy ( $46.6 \text{ mJ/m}^2$ ) and adhesion power ( $90.6 \text{ mJ/m}^2$ ) increased 24.6 and 12.3%, and the contact angle with distilled water and ethylene glycol reduced 16.7 and 22.6%, respectively. At 2 min-200 w treatment, the surface energy and adhesion power showed slight variation. The reason was that active particles got more energy, and increased the collision probabilities between fibers and particles that consumed the collision energy between fibers and active particles. Therefore, the effect of plasma treatment decreased gradually so that influenced the increase of surface free energy to some extent. The surface energy and adhesion power were obtained 50.0 and  $94.1 \text{ mJ/m}^2$  after 3 min-200 w treatment, enhanced 6.8 and 3.7%, respectively. The contact angle with distilled water and ethylene glycol diminished 7.8 and 17.1%, respectively. Above all, the properties of fibers were influenced by plasma exposure time and output power, hence, 3 groups of obvious effect (1 min-100 w, 2 min-150 w, and 3 min-200 w) and control group were chosen as reinforced fiber of composites to further study.

### **AFM analysis**

AFM investigation was carried out to measure the morphology and roughness of fiber surface. As Figure 2 showed, the untreated fiber had a relatively smooth surface of 35.0 nm roughness. Surface roughness was obtained as 44.9 nm at 100 w for 1 min, and the surface roughness increased significantly, up to 28.3%. Since fiber was etched relatively rougher after 2 min-150 w treatment [27–29], surface of fiber exposed and some nanocracks distributed unevenly on the fiber surface (Figure 2(C)), and surface roughness (47.8 nm) rose by 6.5% compared to 1 min-100 w treatment. However, the change was not significant when the condition was set as enhanced 3 min-200 w with surface roughness (48.3 nm) similar to that of 2 min-150 w, only with higher 1.1%.

### **Tensile strength**

Tensile strength values of low temperature plasma treated ramie fibers and its reinforced composites were shown in Figure 3. Cellulose is more active and easily etched by plasma treatment [30], resulting in fibers damage to some extent. However, etching action enhanced the contact area between fiber and epoxy resin, which can improve interfacial adhesion and tensile strength values of composites. The tensile strength values of untreated fiber and its composites were 668.8 and 194.4 MPa, respectively. The tensile strength values reduced 7.7%, down to 617.3 MPa by 1 min-100 w treatment. At this moment, plasma treated fiber surface haven't affect cellulose chemical structure obviously yet. The pectin and impurities were removed, which improved the combination between fiber and resin and tensile strength (202.3 MPa), enhancing 4.0%. The treatment damage extent deepened along with the increase of output power and plasma exposure time. Compared with 1 min-100 w, tensile strength was obtained to be 487.7 MPa and decreased 21.0% after 2 min-150 w. Fibers were damaged obviously, and some nanocracks emerged on the surface of fiber. Nevertheless, nanocracks led to higher interfacial contact and surface compatibility performed better, and the tensile strength was obtained 235.6 MPa and increased 16.5%. Tensile strength

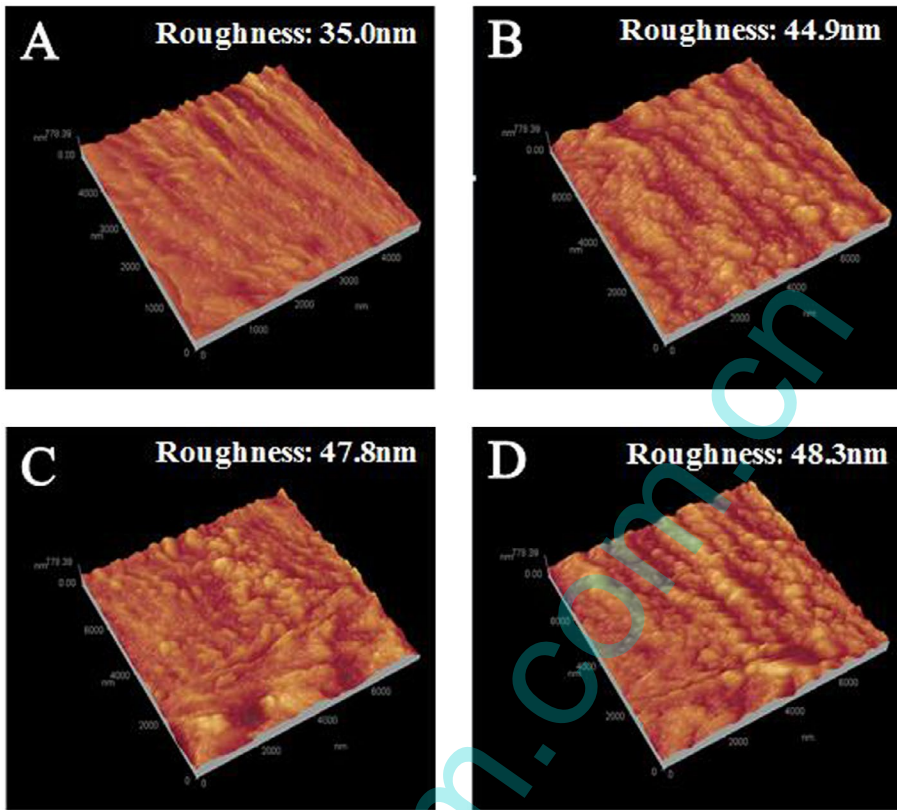


Figure 2. AFM images of ramie fiber with different plasma treatment: (A) Control; (B) 1 min-100 w treated; (C) 2 min-150 w treated; (D) 3 min-200 w treated.

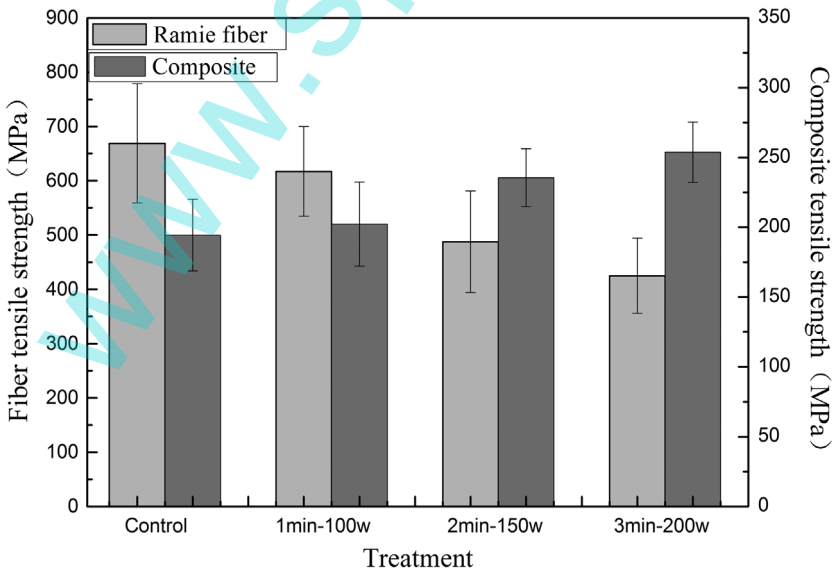


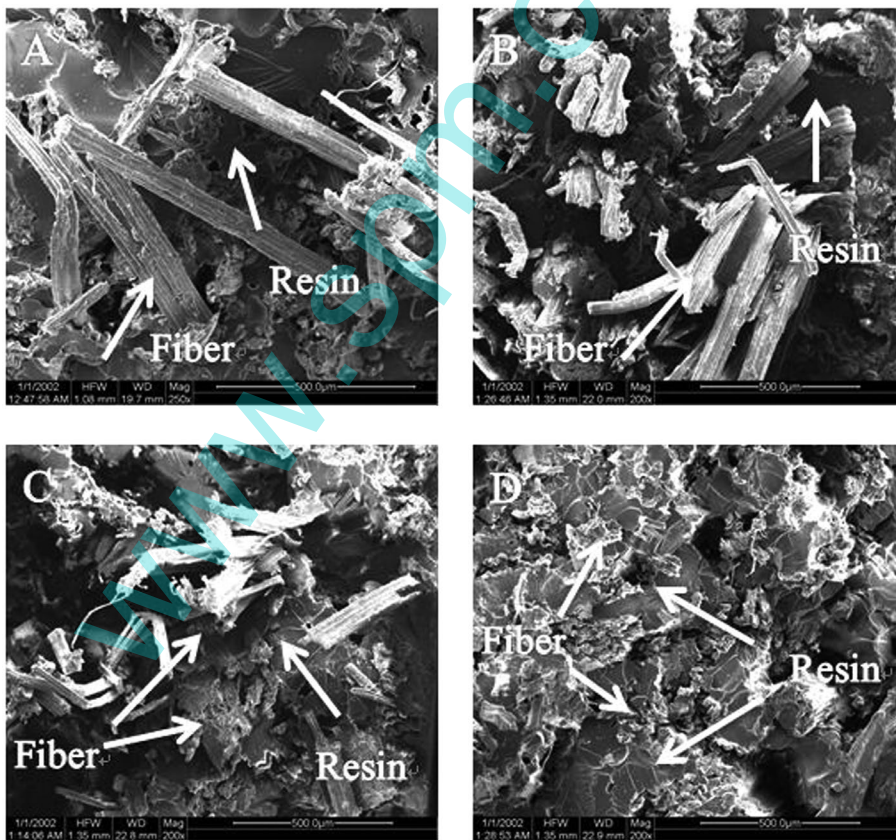
Figure 3. Tensile strength of ramie fiber with different plasma treatment and ramie fiber reinforced composite.



of fibers and composites were obtained 425.1 and 253.8 MPa as result of 3 min-200 w treatment. The ranges were about 12.8 and 7.7%, and the treatment effect changed slowly.

### SEM analysis

SEM micrographs for the fracture surface of ramie fiber reinforced composites with different plasma treatment were displayed at Figure 4. According to critical free energy, once an interface is formed between solid (fiber) and liquid (resin), the condition that interface reaches stability is that the surface energy of liquid is performing at or below the surface energy of solid, and the greater the difference is, the more the stability is [31]. Surface energy of untreated fibers ( $22.3 \text{ mJ/m}^2$ ) was lower than the surface energy of resin ( $41.5 \text{ mJ/m}^2$ ) as Table 1 shows, as well as the bad compatibility of fibers and resin. As shown in Figure 2 (A), the fracture surface of untreated fiber composite was uneven and fibers were pullout without any adhesive resin. This phenomenon can be identified as relatively poor adhesion. Surface energy of the fibers ( $37.4 \text{ mJ/m}^2$ ) got near to that of the resin, which was treated by 1 min-100 w. Consequently, the fibers were not embedded in the resin and the adhesion was not good, but some resin appeared on the fiber surface (Figure 4(B)). With 2 min-150 w



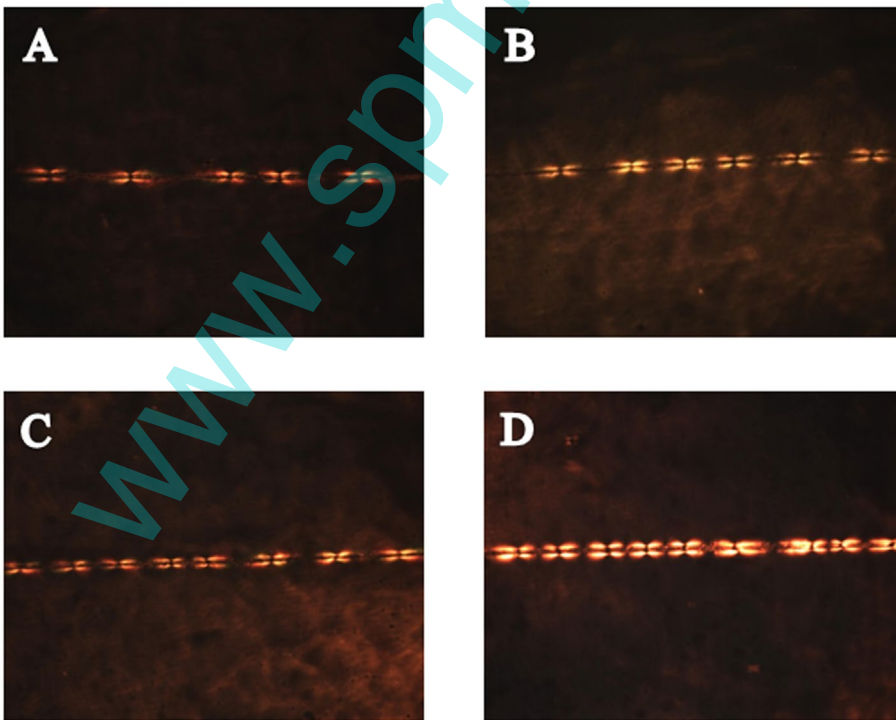
**Figure 4.** SEM images of ramie fiber reinforced composites with different plasma treatment: (A) control; (B) 1 min-100 w treated; (C) 2 min-150 w treated; (D) 3 min-200 w treated.



treatment, the surface energy was changed to  $46.6 \text{ mJ/m}^2$  and the composite performed better with stable interfacial properties. From Figure 4(C), a small amount of fibers were pullout, and fibers were deposited into resin in the course of tension. Fibers and resin were fractured together with an even fracture surface. After 3 min-200 w treatment, the pectin impurity was basically cleaned, and the surface energy of fibers was gotten at  $50.0 \text{ mJ/m}^2$ , which enhanced interfacial properties. From Figure 4(D), the sample was dispersed ductile fracture with a flat fracture, and fibers were covered with resin, which formed the layers on fibers. The result showed that the interfacial adhesion was enhanced obviously.

### *Interfacial shearing stress of single-fiber composite*

Figure 5 and Table 2 showed the results of interfacial shearing stress of single-fiber composite. At the same tension, the number of fracture increased as plasma parameter changed. As given in formula (A5), the interfacial shearing stress of single-fiber composite was influenced by the properties of fiber, resin and interface. Through the characteristic in Table 2, the untreated fiber had 28 breaking points and fragment length was 0.9 mm, with the highest fiber strength (668.8 MPa) was highest. However, as Figure 5(A) shown, the outer pectin impurity of ramie fibers could seriously hindered the combination with epoxy resin so that the shear stress transferred badly. Before fibers broke, the composite had failed. Therefore, the interfacial shearing stress was lowered at 11.2 MPa. When the treatment was at 1 min-100 w, the shear stress (14.4 MPa) was enhanced 29.0% by plasma treatment. As



**Figure 5.** Optical photomicrograph of a fiber fracture with different plasma treatment: (A) control; (B) 1 min-100 w treated; (C) 2 min-150 w treated; (D) 3 min-200 w treated.

**Table 2.** Analysis of single plasma-treated ramie fiber fragmentation.

Treatment condition		Average break $N/(n)$	Average fragment length/(mm)	Fragment length $L_c /$ (mm)	Fiber strength / (MPa)	Interfacial shear strength/ (MPa)
Irradiation time/(min)	Power /(w)					
–	–	28	0.7	0.9	$668.8 \pm 109.8$	$11.2 \pm 1.8$
1	100	39	0.5	0.7	$617.4 \pm 89.5$	$14.4 \pm 2.2$
2	150	50	0.4	0.5	$487.3 \pm 70.1$	$17.2 \pm 2.2$
3	200	46	0.5	0.6	$425.2 \pm 76.2$	$11.5 \pm 2.3$

Figure 5(B) shown, the average break was increased, and the capacity of transferring the stress to epoxy resin was improved. The pectin impurity was removed and the fiber was etched properly at 2 min-150 w. The resin was fully combined with fibers and interface transferred shear stress effectively. Fragment length was shortened by 0.5 mm. Although fibers were damaged by plasma treatment, the improvement of interfacial property had greater influence on the composite, and breaks were distributed uniformly (Figure 5(C)). Compared with the untreated single-fiber composite, the interfacial shearing stress was increased 54.0%, obtained at 17.2 MPa. After 3 min-200 w treatment, a sturdy interface made the stress distribute uniformly, and the birefringence phenomenon was especially obvious (Figure 5(D)). However, the tensile strength of fiber was seriously damaged, which affected the performance of composite. The interfacial shearing stress was similar to untreated single-fiber composite, declined to 11.5 MPa.

## Conclusion

This paper studied influence of the low temperature plasma treatment of ramie fiber on mechanical properties of ramie fiber reinforced composite, and came to following conclusions:

- (1) The wettability was improved by low-temperature plasma treatment. The surface energy of plasma-treated ramie fibers and adhesion work with epoxy resin were increased 124.5 and 59.1% after 3 min-200 w treatment, respectively.
- (2) From the analysis of SEM and AFM, it is discovered that fibers were etched by plasma treatment. The contact area between fibers and resin was increased so that favored fibers adhering with resin effectively. At 3 min-200 w treatment, fibers were covered basically and fibers and resin were fractured simultaneously.
- (3) Fibers were damaged by treatment to some extent, while the adhesion between fibers and resin were improved, therefore the tension strength of composites were increased 30.5% compared with untreated fiber reinforced composites.
- (4) As single-fiber composite test exhibited, interfacial shearing stresses of single-fiber composite were influence by the properties of fiber, resin and interface. Compared with untreated fiber reinforced composites, the best result of interfacial shearing stress (17.2 MPa) was enhanced 54.0% by 2 min-150 w treatment.

In brief, in order to improve the mechanical properties of ramie fiber reinforced composites, the optimization of reaction conditions play the decisive role in the whole experiment.

## Disclosure statement

No potential conflict of interest was reported by the authors.

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