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The uniform treatment of carbon fiber surface in three-directional orthogonal fabric by oxygen-plasma

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An oxygen-plasma treatment of carbon fibre surface in three-directional orthogonal fabric preforms was investigated in this paper. The effects of this treatment on the surface wettability and chemical components of the fibres in both the interior and surface regions of the fabrics were analyzed by using dynamic capillary method and X-ray photoelectron spectroscopy (XPS), respectively. A microdebonding method was employed to determine the uniformity of interfacial shear strength between the fibre and matrix in different regions of the carbon fabric-phenolic composites, and the flexural strength was also tested to evaluate the effects of the treatment. The results indicated that, the oxygen-plasma significantly improved the interfacial adhesion by etching, activating the surface of the fibres, and generating the oxygenic functional groups. However, for the fibres in different regions of the fabric, the degrees of the treatment were different, and a longer treatment time was needed to get the relative uniform effect through the fabric. Meanwhile, the loss of tensile property of the fibre due to treatment was investigated and found to be small in the range of useful treatments.

1. Introduction

Multidirectional carbon fiber fabric preforms have been developed and used as the reinforcement in the liquid molding process of advanced composites to overcome the delamination in the structure [1, 2]. For the special service performance of the composites, however, the fabric preforms with high weave density for the reinforced fiber to obtain a certain volume of fiber are often used, thus often affect the impregnation of resin to the fiber when the matrix resin is injected into the fabric [3]. The generation of voids and defects at the interfaces will affect the load transfer between the fiber and matrix, and finally affect the overall properties of the composites. Therefore, it is the key factor for the multidirectional structure to deal with the wettability and the interfacial adhesion between the fiber and matrix. To this purpose, many investigations have been carried out [4–7], which include the improvement of resin matrix, the analysis and calculation for resin flow model, the *in situ* appraising the flow process and process improvement such as the research on the relationship between injection stress and the resin wettability. Nevertheless, the research on activation treatment on the fiber surface of fabric has not been reported up to now.

Recently, extensive research has been devoted to the surface treatment of carbon fiber filaments in order to improve their bonding to the resin matrix, including dray (gaseous) or wet oxidation [8, 9], electrochemical methods [10–12], polymer coating [13, 14], ultrasonic irradiation [15], plasma etching or grafting [16, 17], and so forth. After treatment, the interfacial adhesion was improved at all levels through increasing the surface free energy or introducing chemical groups on the fibers [18–20]. Compared with other treatment methods, plasma treatments have been widely investigated due to their several advantages [21–24] such as significant improvements of the interfacial adhesion, slight decrease of the tensile properties of the fibre, no

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waste of chemical solution, and environmental friendly processing.

The objective of this paper is to investigate the potential of the oxygen-plasma treatment to uniformly improve the wettability and the interfacial bonding strength in three-directional orthogonal fabric preform, and to develop an understanding of its effect on the mechanical properties of the fabric composites. In this study, the orthogonal fabrics were chemically treated by oxygen-plasma etching, and the treatments were carried out for different periods up to 40 min. XPS, dynamic wettability and microdebonding techniques were used to determine the uniformity of the treatment in different regions of the fabric.

2. Experimental

2.1. Materials

The fibers used for this study were polyacrylonitrile based high-strength and high-modulus carbon fibers (type TSX-111), produced by Jilin Carbon Factory, and specially provided with a lubricant (weaving-type) sizing to protect the fibre against damage during its weaving process. The carbon fibres consist of 1000 filaments in a one-ply construction. Then it was weaved into three-directional orthogonal preform structure (with apparent density of 3.63 g cm^{-1}), which was 10 mm thick, 100 mm wide and 300 mm long, as shown in Fig. 1. The matrix used for the composite preparation was a phenolic resin (type 616-ammonia) produced by Beijing Research Institute of Material and Technology, which was resolved in the ethanol in certain proportion.

2.2. Surface treatment

Before the treatment, with flowing N_2 protection, the weaving-type sizing on the carbon fibre of the three-directional orthogonal fabric preform was removed through thermal decomposition method. It was then placed inside the chamber of a plasma reactor horizontally and the direction along its thickness was vertical to the plasma electrodes, as shown in Fig. 1, followed by plasma treatment. Total of more than 30 treatments were tried by varying the atmosphere, pressure, power and treatment time, to select preferable conditions. In this paper, oxygen was employed as the activation gas; the fabric preforms were treated at 50 Watts of plasma power, 30 Pa of vacuum pressure, and within 40 min of treatment time. To further investigate the characteristics of the oxygen-plasma treatment for the three-directional fabric preform, the carbon fibre surface of unidirectional uncoated filaments was also treated together with the three-directional fabric in the plasma chamber.

2.3. Composites preparation

The three-directional orthogonal composites were manufactured using the high pressure-VA-RTM (vacuum-assisted resin transfer moulding) technology. In the process, the solution of the phenolic resin was injected into the die, where the reinforcement fabric had been placed. The system was evacuated when the temperature reached 110°C and held for 50 min. Then the operations of resin injection and subsequent evacuation were repeated for several times. Eventually, the resin was cured at 170°C for 3 hours. At the same time, the unidirectional carbon fibre-phenolic composites were manufactured using the above unidirectional filaments by compression moulding technology. Some parallel fibre bundles after impregnation with phenolic resin system were aligned in the concave die and the convex die inserted. The resin system was cured at 110°C for 1.5 hours and 170°C for 3 hours at the pressure of 2.0 MPa.

2.4. Contact angle measurement

After the treatment, the wettability between carbon fibre and distilled water was determined, in both regions of interior and surface of the three-directional fabric preform, to investigate the effect of plasma treatment.

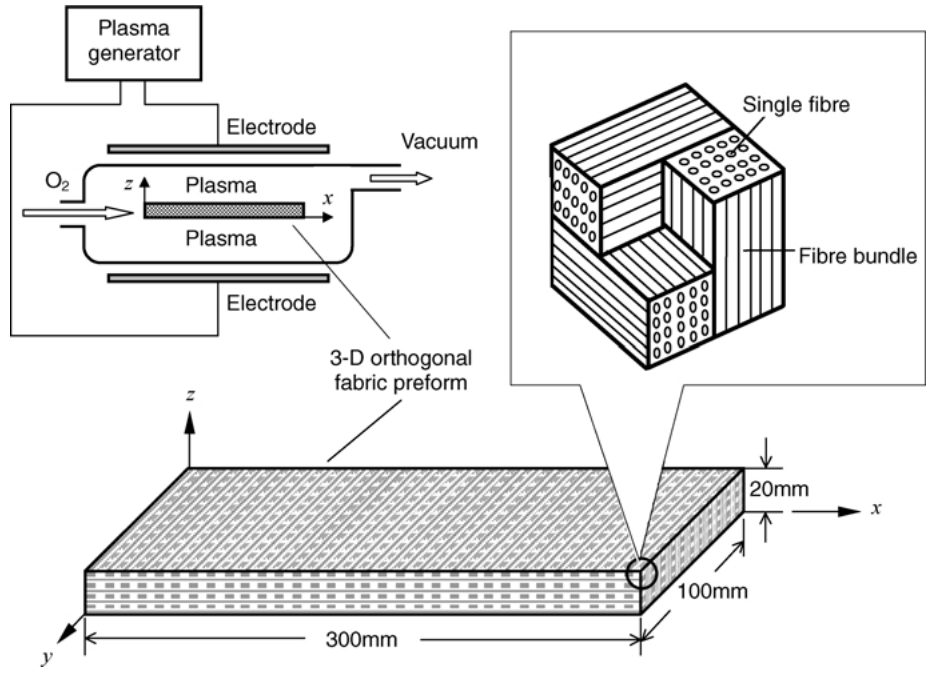

Figure 1 Schematic diagrams of plasma treatment and the structure of three-directional orthogonal fabric preform.

TABLE I Summary of regions in fabrics and composites for various tests

Testing	Region	Coordinate axis of the fabric or composite X (mm)	Y (mm)	Z (mm)
Contact angle[a]	Surface	220–230	0–100[b]	1–3
	Interior	220–230	0–100[b]	4–6
XPS[a]	Surface	240–250	0–100[b]	1–3
	Interior	240–250	0–100[b]	4–6
Microdebonding[c]	Surface	210–213	50–60	1–3[d]
	Interior	210–213	50–60	4–6[a]
Flexural strength[c]	–	10–210	5–45, 55–95	0–10

[a]Samples of fibre bundles pulled-out from the fabric.
[b]Direction of fibre bundles pulled-out.
[c]Samples cut from the composite.
[d]Selecting and pushing an individual fibres of x-direction within the region.

In the test, several fibre bundles were pulled out from the surface and interior regions of the fabric respectively to investigate the uniformity of treatment, as shown in Table I. A dynamic capillary method [25] was used to measure the wetting capacity and saturation time on a SB312-II Dynamic Wetting Determination Equipment. The testing temperature was $25 \pm 0.1^{\circ}$C, and the contact angle between the carbon fibre and the water was calculated using the following equation.

$$\gamma_{sv} - \gamma_{sl} = \frac{64 V_f^2 H_f^2 \rho_f \eta_l m^2}{K^2 W_f D_f \rho_l^2 (V - V_f)^3 t}$$

where γ_{sl} is the free energy of the fibre-water interface, and γ_{sv} is the surface free energy of the fibre in equilibrium with the saturated vapor; ρ_f, D_f, H_f and W_f are, respectively, the density, the diameter, the height and the total weight of the fibre; ρ_l and η_l the density and the viscosity of the water; V and V_f the total volume of the wetting system and the fibres; m is the weight of the water absorbed by the fibres at the time of t; K is the constant, whose value is between 3.3 and 4.0 for the fibre. The contact angle (θ) between the fibre and the water was calculated by the following equation.

$$\cos\theta = \frac{\gamma_{sv} - \gamma_{sl}}{\gamma_l}$$

where γ_l is the surface tension of the distilled water.

2.5. X-ray photoelectron spectroscopy (XPS) analysis

Surface chemical analysis of the fibres in different regions (according to Table I) of the fabric preform before and after plasma treatment was carried out using XPS on a Shimadzu ESCA-750 x-ray photoelectron spectrometer using Mg K_α X-rays at power of 240 W. The instrument was calibrated by Ag 3d spectra which has a peak half width and height of 1.15 eV and 35 kcp, respectively. Functional groups on the fibre surface, such as hydroxyl (C–OH), carbonyl (C=O) and carboxyl (COOH) groups were evaluated by curve fitting of the C_{1s} spectrum using Gaussian distribution.

2.6. Mechanical testing

The mechanical properties including the interlaminar shear strength (ILSS) for the unidirectional composites, and micro interfacial shear strength (IFSS) for both of uni- and multidirectional composites were characterized using short-beam shear and micro-indentation (microdebonding) methods respectively. The ILSS tests were carried out on a Shimadzu AG-20KNIM1STD universal testing machine according to ASTM 2344. The microdebonding tests were performed according to ASTM STP 893 [26, 27], and the specimens with the thickness of 3 mm were respectively cut from the unidirectional composites (perpendicular to the direction of the carbon fibres) and the fabric composites (as shown in Table I). Then the section surface was polished using conventional metallograpgic techniques. In the test, a single fibre was selected, (for fabric composites, selection was based on the different region such as the interior or surface of the materials). Then a diamond micro-probe was used to push axially against the end of the fibre, with the loading speed of 0.01 μm s^{-1}, until interfacial debonding occurred. The load at debond was input to a finite-element analysis program, which calculated the value of IFSS. Each value was the average of more than thirty successful measurements.

Meanwhile, the flexural strength of the three-directional orthogonal fabric composites and the tensile strength of the single fibres were measured to evaluate the effect of treatment. For the flexural tests, specimens with the size of 10 mm × 40 mm × 200 mm were cut from the fabric composites as shown in Table I. A crosshead speed of 5 mm min^{-1} was used according to ASTM 790-90, and the specimens fractured in the test were used for microscopic observation of the fracture surface on a Hitachi S-4300 scanning electron microscopy. Tensile testing of the single-fibre from unidirectional filaments was carried on a WD-I universal testing machine using a 2 N load cell. The loading speed of 1.0 mm min^{-1}, and the gauge length of 20 mm were selected respectively. The tests were performed using at least fifty samples at each treatment condition, and the tensile strength was calculated using Weibull statistic distribution.

3. Results and discussion

3.1. Treatment for unidirectional composites

To understand the effect of oxygen-plasma treatment on interfacial bonding within the carbon-phenolic composite system, and determine the evaluation method of interfacial bonding for the three-directional composites, the ILSS and IFSS of unidirectional composites were first examined under different treatment time of fibre filaments (as shown in Fig. 2). This indicated that, after oxygen-plasma treatment, the interfacial bonding of unidirectional carbon-phenolic composite was improved greatly, especially when treated for 20 min, the value of ILSS increased 23 percent and the IFSS improved about 30 percent. It is generally considered that the oxygen-plasma improved the interfacial bonding performance by etching, cleaning, or activating the surface of the carbon fibre, and generating the active chemical groups. Their physical and chemical effects result in the enhancement of the adhesion strength. It will be discussed and proved below.

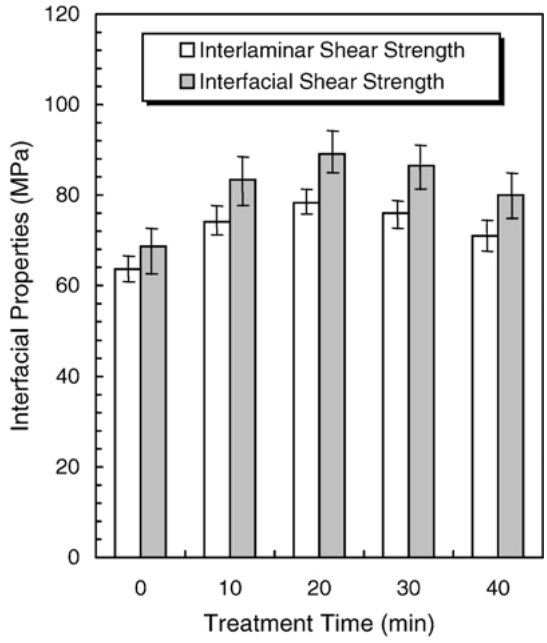

Figure 2 Effect of treatment time on the interfacial properties of unidirectional composites.

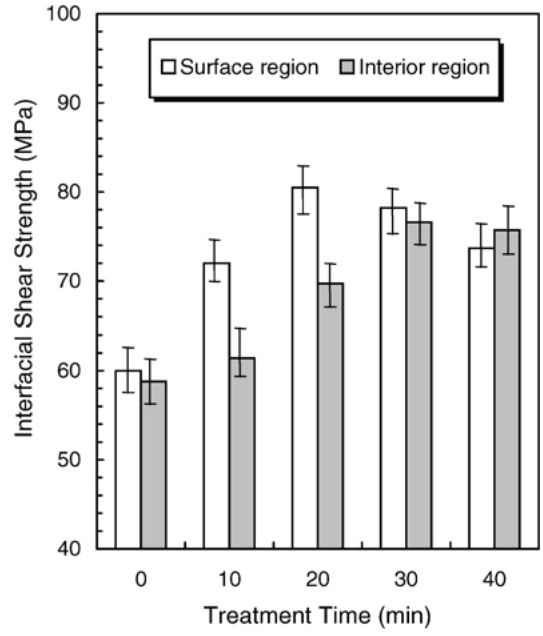

Figure 3 Effect of treatment time on the interfacial shear strength of the fabric composites.

Fig. 2 showed that the interfacial shear strength between single fibre and matrix resin as measured by microdebonding and ILSS follow similar trends to some extent. As we know, short beam shear method is applicable to determine interlaminar shear strength of all types of parallel fibre reinforced polymer composites, and the data can be used for evaluating the interfacial bonding property between the fibre and matrix in the mid-plane of the composite. However, the data depends strongly on the properties, proportions and distributions of the constituents. It is really difficult to evaluate the interfacial bonding for three-directional orthogonal fabric composites because no interlaminar but tensile failure often occurs. In addition, it is difficult to ensure the same distribution of the reinforcement in any two samples cut from the fabric composite. In this paper, therefore, the microdebonding method was utilized to characterize interfacial adhesion, especially in different regions of the sample, and evaluate the uniformity of surface treatment for the three-directional orthogonal fabric.

3.2. The effect of treatment on IFSS of fabric composites

The treatment of the surface of carbon fibre in three-directional orthogonal fabric preform by oxygen-plasma involves in two problems. The one is whether the plasma can activate the fibre surface in the interior regions of the fabric, and the other is whether the extent of treatment in the interior regions is the same as that at the surface region of the fabric. To understand these problems, on the basis of the degree of treatment of unidirectional filaments, the effect of oxygen-plasma treatment on three-directional orthogonal fabric preforms had been investigated, as shown in Fig. 3, the IFSS of the surface region and interior region along the thickness direction of the fabric composite were obtained.

With increasing treatment time, the IFSS values at the surface region increased greatly at the initial stage, after reaching a maximum value at 20 min, the IFSS decreased slowly. Compared with Fig. 2, It had the same tendency toward the data from the unidirectional composites. However, in the interior region of the fabric composite, the IFSS values increased slightly at the initial stage. After treated 20 min, the IFSS increased greatly and reached a maximum value at 30 min. As a result, the degrees of the treatment were different for the fibres in different regions of the fabric, and a longer treatment time was needed to get the optimum effect through the fabric than that of unidirectional filaments.

As we know, plasma is a partially ionized gas containing electrons, ions, free radicals, and neutral atoms or molecules, also called the fourth state of matter. In a plasma chamber, un-ionized particles can be ionized through the collision with the accelerated particles, which have been ionized, and thus they transform energy each other. In a certain region, with the increase of the density of ionized particles, the degree of treatment of the fibre surface increases. Obviously, the compact structure of the fabric hindered the movement of the accelerated particles; as a result, at the initial stage, the interfacial bonding at the surface region was higher than that in the interior regions.

After all, these particles such as electrons, ions, radicals, atoms and molecules of the oxygen-plasma are small enough in size compared with the fabric structure. Under a longer treatment time, such as 30–40 min, they infiltrated into the compact fabric to activate the fibre surface in the interior regions. However, although these conditions ensured that the interior regions of the fabric obtained a satisfactory treatment, the fibres at the surface region were treated excessively, and thus resulted in the IFSS of the surface region decreased slightly (at the treatment time of 30 min).

3.3. Effect of treatment on chemical components of fibre surface

The result of XPS analysis of the five samples pulled-out from different regions of the fabric, before and

TABLE II Atomic percentages and oxygenic groups on fibre surface, as obtained by XPS

Treatment time (min)	Region	Atomic percentages[a]		Content of functional groups, area (%)[b]			
		C	O	Graphite	C—O(R)	C═O and/or Quinone	O═C—O(R)
Control	–	96.1	3.9	78.1	13.5	6.2	2.2
20	Surface	89.5	10.5	74.3	16.4	6.4	2.9
	Interior	93.8	6.2	75.6	15.0	5.8	3.6
30	Surface	88.6	11.4	74.2	13.3	5.7	6.8
	Interior	90.5	9.5	74.9	14.5	5.2	5.4

[a]Obtained from C_{1s} and O_{1s} spectrum.
[b]The ratio of each peak's area for C_{1s} spectrum obtained by curve fitting.

after treatment, using the treatment time of 20 and 30 min respectively, were given in Table II. From a semi-quantitave comparison of relative atomic percentages of oxygen and carbon on the fibre surface for the oxygen-plasma treatments, it was obviously found that plasma oxidation increased greatly the oxygen concentration, thus indicating the creation of oxygenic functional groups on the fibre surface. In addition, Table II also showed that carbon fibres in different regions of the fabric were oxidized to different degrees. In the case of 20 min treated, the fibres at the surface region had a much more percentage of oxygen than that of the fibres in the interior region, which proved that the fibres at the surface region were oxidized more than those in the interior region. After 30 min treated, however, the fibres in both regions were oxidized to a similar degree. This trend corresponded with that of the interfacial bonding.

High-energy resolution investigation of the C_{1s} peak envelops was carried out to determine the types and contents of oxygenic functional groups, the results were also illustrated in Table II. Beside the major peak identified at 284.6 eV due to graphitic carbon, three additional shifted peaks were found at 1.6, 2.7 and 4.1 eV due to hydroxyl and/or ether, carbonyl and/or quinone, and carborxyl and/or ester groups respectively. It revealed that the relative numbers of C—O(R) groups had a much more increase after 20 min treated, while those of O═C—O(R) groups increased considerably after 30 min treated. It seemed that the condition for each functional group produced was quite different, and these functional groups could be converted from one type to another type with the change of treatment degree (such as from C—OH to O═COH). Moreover, the numbers and special types of functional groups contributed differently to the fibre-matrix interactions (polar interaction and chemical reaction). Although these reactions have not been deeply understood, oxygen-plasma introduced a numbers of oxygenic functional groups onto the fibre surface, thus were benefits to the interfacial bonding.

In general, XPS results proved that, in different regions of the fabric, the fibre surfaces were oxidized to different levels, and a nearly uniform treatment could be obtained in the fabric by increasing the treatment time.

3.4. Effect of treatment on wettability of fibre surface

Fig. 4 exhibited the effect of treatment time on the wettabilities between carbon fibres and distilled water, which the fibres were respectively pulled-out from the interior and surface regions of the fabrics. It revealed that the plasma treatment decreased the contact angle

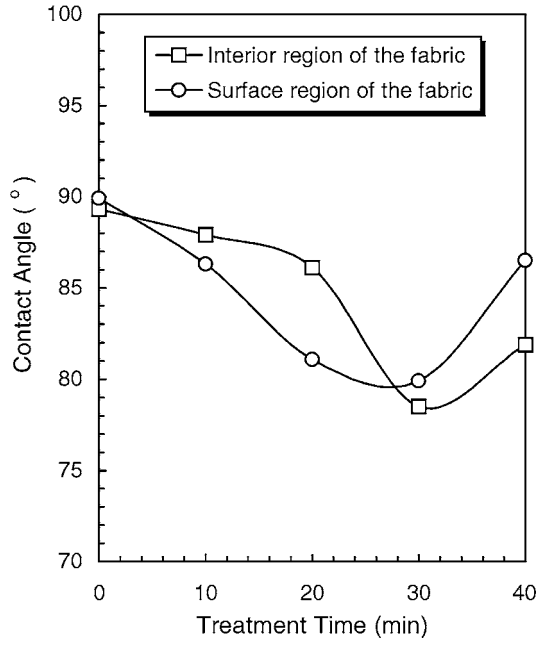

Figure 4 Wettability between carbon fibers and distilled water.

between distilled water and the fibres in the both regions of the fabric. As mentioned above, plasma-oxidation introduced chemical active functional groups onto the fibre surface, thus increasing the polar part of the surface free energy of the fibres. On the other hand, the

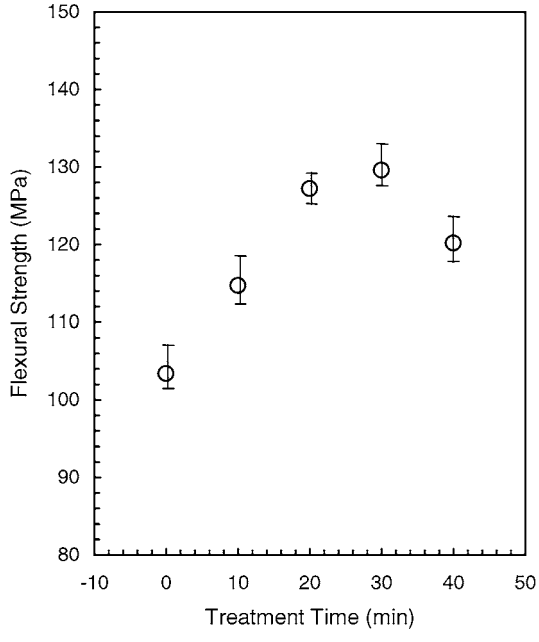

Figure 5 Effect of treatment time on flexural strength of carbon fabric-phenolic composites.

plasma also increased the roughness of the fibre surface by etching and cleaning the fibres thus increasing their surface area. As a result, the plasma increased the dispersive part of the surface free energy of the fibres. Both chemical and physical contributions resulted in the improvement of the wettability. However, when the treatment time was longer than the 30 min, the wettability decreased due to excessive etching and removing the modified layer of the fibre surface.

Comparing the wettabilities of the fibres in the different regions of the fabric, it was found that their changing trends were different, especially at the initial stage of the treatment. At this stage (from 0 min to 20 min), the contact angle decreased greatly at the surface region, while slightly in the interior region. It was well accorded with the results of XPS and interfacial bonding strength.

3.5. Effect of treatment on flexural strength of fabric composites

Fig. 5 exhibited the relationship between oxygen-plasma treatment time and flexural strength of the fabric composites. As treatment time increased, the flexural strength increased greatly, subsequently decreased. When the treatment time was 30 min, the strength was the highest, 130 MPa and 25% higher than that of untreated specimen. This trend was in accordance with the

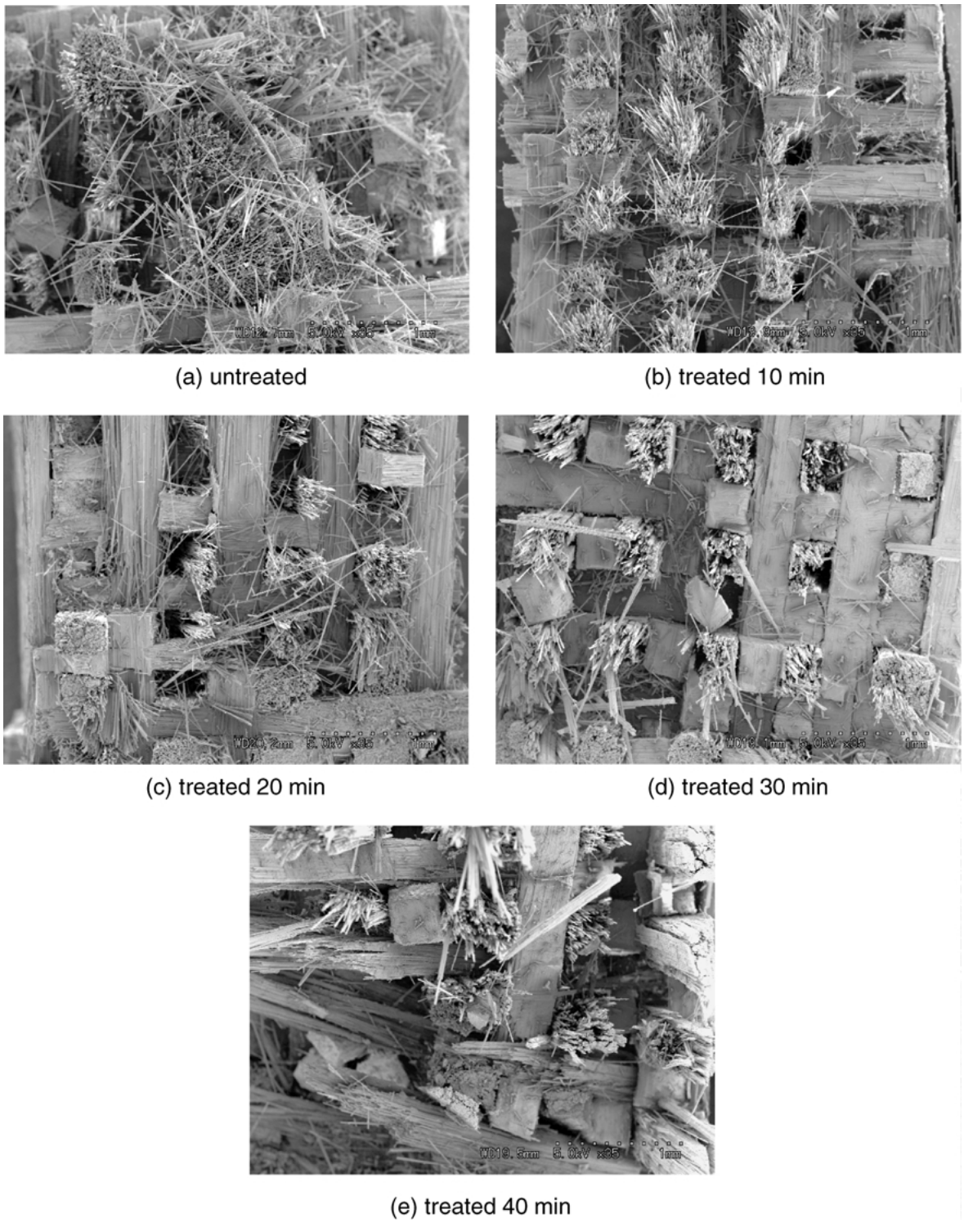

Figure 6 Scanning electron micrographs of the fracture surfaces after flexural tests.

changes of averaged interfacial shear strength in both interior and surface regions of the composites as shown in Fig. 3.

The characteristics of all of the reinforced fibre, resin matrix and interfacial adhesion contributed to the flexural strength of sample. For a given system of constituent materials (phenolic resin matrix and carbon fibres) such as the fabric composites in this study, only the interfacial bonding determined the flexural strength of the composites. Accordingly, the flexural strength of the composite was depended upon the interfacial adhesion strength, thus their trends were the same.

The fracture surfaces after the flexural tests were observed by scanning electron microscopy, as shown in Fig. 6. As we know, under the flexural test, different regions in the specimen were applied different combined stress. Moreover, compared with unidirectional composites, the distributions of the stress in the three-directional composites were more complex. Therefore, it was difficult to determine the difference of the interfacial bonding in the interior and surface regions from the fracture surface. Nevertheless, the general characteristics of interfacial adhesion for each specimen, before and after treatment, could be determined using a full view (panorama) way, as shown in Fig. 6a–e. It indicated that oxygen-plasma treatment changed the flexural fracture mode of the fabric composites. For the untreated specimen, it exhibited a messy and scattered fracture surface, a lot of x-directional fibres were pulled-out due to poor interfacial adhesion; While a series of flat and neat fractures, comparatively, appeared in association with a strong interfacial bonding in the photographs of the treated specimens. Comparing these figures, we could conclude that the fracture surfaces were quite in accordance with the interfacial adhesion.

3.6. Effect of treatment on fiber strength

To get a relative uniform improvement of interfacial adhesion through the fabric, a longer treatment was needed than that of unidirectional filaments. Thus it should be fully considered the effect of plasma treatment on the strength of fibres in the fabrics. In this study, to avoid the damage of the fibres during pulled-out from the compact fabric, samples for tensile strength tests were selected from unidirectional filaments. Table III displayed the relationship between the tensile strength of single fibre and treatment time, the practical outcome was calculated according to Weibull statistical analysis. After fiber filaments have been treated at 30 min, the tensile strength decreased by 5.3 percent. The small decreases proved the etching and cleaning effects of the oxygen-plasma, and generating some micro- holes and grooves on the fibre surface, which resulted in the stress concentration and precocious failure of the fibre during the test. Nevertheless, from the above analysis, it indicated the etching effect of plasma on fiber surface of three-directional fabric, especially in its interior regions, was not more than that of unidirectional filaments. Therefore, the loss of tension properties of the fibres due to the treatment was small in the range of useful treatments.

TABLE III Effect of treatment on the tensile strength of single fibre

Treating time (min)	Shaping parameter	Characteristic tensile strength (GPa)	Mean tensile strength (GPa)
0	6.51	3.61	3.40
10	6.55	3.53	3.36
20	6.50	3.48	3.30
30	6.53	3.46	3.22
40	6.58	3.24	3.07

4. Conclusions

Oxygen-plasma treatment of three-directional orthogonal carbon fabrics resulted in an improvements of the mechanical properties of their phenolic composites. As an overall result, the interfacial shear strength of the fabric composites was greatly increased by plasma oxidation. The treated fibres exhibited a very significant increase of wettability, thus improving the processability of their fabric composites. XPS analysis results revealed a much higher oxygen percentage on the surface of treated fibres.

IFSS, wettability, and XPS analysis results proved that, in different regions of the fabric, the fibre surfaces were oxidized and etched to different levels, and a nearly uniform treatment of the fibres in both the interior and the surface regions of the fabric could be obtained by increasing the treatment time compared with the conventional carbon filaments. After treated 30 min, the flexural strength of the fabric composite was increased more than 25%. The flexural micrographs supported the ability of the plasma to enhance the adhesion of the fibre to the matrix. Moreover, the loss of tensile property of the fibre due to treatment was found to be small in the range of useful treatments.

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References

1. S. RAMAKRISHNA, H. HAMADA and K. B. CHENG, *Composites, Part A* **28** (1997) 25.
2. C. M. PASTORE, *Journal of Mechanics of Composite Materials* **36** (2000) 165.
3. A. CHAN and S. T. HWANG, *Polymer Engineering and Science* **31** (1991) 1149.
4. J. BRANDT, K. DRECHSLER and F. J. ARENDTS, *Composites Science and Technology* **56** (1996) 381.
5. R. K. SURYA and F. ERIC, *ibid.* **57** (1997) 293.
6. M. U. KWANG and W. I. LEE, *Polymer Engineering and Science* **31** (1991) 765.
7. C. W. LEE and B. P. RICE, *SAMPE Journal* **34** (1998) 6.
8. A. FUKUNAGA, S. UEDA and M. NAGUMO, *Carbon* **37** (1999) 1081.
9. W. H. LEE, J. G. LEE and P. J. REUCROFT, *Applied Surface Science* **171** (2001) 136.
10. J. M. PARK, Y. M. KIM and K .W. KIM, *Journal of Colloid Interface Science* **231** (2000) 114.
11. R. V. SUIBRAMANIAN and J. J. JUKUBOWSKI, *Polymer Engineering and science* **18** (1981) 590.
12. S. YUMITORI and Y. NAKANISHI, *Composites, Part A* **27** (1996) 1051.

13. J. P. BELL, J. CHANG, H. RHEE and R. JOSEPH, *Polymer Composites* **8** (1987) 41.
14. W. J. TOMLINSON and R. S. BARNES, *Journal of Materials Science Letters* **11** (1992) 440.
15. L. LIU, Y. D. HUANG and Z. Q. ZHANG, *Journal of Applied Polymer Science* **81** (2001) 2764.
16. R. LI, L. YE and Y. W. MAI, *Composites, Part A* **28** (1997) 73.
17. C. L. HAMERMESH and P. J. DYNES, *Journal of Polymer science* **13** (1975) 663.
18. N. DILSIZE and J. P. WIGHTMAN, *Journal of Colloids and Surfaces* **164** (2000) 325.
19. Y. HUANG and R. J. YOUNG, *Composites Science and Technology* **52** (1994) 505.
20. R. H. BRADLEY, X. LING and I. SUTHERLAND, *Carbon* **31** (1993) 1115.
21. S. FEIH and P. SCHWARTZ, *Journal of Adhesion Science and Technology* **12** (1998) 523.
22. K. E. ATKINSON, G. J. FARROWT and C. JONES, *Composites, Part A* **27** (1996) 799.
23. M. A. MONTES-MORAN, A. MARTINEZ-ALONSO, J. M. D. TASCON and R. J. YOUNG, *Composites, Part A* **32** (2001) 361.
24. A. BISMARCK, E. KUMRU and J. SPRINGER, *Journal of Colloid Interface Science* **210** (1999) 60.
25. S. CHWASTCAK, *Journal of Interface Science* **42** (1973) 298.
26. J. F. MANDELL, D. H. DOND and F. J. McGARRY, *ASTM STP* **893** (1986) 87.
27. Y. D. HUANG and Z. Q. ZHANG, *Journal of Materials Processing Technology* **37** (1993) 559.

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