

Surface characteristics of kidney and circular section carbon fibers and mechanical behavior of composites

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Abstract

A comprehensive experimental study was conducted to identify the differences of the kidney section carbon fibers and circular section carbon fibers in the surface characteristics of fibers and mechanical properties of composites. It was revealed that the kidney fibers with larger specific surface area have a better adsorption characteristic and higher impregnating performance compared with the circular fibers. Mechanical tests under interfacial shear, interlaminar shear, flexural and compressive load have shown that the kidney fiber/epoxy composites outperform the circular fiber/epoxy composites by a significant improvement of 23.5, 12.7, 7.5 and 4%, respectively. The tensile strength is nearly the same for the two composite systems, but the failure of kidney fiber/epoxy tensile specimens undergoes a cumulative damage progression process and is different from that of circular fiber/epoxy tensile specimens. Also, the kidney fibers have a more non-uniform distribution in composites and larger contact areas with each other compared with the circular fibers, and the mechanical properties of kidney composites have a larger coefficient of variation than those of circular composites.

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1. Introduction

Carbon fibers are currently the most extensively used reinforcements for composites because of their availability and reliability. One of the main requirements for continuous fiber-reinforced composite materials to be successfully used in practice is their mechanical performance. Most work in the development of composites has considered fiber/matrix adhesion to be a necessary factor to ensure good mechanical properties. The sensitivity of the mechanical behavior of composites to fiber/matrix adhesion has long been realized [1,2]. Fiber/matrix adhesion involves very complex physical and chemical mechanisms. The majority of previous efforts were concentrated on the chemical aspects of fiber/matrix adhesion through the use of surface treatments and coatings [1]. However, not much attention has been paid to the physical aspects of fiber/matrix interaction. One of the most important physical aspects is the geometry of reinforcing fibers, which influences

adhesion between fiber and matrix, stress transfer and local mechanisms of failure [3,4]. In addition to chemical bonding, the fiber/matrix bond strength in shear is largely dependent on the roughness of the fiber surface and the fiber/matrix contact area. Recently carbon fibers with deformed cross-sectional shapes have been developed. In structural mechanics, as the optimization of the stress distribution of materials, some design engineers proved that hollow or noncircular-type fiber is better than circular one in mechanical properties and that they have applied in many structural materials, such as I-beam train road, construction support pipe/pile rod, etc. [5–8]. Especially, C-type carbon fiber has a curved area in the surface contacting with matrices that can improve interfacial bonding force. The phenomena result may solve a delamination, playing a great part in the mechanical properties of carbon fiber-reinforced composites [9,10].

In this work, we are to investigate the surface characteristics of circular and kidney fibers and study the two composite systems reinforced by carbon fibers with different fiber cross-sectional shapes (circular and kidney) to identify the influence of fiber cross-sectional irregularity on mechanical properties of composites.

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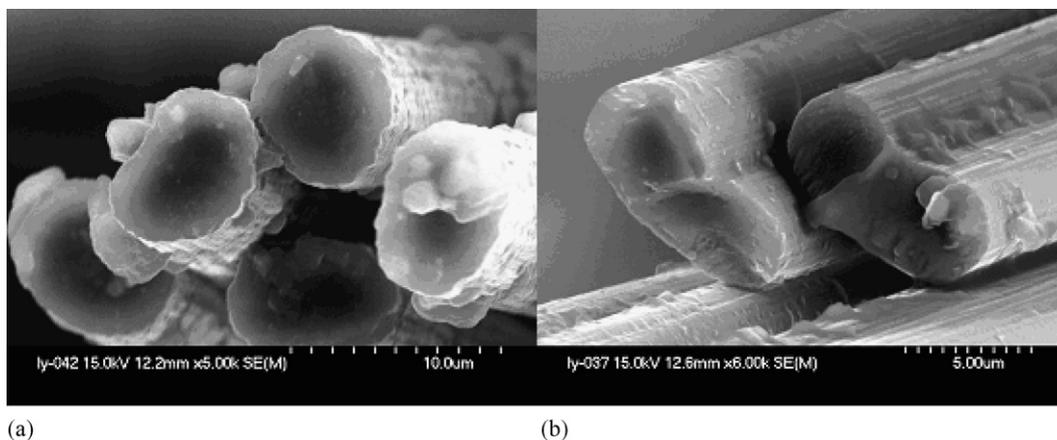


Fig. 1. Photographs of fiber shape. (a) Circular fiber; (b) kidney fiber.

2. Experimental

2.1. Materials

Two types of polyacrylonitrile (PAN) carbon fibers used were produced by Institute of Coal Chemistry affiliated with the Chinese Academy of Sciences in the same manufacturing conditions such as spinning, holding time of stabilization and carbonization. The average diameter of carbon fiber with circular cross-sectional shape is $7\ \mu\text{m}$, shown in Fig. 1a. The cross-section of kidney fiber with the width of $9\ \mu\text{m}$ and the thickness of $3\ \mu\text{m}$ is shown in Fig. 1b.

The matrix system used was E-618 epoxy resin system consisting of diglycidyl ether of bisphenol-A (supplied from Yue-Yang Chem. Co. of China), curing agent: phthalic anhydride and benzyl dimethylamine at 100, 70 and 1 parts by weight, respectively.

Using the carbon fibers and epoxy resin, the prepreg was put unidirectional into a mold to manufacture composites. The prepreg was pressed and cured under 5 MPa pressure for 2 h at 90°C , then under 10 MPa pressure for 2 h at 120°C and last under 10 MPa pressure for 4 h at 150°C by hot-press machine and we could obtain specimens with fiber mass fraction of $65(\pm 1.5\%)$ and fiber volume fraction of $58(\pm 1\%)$.

2.2. Measurements

Cross-sections of fibers and failure of fiber-reinforced composites were inspected by scanning electron microscopy (SEM) Hitachi S-4700. The adsorption characteristics of fibers were determined by generating N_2 adsorption isotherms at -196°C using ASAP2405 apparatus (USA). BET specific surface area, pore volume, and pore average diameter of fibers were obtained from the nitrogen adsorption isotherms.

The wettability of the fibers was measured by a capillary method in an impregnating apparatus SB-312 made in China. In the experiment, some bundles of fiber were aligned and inserted into a polyethylene tube with diameter 2 mm and length 30 mm. The bundles protruded 2 mm outside the tube. The ends of the bundles were impregnated by water and normal octane, respectively.

For the investigation of mechanical properties, a micro-composite (single fiber composite) test was performed to determine the interfacial shear strength (IFSS) of the kidney and circular section carbon fiber/epoxy resin composites. The micro-composite specimens were prepared in paper frames with dimensions of $20\ \text{mm} \times 100\ \text{mm}$. The free fiber length was approximately 30 mm. Some resin droplets were placed against a monofilament and cured (Fig. 2a). A single fiber pull-out test was carried out using an interfacial micro-bond evaluation instrument made by Tohei Sanyon Corporation of Japan (Fig. 2b).

Interlaminar shear strength (ILSS) of the composites was measured by short-beam bending test according to ASTM D-2344 using an Instron 1125. A span-to-depth ratio of 5:1, cross-head speed of $2\ \text{mm}\ \text{min}^{-1}$, and sample thickness of 2 mm were used. Flexural properties of composites were determined according to ASTM D-790 ($L/d = 15$; cross-head speed = $1.5\ \text{mm}\ \text{min}^{-1}$).

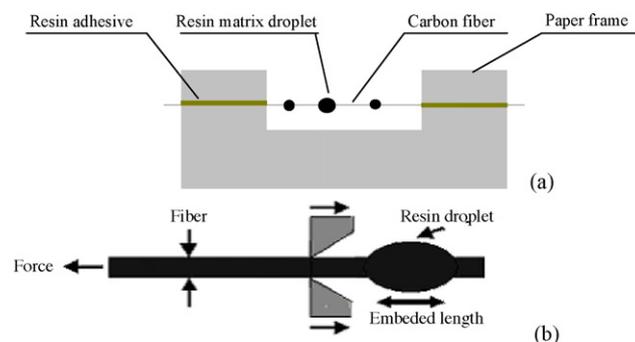


Fig. 2. (a) Single fiber pull-out specimen. (b) Schematic drawing of IFSS test.

Tensile tests were carried out, following ASTM standard D3039-93 at a cross-head speed of $2\ \text{mm}\ \text{min}^{-1}$. Compressive strength was measured according to the ASTM D-695-58 at a crosshead speed of $1\ \text{mm}\ \text{min}^{-1}$.

More than eight specimens were tested for each of composites studied and the average value was taken in the present work studied.

3. Results and discussion

3.1. Adsorption characteristic of fibers

Carbon fibers which are used as reinforced materials should possess a reasonably large specific surface area, which allows the exposure of active phase to the matrix. Fig. 3 shows N_2 adsorption–desorption isotherms of the kidney and circular fibers. The isotherms of both groups (kidney and circular) resemble each other and are classified as type I in the classification of International Union of Pure and Applied Chemistry (IUPAC) [11]. At the any relative pressure, the kidney fibers adsorbed noticeably more N_2 than the circular fiber.

As previously mentioned, nitrogen isotherms were analyzed by the Brunauer-Emmett-Teller (BET) method to obtain the specific surface area, pore volume and average pore diameter of carbon fibers studied. Typical results are shown in Table 1. It should be noted that the specific surface area, pore volume and average pore diameter of kidney fibers with equivalent production conditions are larger than those of circular fibers, respectively. It is indicated that the adsorption characteristic of

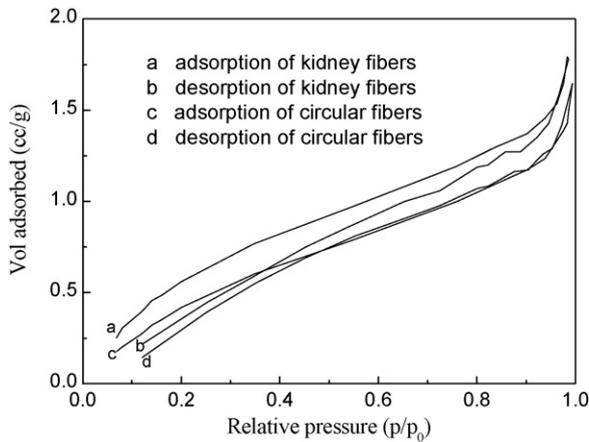


Fig. 3. Nitrogen adsorption–desorption isotherms for kidney and circular section carbon fibers.

Table 1
Pore structure characteristics of carbon fiber

	Pore volume (ml·g ⁻¹)	Specific area (m ² ·g ⁻¹)	Average diameter (nm)
Kidney section fiber	0.003	3.03	3.7
Circular section fiber	0.002	2.64	3.5

kidney fibers is better than that of circular fibers with similar manufacturing conditions.

3.2. Impregnating performance of fibers

Fig. 4 shows the dynamic wetting curves of water and normal octane on kidney and circular fibers. It indicates that the wetting weight of kidney fibers at equilibrium is larger than that of circular fibers and the complete wetting time of kidney fibers is longer than that of circular fibers in water and normal octane, respectively. It is because the specific surface area, pore volume and average pore diameter of kidney fibers are larger than those of circular fibers, respectively, which is shown by BET measurement [12,13].

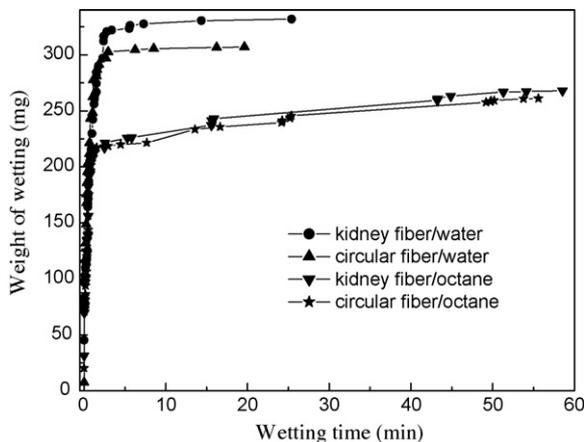


Fig. 4. Dynamic wetting curves of water and normal octane on carbon fibers.

3.3. IFSS and ILSS of composites

For fibers with irregular cross-sections, such as kidney section fibers, the original shear lag model would not provide accurate calculations for interfacial shear strength, because it assumes a circular fiber cross-section. In this paper, a modified shear lag model [14] is employed to calculate the interfacial shear strength by the following Eqs. (1) and (2).

$$\tau_f^* = \frac{\sigma_p n^* \pi d^*}{S_f} \coth\left(\frac{n^* L}{d^*}\right) - \tau_{rf} \quad (1)$$

$$n^* = \left[\frac{E_m}{E_f(1 + \nu_m) \ln[S_R/S_f]} \right]^{1/2} \quad (2)$$

where τ_f^* is the fiber/matrix interfacial shear strength; σ_p the maximum fiber tensile stress during pullout test; n^* the dimensionless shear lag constant; d^* the equivalent diameter of fiber deduced from fiber cross-sectional area; L the embedded length of fiber; τ_{rf} the residual shear stress at the fiber surface or at an annulus; E_f or E_m the tensile modulus of fiber or that of matrix; ν_m the Poisson's ratio of matrix; S_f or S_R is the circumference length of fiber cross-sectional area or that of an annulus in matrix.

IFSS, τ_f^* , values were calculated using Eq. (1) for the modified shear lag models, assuming the residual stress was zero and the results are shown in Table 2.

Table 2 represents mechanical results studied on IFSS, ILSS, flexural strength, compressive strength and tensile strength of composites reinforced with two types of carbon fibers. These results indicate that the kidney fiber/epoxy composites show the higher values in almost all mechanical properties than the circular fiber/epoxy composites.

It is noted that when Eq. (1) is used, the IFSS for kidney composites is 23.5% greater than the IFSS for circular composites. In addition, the kidney composites offer a large (approximately 12.7%) improvement in mean ILSS over the circular composites. This result is likely due to the fact that the basic properties of kidney fibers widen specific surface area and enhance the contact area with matrix, resulting in effectively transferring the applied stress.

3.4. Flexural property of composites

The flexural strength reveals that kidney fiber-reinforced composites are improved by 7.5%, comparing with circular fiber-reinforced composites. This is probably due to the stiffness of kidney fibers itself and effective stress interaction between fibers and matrix.

We can verify it as observing the failure mode by using SEM pictures. Fig. 5a and b shows the failure surface of kidney fiber/epoxy composites and that of circular fiber/epoxy composites after flexural test, respectively. These pictures represent that the matrix is better engulfed by the hollowed-out area of kidney fiber, which allows the matrix to secure more bonds and better adhesive force between two phases, which can effectively transfer the load applied to the fiber-reinforced composite system.

Table 2
Mechanical properties of composites

	IFSS (MPa)	ILSS (MPa)	Tensile strength (MPa)	Compression strength (MPa)	Flexural strength (MPa)
Kidney	26.8 ± 0.9	73.7 ± 1.2	1682.6 ± 82.6	805.8 ± 27.2	1468.1 ± 51.6
Circular	21.7 ± 0.7	65.4 ± 0.8	1669.8 ± 80.3	774.9 ± 21.0	1366.2 ± 43.7

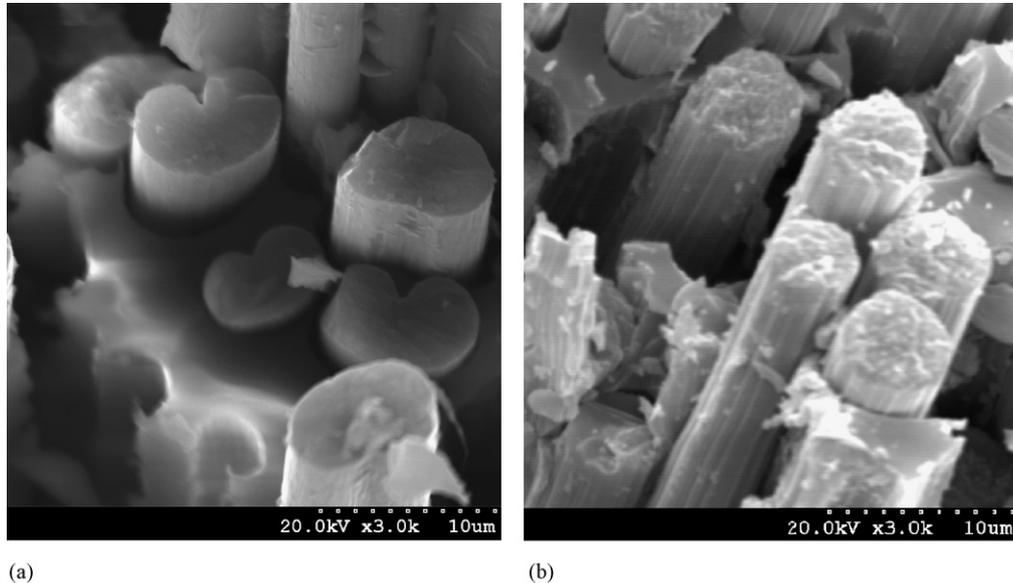


Fig. 5. SEM photographs of carbon fiber/epoxy composites. (a) Kidney fiber; (b) circular fiber.

3.5. Compression of composites

The compressive testing results are shown in Table 2. It is clear from these that the kidney composites have a better compressive performance. For the same volume fraction, the kidney fiber/epoxy composites have a compressive strength of 805.8 MPa, compared with 774.9 MPa for the circular fiber/epoxy composites, a difference of 4%. However, the scatter of results in the former case is somewhat great with a coefficient of variation of 3.4%. Thus, it could be argued that the apparent increase in compressive strength is a little uncertain. It is suggested that the kidney fiber/epoxy composites outperform the circular fiber/epoxy composites in this instance. Thus, at this stage, it is difficult to draw a definite conclusion as to the extent of increase in compressive strength due to the kidney fiber shape, but it does appear that some improvement has occurred.

3.6. Tension of composites

It is well known that the tensile strength of carbon fiber is substantially higher than that of epoxy. Therefore, the tensile strength of composites is primarily determined by the tensile strength of carbon fibers. The tensile testing results in the longitudinal direction are shown in Table 2. The values of tensile strength of the two composite systems are nearly the same, though some fiber breakages occurred for kidney fiber/epoxy composites before the peak force was reached. However, the failure behavior of kidney fiber/epoxy tensile specimens is different from that of circular fiber/epoxy specimens. For the

circular fiber/epoxy system, when the applied force reaches the maximum, fiber breakage appears and the failure of specimen happens suddenly, followed by the force decrease abruptly to zero. However, the failure of kidney fiber/epoxy tensile specimens undergoes a cumulative damage progression process. When the breakage of some fibers occurs, the remaining fibers still withstand some load, followed by subsequent breakage of more fibers. The process continues until the final failure of the specimen happens without sudden drop of the load. In Fig. 6, the area under the force/strain curve for kidney fiber/epoxy specimens is clearly larger than that of circular fiber/epoxy, which

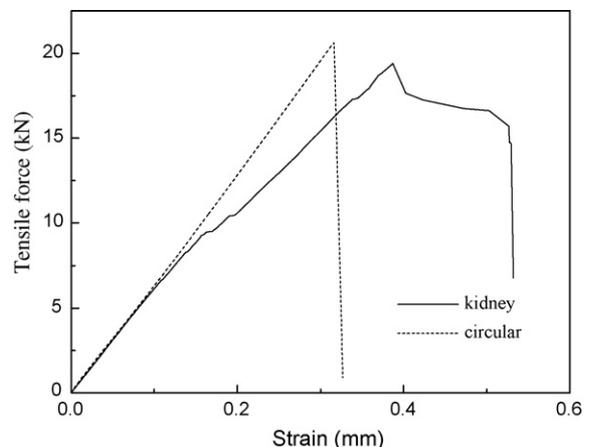


Fig. 6. Tensile stress/strain curves for two types of carbon fiber/epoxy composites.

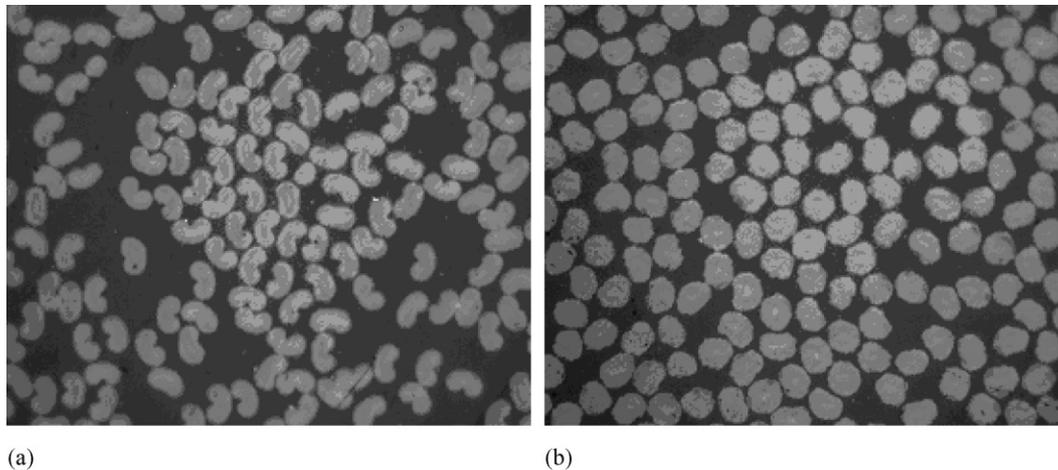


Fig. 7. Optical micrographs of unidirectional carbon fiber/epoxy composite showing fiber distribution. (a) Kidney fiber; (b) circular fiber.

means that the fracture of kidney fiber/epoxy absorbed more energy [2].

For kidney carbon fibers, the fibers have the preferred alignment because of the fiber rotation during fabrication of composites. As a result, there are some fibers overlapped with each other with large contact areas and the matrix resin is too thin to play the role for stress transfer. When tensile stress is continuously applied to the specimens of carbon fiber/epoxy, some kidney fiber breakage will happen firstly, resulting in the kidney fiber deviation from the curve of the circular fibers at low strain [2].

3.7. Micrographs of composite sections

For the two composite systems studied in this work, the reinforcing fibers have almost identical mechanical properties, differing only in their cross-sectional shapes. The specimens of composites also have a similar fiber volume fraction. However, micro-structures of the composites are somewhat different from each other. Fig. 7a shows the cross-section of a unidirectional specimen for the circular fiber/epoxy composite. Since the fiber cross-sectional shape is circular, the epoxy resin can easily impregnate the fibers and make the fibers in composites have less contact areas with each other. However, for the kidney fiber/epoxy composites, the fibers have the preferred alignment because of the fiber rotation during fabrication of the prepreg and composite laminates. In the processes of making prepreg and curing composites, the pressure is applied perpendicularly onto the plane of prepreg or laminates, and the fibers in the prepreg or laminates will rotate in the liquid resin to some extent due to the resin transverse flow, so that the long axis of the fiber cross-section tends to be perpendicular to the direction of the applied pressure. As a result, there are some fibers overlapped with each other with large contact areas as shown in Fig. 7b. In such areas the matrix resin is too thin to play the role for stress transfer, so that these areas become weak bonding places. Gaps between fibers may exist when fibers are overlapped since the cross-section of the fibers is kidney-shaped and there are two parallel groves on the fiber surface along the fiber axial direction.

Therefore, the mechanical properties of the kidney fiber/epoxy composites have a larger coefficient of variation than those of the circular fiber/epoxy composites.

4. Conclusion

The nitrogen adsorption characteristic of kidney fibers was found to be better than that of circular fibers. Between the two types of fibers, kidney fibers show the higher impregnating performance. These results can be explained by fact that the specific surface area of kidney fibers is larger than that of circular fibers.

IFSS, ILSS and flexural strength tests have indicated that the kidney composites perform significantly (23.5, 12.7 and 7.5%) better than those manufactured using circular fibers, respectively. Similarly, under compressive load the kidney composites offer a performance advantage of approximately 4%, although this needs further verification to be conclusive. The failure of kidney fiber/epoxy tensile specimens undergoes a cumulative damage progression process and is different from that of circular fiber/epoxy tensile specimens, although the tensile strength is nearly the same for the two composite systems. These results are probably due to the basic properties of kidney fiber which can improve interfacial binding forces and widen interfacial contact area between reinforcement and matrix, resulting in effectively transferring the applied stress. The mechanical properties of kidney composites have a larger coefficient of variation compared with those of circular composites, which can be explained by fact that the kidney fibers in composites have a more non-uniform distribution and larger contact areas with each other than the circular fibers.

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