Tensile Strength and Its Variation of PAN-Based Carbon Fibers. III. Weak-Link Analysis

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ABSTRACT: Both the strength and its variance of carbon fibers depend on the worst flaw that exists in the fiber, or more exactly speaking, on the structure of the ''fiber weak link'' (FWL). To better understand the strength–structure relationship, the fracture-ends morphologies were examined by the scanning electron microscope (SEM). The weak links of carbon fibers were divided into three groups according to its tensile strength, and the effect of the carbon FWLs on the strength variance was also discussed. The observation by SEM, the analysis on fiber tensile properties, and the corresponding discussion of the two sorts of results indicate that both surface flaw and the incompact structure decrease the strength of carbon fiber and enlarge the strength variance of carbon fiber. The modulus seems to influence the strength of carbon fibers too. It is also confirmed that not only the size of the fracture mirror but also the ratio of the size of the mirror to the fracture surface area (not cross section area) is important for judging the strength of brittle fibers. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 3778–3784, 2008

Key words: carbon fibers; fracture; SEM

INTRODUCTION

It is well known that the strength of carbon fibers is gauge-length dependent.[1] This is called ''size effect''[2] or ''weak-link effect''[3], and it is the consequence of the weak link's existence. Peirce put forward that the fractures in tensile generally occur at the weakest part of a fiber, i.e., fiber weak links (FWLs).[4] Spencer and Smith thought that the weak link is not a point, but a certain length of a fiber. Therefore, it is the structure of fiber weak link that determines its physical properties.[5]

Investigations have been done on the fiber weak link of carbon fibers. It was found that the flaws, such as holes, cracks, impurities, and misoriented crystallites decrease tensile strength of carbon fibers, and they are the main cause of the FWLs.[6] By observing the fracture surface by SEM and examining the fracture ends by TEM, Johnson demonstrated that flaws in carbon fibers are not all equally effective as strength reducing factors.[7] Sharp and Burnay were the first to point out that the defect size may not be the determining factor in the tensile failure and sometimes tensile failure does not necessarily occur at the largest neighboring flaw.[8]

Brittle fibers show characteristic fracture markings after failure, and so the nature of flaws influencing the single fiber strength can be determined by characterizing the fracture surfaces in the scanning electron microscope (SEM).[9] The present work attempts to demonstrate the effect of FWLs on the tensile strength of carbon fibers by relating SEM morphologies of the FWL to tensile strength of carbon fibers.

EXPERIMENTAL

Materials

The sample is the same with the carbon fibers mentioned in the Part I of this paper, which were supplied by Shanghai Carbon Co. Ltd, and of 7.0 μm average diameter in tows of 12,000 filaments.

Tensile test

Tensile strengths were measured using an XQ-I tensile test machine, which was equipped with a 1*N* load cell. Tensile strength of single filaments was measured at gauge length of 40 mm in the stretching speed of 5 mm/min. Because of the recoil in the tensile test of carbon fibers, each half of the fractured fiber will experience compressive stress. If the stress exceeds the critical recoil compressive strength of carbon fiber, compressive failure occurs, and there is no actual tensile failure ends left for the surface morphology observation. To solve this problem, one more step that

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TABLE I
Effect of Test Condition on Rate of Reserved Tips

Test condition	Number	Fracture end reserved	Rate of reserved ends (%)
Without grease			
1 mm/min	50	6	12
5 mm/min	321	30	9.4
With grease			
5 mm/min	402	267	66.8

was not only mentioned but also included in the tensile test process in Part I was conducted as follows: before the paper card frame was cut into halves for tensile test, the fiber specimen was coated with water-soluble grease to increase the retention rate (see Table I) of the actual fracture-ends *t* for subsequent SEM examination.

Each broken carbon fiber was labeled to relate the fracture morphology to the corresponding tensile properties of carbon fiber.

SEM observation

The fiber fracture ends were examined by using the SEM (JSM-5600LV Scanning Electron Microscopy).

RESULTS AND DISCUSSION

The SEM micrographs of single fiber fractured ends, which have been carefully selected from a very large number of photographs, were presented in this section, and they represent, in our opinion, the more frequently occurring types of fracture in the case of single carbon fiber.

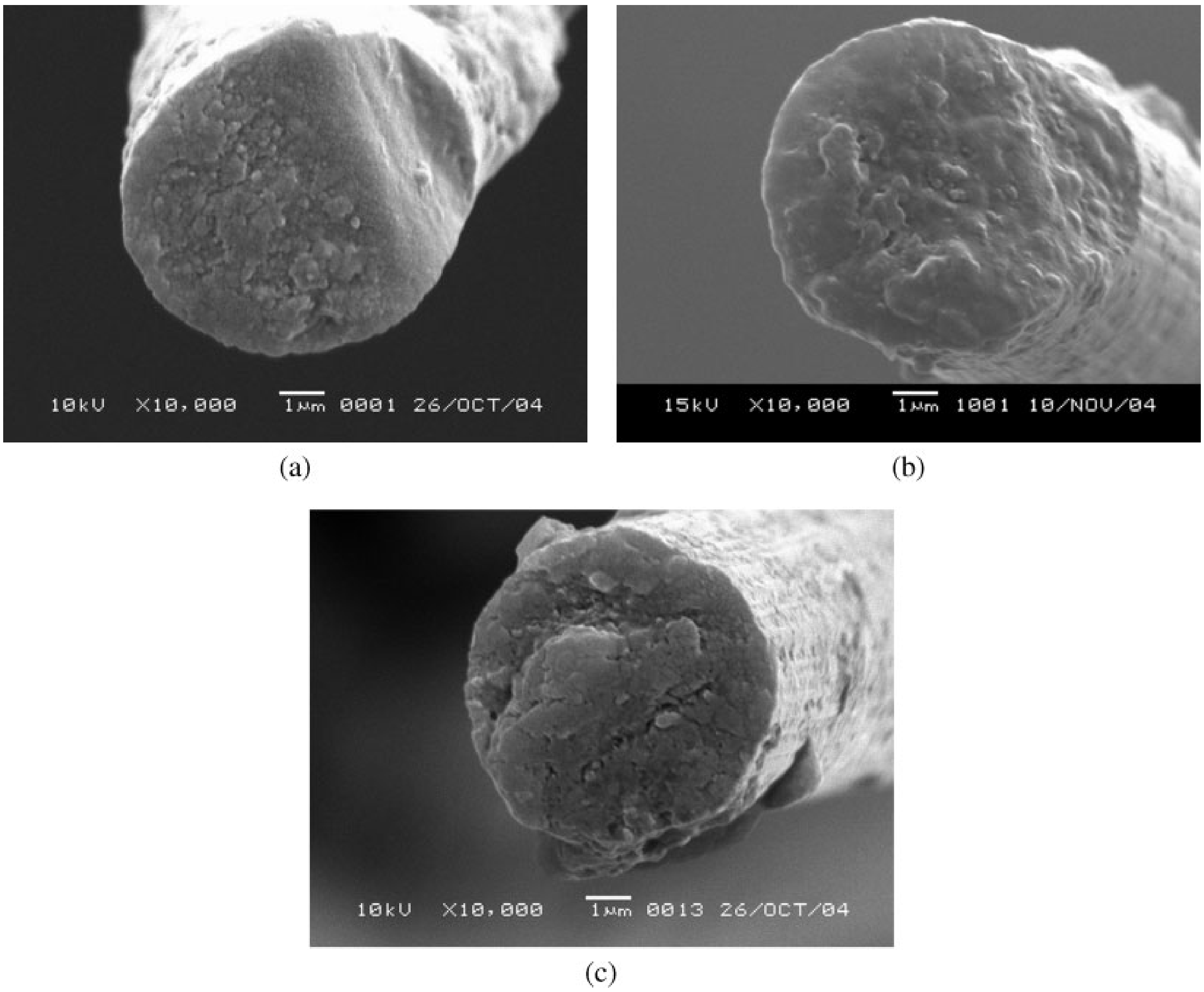

Figure 1 Fracture surface of high-tenacity carbon fiber (a–c).

TABLE II
Tested Tensile Strength of High-Tenacity Carbon Fiber

Figure 1	Diameter (μm)	Fractured force (cN)	Fractured stress (GPa)	Fractured strain (%)	Modulus (GPa)
(a)	7.54	21.10	4.72	1.28	370.85
(b)	6.62	13.69	3.98	0.98	406.92
(c)	7.23	16.63	4.05	1.52	264.78

Fracture-end retention

The carbon fiber, in general, will break off from the window card during fracture process. It is well known that carbon fibers are brittle and difficult to be measured in single fibers, saying nothing of retaining the broken ends in tensile actually. However, most of the fracture ends were reserved to make the SEM observation of fracture surface possible if the fibers were coated with grease before tensile test.

High-tenacity fiber fracture

The SEM micrographs of the fractured ends of the carbon fibers whose fractured stress were near or beyond 4 GPa are shown in Figure 1. The corresponding strength data of tensile tests are listed in Table II.

As can be seen in Figure 1, the fracture-ends seem to be irregular, some of them are smooth while others are rough. But most of the fracture-ends of high-tenacity carbon fibers are not plane. The curved fracture surfaces indicate that there maybe is resistance in the crack propagation process and the crack propagation changes its growing direction, and so the more dynamic crack development need higher strain energy. Another possibility is that several separate cracks develop, and then these become linked by shear failure between them.

As to the morphology characteristic, the group of carbon fibers had no visual defects in the breaking ends, so that the stress at break is high for each of them. No obvious defect or obvious failure mirror on the tensile fracture surface of high-tenacity carbon fiber indicate that the stress distribution was even, and so the tension applied on the fiber must be very large to cause the stress concentration.

The stress–strain curves of high-tenacity carbon fibers are presented in Figure 2. The representative tensile curve was obtained by averaging all the tensile curves. It can be seen from both Figure 2 and Table II that the modulus of most of the high-tenacity carbon fibers is higher than the average modulus. In fact, the average modulus of the high-tenacity carbon fibers is 299.52 GPa, higher than the average modulus of all 402 samples, 234.92 GPa.

Low-strength fiber fracture

Figure 3 presents the SEM micrographs of the fractured ends of the carbon fibers whose strength were lower than 3 GPa. The corresponding results of the fiber tensile tests are listed in Table III.

Compared with fracture-ends of high-tenacity carbon fiber, the fracture-ends of low-tenacity have their own characteristics different from those fibers with high tenacity, which are described as follows.

The first difference is that most of the fracture-ends of low-tenacity carbon fibers are plane, which means that once the crack is formed, the crack will soon grow without any resistance till the failure of the fiber.

The second difference from those of high-tenacity carbon fibers is that there are obvious fracture mirror and mist hackle. From the location of the fracture mirror, it can be seen that fracture of most low-tenacity carbon fibers originated from the surface of the fiber. Surface flaws such as surface pits, surface imperfections, and crack are found to be more severe and popular stress concentrations.

It can be seen from Figure 4 and Table III that the modulus of most of the low tenacity is lower than the average modulus. The average modulus of the high-tenacity carbon fibers is 198.83 GPa, higher than the average modulus of all 402 samples, 234.92 GPa.

Median-strength fiber fracture

Figure 5 shows several fracture-ends of median-tenacity carbon fiber, and their tensile strength is listed in Table IV. Figures 5(a) and 5(b) looks like the fracture surface of low-tenacity fiber, but the fracture mirror is smaller in size. There is obvious fracture mirror in Figure 5(b), but the hackle is no so clear, and small holes were found on the fracture surface. Because of the fracture mirror, it was confirmed that the fracture was not originated from any of the internal flaws. But the internal flaws still played an important role in the fracture process because of their presence on the fracture surface. It is deduced that the tensile strength is

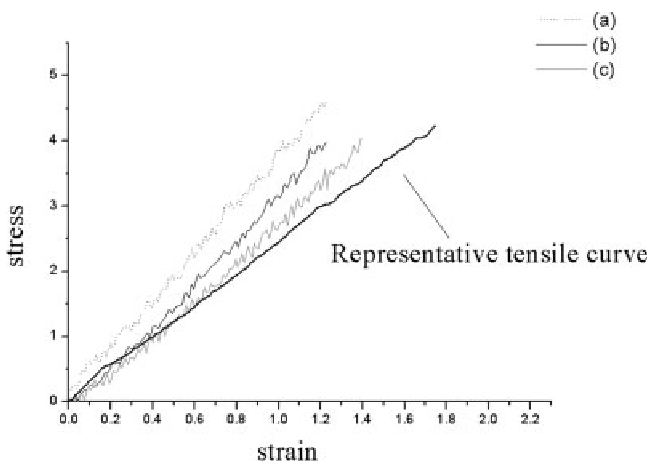

Figure 2 The stress–strain curve of high-tenacity carbon fibers.

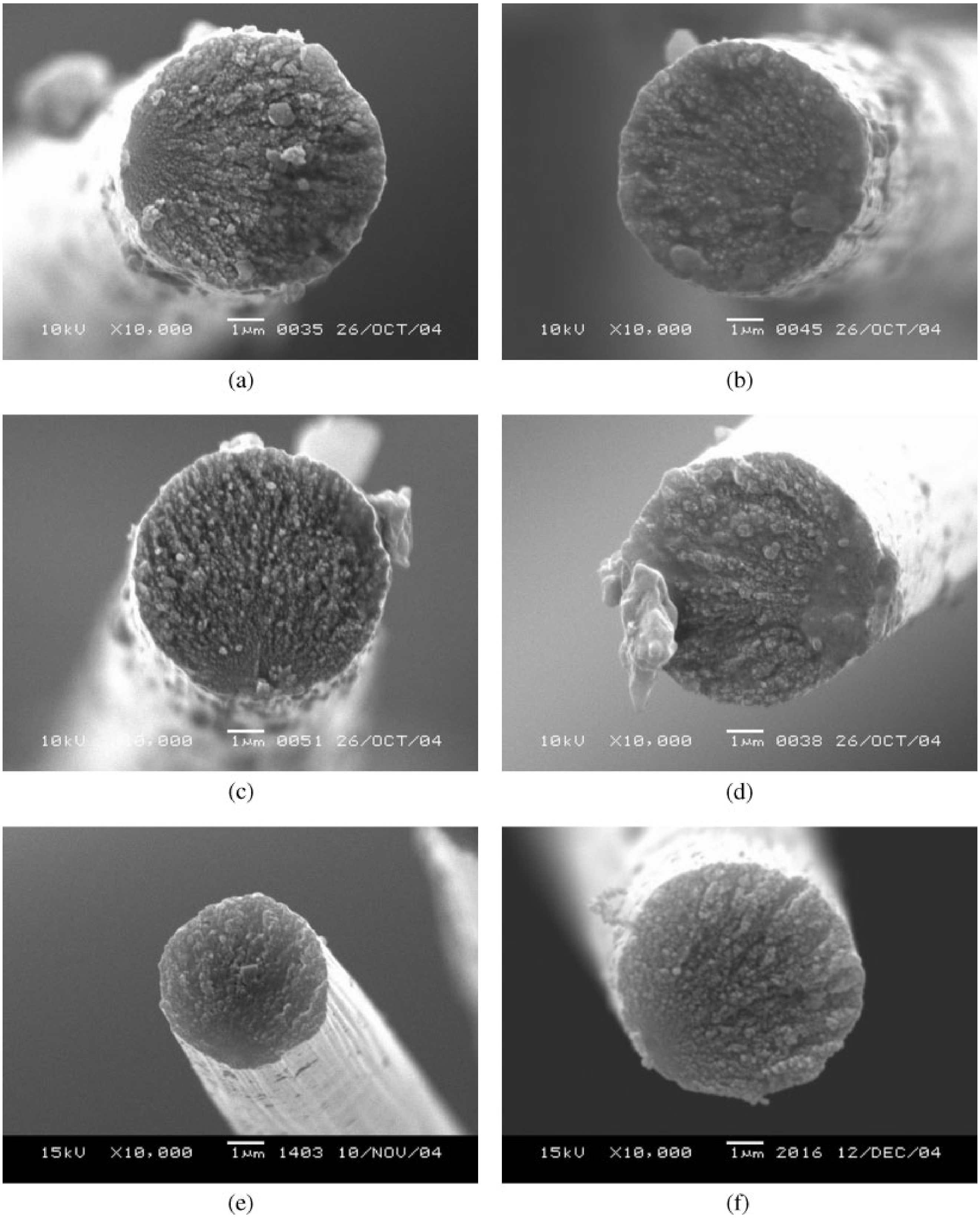

Figure 3 Fracture surface of low-tenacity carbon fiber (a–f).

lower because of the internal flaws, which make it more possible for the stress concentration resulted from the surface crack to exceed the local tensile strength and for fiber to break. Figures 5(c) and 5(d) show that the hackles were interrupted, and the remainder of the fracture surface was uneven then. One

TABLE III
Tested Tensile Strength of Low-Tenacity Carbon Fiber

Figure 3	Diameter (μm)	Fractured force (cN)	Fractured stress (GPa)	Fractured strain (%)	Modulus (GPa)
(a)	6.92	10.96	2.91	2.30	189.15
(b)	6.92	9.90	2.63	1.38	198.98
(c)	6.92	9.41	2.50	1.08	232.33
(d)	7.08	10.92	2.78	1.02	269.02
(e)	6.62	7.45	2.17	1.07	205.02
(f)	6.92	8.96	2.38	1.14	208.58

half of the fracture surface was low-tenacity model while the other half was high-tenacity model. There is a man-made damage on the fracture surface showed in Figure 5(e), the fracture surface was smaller than the cross section area, and so the actual stress was larger than the listed one; there is no fracture mirror on the fracture surface, and so this fiber is of typical high-tenacity fracture. Figure 5(f) is another plane fracture surface and there is no obvious fracture mirror on the fracture surface.

It can be seen from Figure 6 and Table IV that the modulus of most of the median-tenacity is close to the average modulus. The average modulus of the high-tenacity carbon fibers is 244.56 GPa.

Analysis and discussion

The most popular fracture patterns of carbon fibers are presented in Figure 7. Figure 7(a) described the characteristic of fracture morphology of the high-tenacity carbon fibers. Most of the fracture surfaces of the high-tenacity carbon fiber are not plane, which make the fracture surface larger than the cross section area of the fiber. Figure 7(b) represents fracture morphology of the low-tenacity carbon fibers. Almost all fracture surfaces of the low-tenacity carbon fiber are plane, and there are obvious fracture mirror and hackle region on the fracture surfaces. From the location of the fracture mirror, we can see that most fractures of the low-tenacity carbon fibers originated from the surface flaws. Figures 7(c) and 7(d) are the scheme for the fracture surfaces of the median-tenacity carbon fiber. Figure 7(c) looks like low-tenacity carbon fiber, but the size of fracture mirror is much smaller. Figure 7(d) is the scheme for the half low-tenacity and half high-tenacity fracture pattern.

Suppose that the fracture process can be divided into two parts: fracture initiation and crack propagation. For the high-tenacity carbon fibers, there is scarcely obvious fracture initiation, and the crack did not propagate in a plane. There are probably two reasons: the crack propagation changed its direction or several cracks were linked by the shear failure among them. Both of the reasons show the resistance in the crack propagation process. For the low-tenacity carbon fibers, there are always obvious fracture initiations, and once the crack formed, it propagated across the whole fiber without any resistance. For the median-tenacity carbon fibers, either the stress concentration acting as fracture initiation was not large enough or the crack propagation was interrupted by more compact structure of the fiber. The comparison of these three kinds of fracture surface suggested two basic elements of the weak links of low-tenacity carbon fibers: first, there should be flaws or cracks that caused the stress concentration and initiated the fracture, that is the fracture mirror on the fracture surface. Second, the incompact structure of the remainder part of FWLs makes the crack growth at small stress outwards from the fracture mirror.

The fracture origination is usually flaws or cracks, which have been discussed a lot. Less attention is paid on the tensile strength of the rest part of the fiber that is also important because only when the stress concentration exceeds this strength can the failure occur. For example, in Figure 5(b), the fracture occurred at the internal flaws because the local tensile strength is lower than the normal structure.

The modulus of high-tenacity carbon fibers is much higher than that of low-tenacity carbon fiber, which also prove that the structure parameter do have effect on the strength of carbon fibers.

Sometimes fracture surface is curved fracture surface, especially for the high-tenacity fracture. Not only the size of the fracture mirror but also the ratio of the size of the mirror to the fracture surface area (not cross section area) is important. For example, in Figures 5(c) and 5(d), half of the fracture surface is uneven, and so the tensile strength is just median with obvious fracture origination.

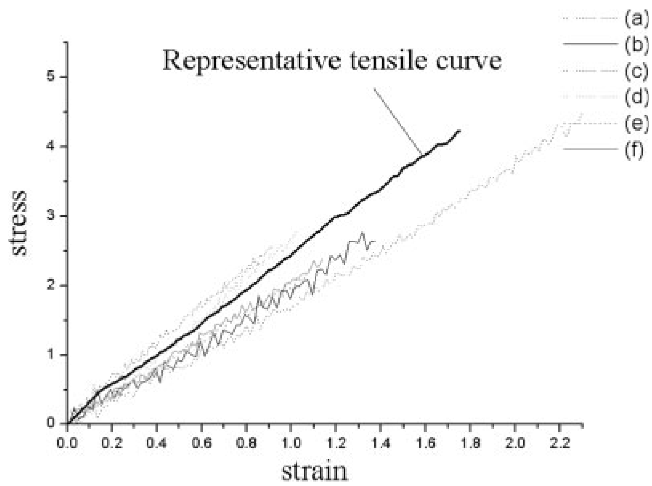

Figure 4 The stress–strain curve of low-tenacity carbon fibers.

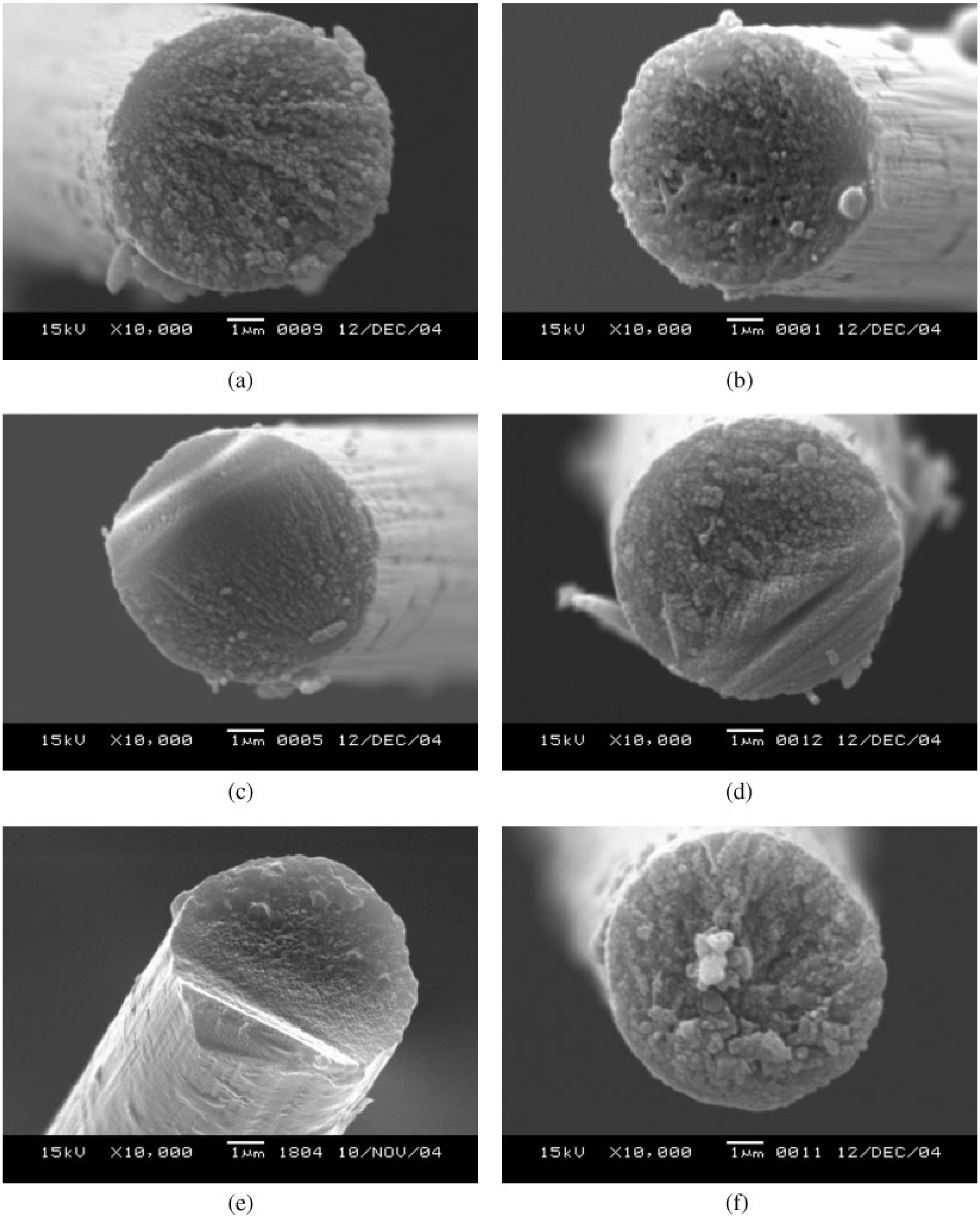

Figure 5 Fracture surface of median-tenacity carbon fiber (a–f).

TABLE IV
Tested Tensile Strength of Median-Tenacity Carbon Fiber

Figure 5	Diameter (μm)	Fractured force (cN)	Fractured stress (GPa)	Fractured strain (%)	Modulus (GPa)
(a)	7.85	15.53	3.21	1.61	197.83
(b)	7.54	14.18	3.18	1.24	253.14
(c)	7.23	15.53	3.78	1.43	264.94
(d)	7.69	15.90	3.42	1.45	234.71
(e)	6.77	11.29	3.14	1.42	221.44
(f)	7.23	13.65	3.33	1.61	204.54

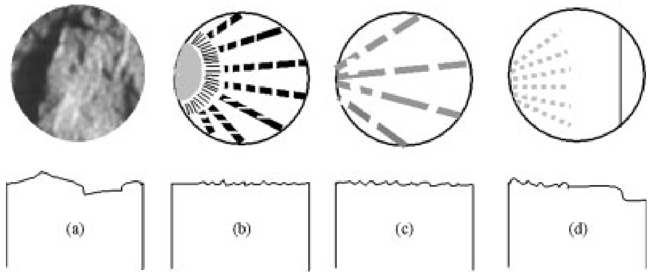

Figure 7 The fracture morphology scheme.

CONCLUSIONS

In a word, the fiber strength depends on both the flaws or cracks on the fiber surface and the inherent structure. In high-tenacity fiber, there is no obvious flaw that initiates catastrophic failure in the fracture surface and the fracture is almost curved surface while in low-strength fiber; a obvious initiation cause failure of the whole fiber, and most of the fracture surface is plane. Most of the stress concentrators that cause the failure of carbon fibers at low strength are surface defects. Surface deposits, surface pits, surface crack are the most popular defects. So the two basic elements of the weak links of low-tenacity carbon fibers are fracture origination and low stress inherent structure. In fact, the inherent structure is also an important factor of tensile strength of carbon fibers.

The modulus seems to influence the strength of carbon fibers too. It seems that the higher the modulus of carbon fiber is, the higher is the strength of carbon fibers.

Because the possibility of the appearance of the rough fracture surface and the diameter variation of carbon fibers, not only the size of the fracture mirror but also the ratio of the size of the mirror to the fracture surface area is important for judging the strength of brittle fibers.

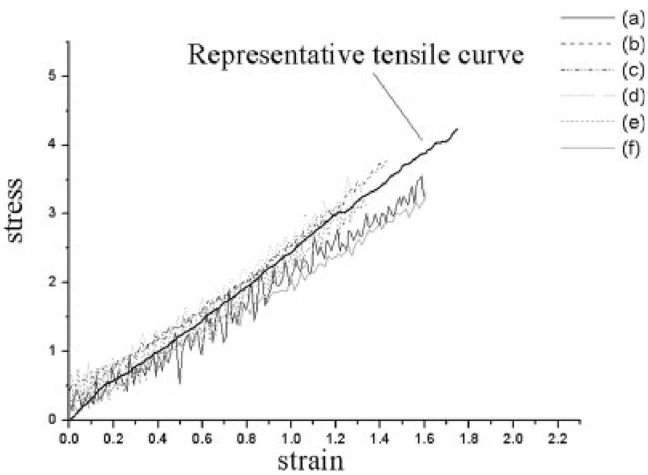

Figure 6 The stress–strain curve of median-tenacity carbon fibers.

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