THE INTERLAYER CRACK EXTENSION MODE IN LAMINATED CARBON/CARBON COMPOSITES

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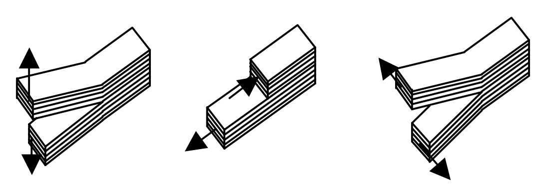
Introduction

Carbon fiber reinforced carbon matrix (C/C) composites possess good mechanical properties and also retain their strength and modulus at high temperature. Therefore, they are primarily developed and designed for high temperature structural applications. In fact, C/C composites retain their strength in an inert atmosphere well above 2000□, where superalloys and even ceramics lose their strength. In addition, C/C composites have low thermal expansion, good wear resistance, and excellent biocompatibility. As a result, applications were also found as refractory materials, brake lining for high-speed vehicles, and biomedical materials, etc. [1,2].

Although the mechanical behavior of C/C composites has been investigated by many researchers [3–9], several problems and questions still remain due to the unique and complicated processing involved in the C/ C composite fabrication. For example, fiber strength utilization in C/ C is still disappointingly low in spite of progressive increasing of the tensile strengths of both pitch-based and PAN-based carbon fibers[3]. Compared with carbon fiber reinforced epoxy matrix composites, which typically utilize 90 to 95% of the fiber strength, only 20 to 50% of the rule-of-mixtures prediction could be obtained in C/C composites. In addition to the strength, equally important for the structural applications is the assurance that the structure can resist the initiation and extension of cracks; this is fracture toughness. The predictability of the fracture behavior is a major concern for the safe design of loadbearing structural parts. However, improper processing or parameters for C/C composite fabrication could lead to low fracture toughness or even catastrophic failure. Interface plays a very important role in the mechanical and fracture behaviors of C/C composites. Especially, delamination is a familiar mode of interface fracture. In order to evaluate the interface fracture toughness, three kinds of interlaminar crack extension modes have been brought forward (Fig.1). In this paper, we only discussed two of them, that is , mode □ and □. On the basis of failure evolution features in composites□ the delamination crack extension energy $G_{\square C}$ □$G_{\square C}$□ the extension patterns and the influencing factors are researched.



mode□ mode □ mode □

Fig.1 Schematic showing of interlaminar failure for 2D composites

Experiments

Specimen preparation

The 3K PAN based carbon fiber tows, after being plain woven, were chosen as reinforcements for C/C composites. The plain weaving fabrics were cross-plied to form rectangular preforms. In one end of the preform, a pre-crack was introduced by laying a piece of paper between the plies. Figure 2 shows the dimension of the preform.

2DC/C composites were fabricated by isothermal chemical vapor infiltration (ICVI) process. During the process, propylene diluted with nitrogen decomposes at the temperature of 800---1300□, leading to the product of pyrolytic carbon.

Mechanical test

(1) $G_{□C}$ test

As shown in Fig.3, the double cantilever beam(DCB) was selected in $G_{□C}$ test. In both $G_{□C}$ and $G_{□C}$ tests, a Hi-scope was used to monitor and record the initiation and extension of interlaminar crack on the side-surface. The side-surface of all specimens were polished and then painted in white before testing. The tests were conducted at room temperature in air, under the loading speed 0.5mm/min.

(2) $G_{□C}$ test

As shown in Fig.4, the end notched flexural (ENF) specimen was chosen in $G_{□C}$ test. The experimental conditions kept the same as G_{c} test.

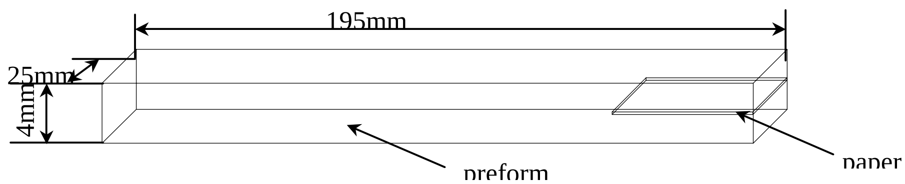


Fig.2 Schematic of the preform

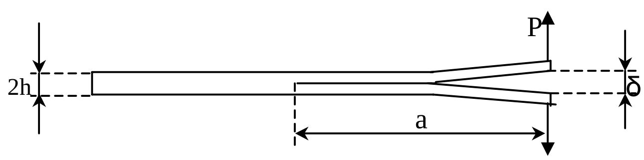


Fig.3 Schematic of DCB apparatus

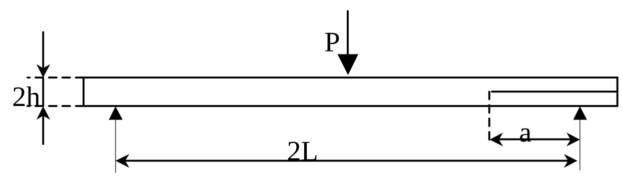


Fig.4 Schematic of ENF apparatus

Results and Discussion

Crack extension in mode delamination

The DCB specimen was adopted to understand the crack extension in mode □ delamination. In the course of the experiment, the load versus displacement curve was plot and the length of crack was measured simultaneously. In general, the compliance of the tested specimen can be calculated as follows.

$$C = \frac{\delta}{P} = ma^n \qquad (1)$$

where δ represents the displacement of crosshead, P the load, a the length of crack, m and n the experimental parameters. The m and n can be calculated according to the logC versus loga curve. The maximal critical crack extension energy

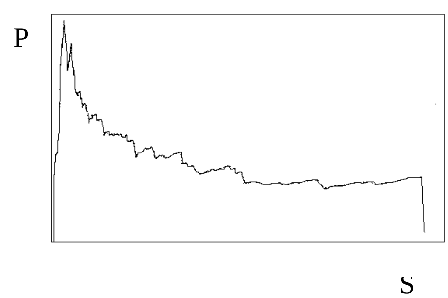


Fig. 5 Load-displacement curve of 2D C/C in DCB process

release rate of mode □ ($G_{□c}$) can be achieved by

$$G_{1C} = \frac{nP_{\max}\delta_{\max}}{2ba} \qquad (2)$$

A typical load versus displacement curve was illustrated in Fig.5.

As shown in Fig.5, the crack propagated intermittently. Initially, as the load increased, the strain energy at the end of the crack built up. The moment the strain energy reached its crest value, the failure occurred, and the crack extended quickly. On the other hand, the strain energy at the end of the crack released at the same high-speed. As a result, the crack stopped propagating after extended a certain distance. In the case of the load kept exerting, the crack extension process mentioned above repeated till the plies separated completely. It is evident that the crack propagation in 2DC/C is an intermittent process. Furthermore, in each stage of crack evolution, the crack extended about 3---15mm. The distance that the crack extended in each period enlarged with the compliance of the specimen increased. Therefore, it can be concluded that, under the condition of $G_{\Box C}$ test, the crack extension characteristic of 2DC/C is periodic and abrupt.

According to Fig.5, the distance of crack extension in each stage was measured. Consequently, the compliance of the specimen in each stage can be attained. Fig.6 illustrates the logarithmic relation between the compliance and the crack extension length in each stage (2D C/C in density of $1.4g/cm^3$). The parameters m and n were gained by linear simulation of the relationship shown in Fig.6. Substituted their values, that is n=2.48 and $m=2.34\times10^{-5}$, to equation (2) and obtained the specimen $G_{\Box C}$ =$33.25J/m^2$. With the same method, $G_{\Box C}$ of 2DC/C in variant densities were achieved and listed in table 1.

Table 1 $G_{\Box C}$ of 2DC/C in variant densities

Density (g/cm3)	1.4	1.48	1.54
$G_{\Box C}(J/m^2)$	31.0	33.25	37.91

In table 1, $G_{\Box C}$ of 2DC/C increases as the density becomes larger. It follows that the crack resistant ability of 2DC/C enhances with the increase of density. According to Marinkovic's work[10], the relationship between the mechanical property and the density of carbon/carbon composite can be described as

$$S = kd^r \qquad (3)$$

where S is the mechanical property of C/C, d is the density of C/C, r is the exponent, and k is the coefficient. Based on equation (3), the logarithmic expression of $G_{\Box C}$ versus density was shown in Fig.7. By linear simulation of the relationship shown in Fig.7, it is achieved that r=2.12, k=1.18, and linear correlation coefficient 0.9344. It is clear that $\log G_{\Box C}$ and logd is in approximate linear relation. The relationship between $G_{\Box C}$ and density of 2DC/C was expressed as follows:

$$G_{IC} = 1.18d^{2.12} \qquad (4)$$

If the fabricating condition alters, the relationship between $G_{\Box C}$ and density of 2DC/C changes accordingly. Despite this, the relationship described above is in conformity to Marinkovic's rule.

Crack extension in mode delamination

End notched flexural□ENF□test is often adopted in the investigation of crack extension in mode □ delamination. Fig.4 is the schematic of ENF apparatus. The compliance of the sample can be calculated by

$$C = \frac{2L^3+3a^3}{8E_{11}bh^3} \qquad (5)$$

While, the critical crack extension energy release rate of mode □ ($G_{\Box C}$) can be calculated by

$$G_{IIC} = \frac{P^2}{2b}\frac{dC}{da} = \frac{9a^2P^2}{16Eb^2h^3} = \frac{9a^2P^2C}{2b(2L^3+3a^3)} \qquad (6)$$

In our work, 4 kinds of samples, including 2DC/C with the fiber volume fraction of 46.6% and 40.7%, 2DC/C filled with pyrolytic carbon (PyC), and 2DC/C filled with graphite, were tested. Also, their $G_{\Box C}$ were calculated respectively. By the way, the densities of these samples are close to $1.7{-}{-}{-}1.72g/cm^3$. The corresponding P-S curves were exhibited in Fig.8. It can be observed that curve (a) is divided into 3 phases. Different to the mode □ crack extension of other 2D C/C samples, which are the typical abrupt extension, the mode □ crack extension of 2D C/C filled with PyC powders shows periodic and abrupt behavior, which is close to the crack extension in mode □

delamination. So, we can say that PyC powders postpone the crack extension in mode □ delamination of 2DC/C. According to equation (5) and (6), compliance and $G_{\text{□}C}$ of the samples were gained and listed in table 2.

As can be seen from the table 2, $G_{\text{□}C}$ goes up with the increase of fiber volume fraction in 2DC/C, and fillers (pyrolytic carbon or graphite) can improve $G_{\text{□}C}$ of 2DC/C. Among all the samples, 2DC/C filled with pyrolytic carbon exhibits the highest $G_{\text{□}C}$, which is expressed clearly in Fig. 8.

In our experiments, the fiber volume fractions in the specimens vary when the carbon fibers alter. Namely, the fiber volume fraction 40.7% is due to the application of 3K carbon fibers, while the value 46.6% attributes to the selection of 1K carbon fibers. Furthermore, not only the different fiber volume fractions result from the changed carbon fiber bundle, but also the varied pore distribution in the samples. Moreover, adding fillers can improve the uniformity of pore distribution. Hence, it is evident that either increasing fiber volume fractions or adding fillers can improve the uniformity of pore distribution in C/C samples. It is just the improvement of the pore distribution uniformity that makes the number of macropores decreased. As a result, the crack extension energy release rate speeds. It must be pointed out that no matter what carbon fiber bundles are chosen, adding fillers can improve the crack extension energy release rate efficiently because of the better pore distribution in C/C. In the 2 kinds of fillers selected, the PyC powder is more similar to the PyC matrix in crystal and molecular structures than graphite, so its interfaces with PyC matrix are stronger. In consequence, 2DC/C filled with PyC displays the highest crack extension energy release rate.

Table 2 Compliance and $G_{\text{□}C}$ of the tested samples

Sample	Compliance C□μm/N□	$G_{\text{□}C}$□J/m^2□
2DC/C with the fiber volume fraction of 46.6%	2.05	189.6
2DC/C filled with pyrolytic carbon	2.04	349.1
2DC/C filled with graphite.	1.85	280.9
2DC/C with the fiber volume fraction of 40.7%	1.37	101.5

Conclusions

(1) As for mode I delamination evolution, $G_{\text{□}C}$ of 2DC/C rises with the density increase. In addition, the relationship between mechanical property and density of the selected specimens accords with Marinkovic's rule.

(2) Adding fillers can improve $G_{\text{□}C}$ and interlaminar bond strength of 2DC/C. In the 2 kinds of fillers used, PyC has a higher efficiency. Besides, in the case of filling PyC, the crack extension mode changes from abrupt to intermittent.

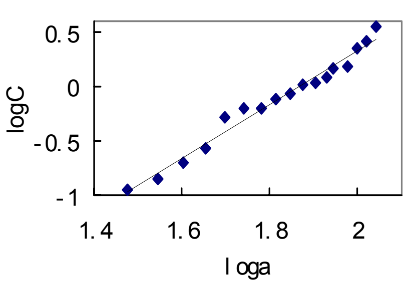


Fig.6 Compliance versus crack length for 2D C/C in DCB process

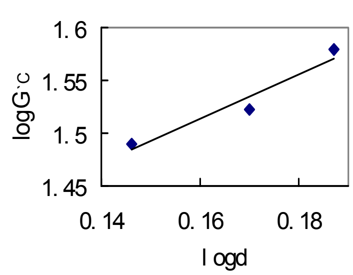


Fig.7 $G_{\text{□}c}$ versus bulk density for 2D C/C

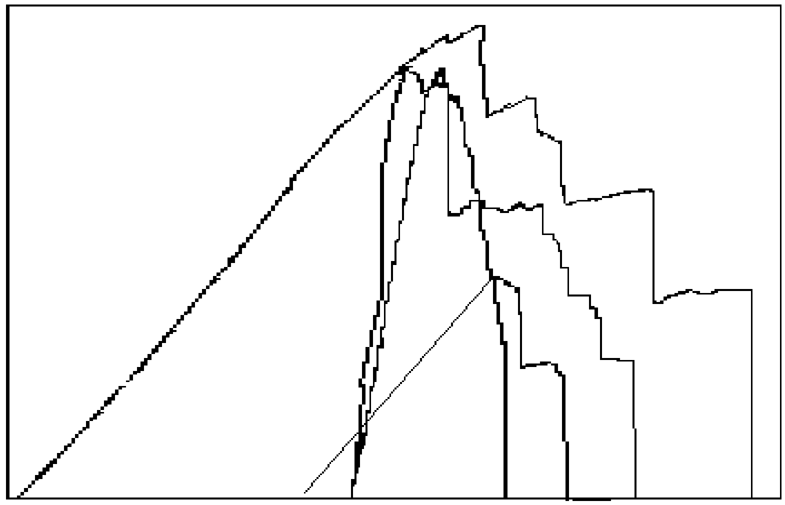


Fig.8 Load versus displacement curves of various 2D C/C in ENF process, (a)---2D C/C filled with PyC, (b)--- 2D C/C filled with graphite, (c)---2D C/C with fiber volume fraction of 40.7%, (d)---2D C/C with fiber volume fraction of 46.6%

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