Effect of stress level on fatigue behavior of 2D C/C composites

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Abstract: Laminated carbon fiber clothes were infiltrated to prepare carbon fiber reinforced pyrolytic carbon (C/C) using isothermal chemical vapor infiltration (CVI). The bending fatigue behavior of the infiltrated C/C composites was tested under two different stress levels. The residual strength and modulus of all fatigued samples were tested to investigate the effect of maximum stress level on fatigue behavior of C/C composites. The microstructure and damage mechanism were also investigated. The results showed that the residual strength and modulus of fatigued samples were improved. High stress level is more effective to increase the modulus. And for the increase of flexural strength, high stress level is more effective only in low cycles. The fatigue loading weakens the bonding between the matrix and fiber, and then affects the damage propagation pathway, and increases the energy consumption. So the properties of C/C composites are improved.
Key words: C/C composites; fatigue behavior; stress level; residual strength

1 Introduction

Carbon/carbon (C/C) composites are the new ultra-high-temperature materials. They combine the outstanding high-temperature properties of carbon materials and the excellent mechanical properties of fiber reinforced composites [1], such as low density, high specific strength and modulus, low coefficient of thermal expansion, high temperature resistant, good thermal shock resistance [2−4]. These superior thermal and mechanical properties make C/C composites an attractive candidate for using as high temperature structural components (e.g., rocket nozzles, aircraft brakes, nose cones and wing leading edges of hypersonic aircraft) [5,6]. However, these working environments deal with not only high temperature, but also vibration of aerodynamic and mechanical sources, which causes the fatigue loads. So a major and often fatal problem for C/C composites is the high and low temperature fatigue performance under long time working conditions [7,8].

The previous studies [9−13] showed that the fatigue performance of C/C composites was superior to that of metal materials, with a fatigue limit 80%−90% of static strength. GOTO et al [9] reported that the fatigue limits of three types of C/C composites differed from 85% to 92% of the static tensile strength. LIAO et al [10] found that the flexural fatigue limit of three-dimensional integral braided C/C composites was 92% of the static flexural strength. They also observed that the flexural strength of C/C composites was improved by 40% after 10^5 cycles, and then decreased [11]. It was explained that cyclic loading weakened the interface bonding strengthening, leading to the decrease of residual thermal stress, which was the reason of strengthening. And some other studies also reported the fatigue enhancement, that is, the residual strength was not decreased but increased after cyclic loading [12−14]. TANABE et al [15] reported that the fatigue degradation occurred in both bending and shear modes. And the fracture mechanisms were concluded to be a combination of wedge effect and stress corrosion with medium.

However, the fatigue behavior of C/C composites is related not only to the material itself, but also to the testing conditions, such as loading frequency, stress ratio, stress level. Therefore, it is necessary to do a lot of systematic and further research in order to grasp the characteristics of fatigue enhancement.

In the present work, the effect of maximum stress level on the mechanical behavior of 2D carbon cloth laminated C/C composites was investigated. The samples were subjected to cyclic loading with two different maximum stress levels, and then statically loaded until fracture. The mechanical properties and microstructure were mainly discussed.

2 Experimental

2.1 Specimen preparation

Two-dimensional plain woven carbon fiber clothes selected in this study were cross ply stacked and punctured vertically as the reinforcements for 2D C/C laminates. These clothes were plain woven using 1K PAN-based T300 carbon fiber tows provided by Jilin Carbon Ltd., Co., and the diameter of the fibers was 6−8 μm. The examined C/C composites were fabricated via chemical vapor infiltration (CVI) process at temperature from 900 to 1200 °C under negative pressure, using highly pure gaseous propylene (C_3H_6) as the precursor and nitrogen (N_2) as the carrier gas. Several densification cycles were carried out to obtain the desired density (approximately (1.73 ± 0.03) g/cm^3 in this study), involving carbonization in an inert atmosphere and re-deposition by CVD process. Finally, the resultant product was graphitized at 2300 °C. The samples were prepared by cutting the laminated composites panel into small rectangular bars, and the nominal dimensions were 52 mm×6 mm×3 mm.

2.2 Mechanical test

The infiltrated samples were tested under 3 stages, including static, cyclic and static three-point bending tests. Firstly, the static flexural strength of the samples was examined. And then, according to the static strength and the selected stress levels (The stress level was defined as the percentage of maximum applied stress to the static flexural strength. It was set to 0.8 and 0.9 in this study), the samples were loaded under cyclic loading for various cycles. At last, the fatigued samples were statically loaded to fracture in order to test the residual flexural strength and modulus. Both the static mechanical tests and the cyclic loading tests were carried out on Instron 8871 servo hydraulic testing machine under ambient laboratory conditions, with the loading direction vertical to the carbon fabric surfaces and the fixture dimension of upper indenter radius 3mm, bearing radius 2 mm, and span length 40 mm. The static tests were conducted with a constant loading speed of 1 mm/min, while the load-controlled fatigue tests were conducted in bending mode with a frequency of 10 Hz (sinusoidal waveform) at a constant stress ratio R (the ratio between the minimum stress to the maximum stress in the wave form) fixed at 0.1.

2.3 Microstructure analysis

Scanning electron microscopy (SEM) (JSM-5610LV) was used to examine the fracture surfaces and microstructure of C/C composites.

3 Results and discussion

3.1 Conventional static testing

The actual photographs of the samples before and after three-point bend loading is shown in Fig. 1. Figure 1(a) shows the original sample, Fig. 1(b) is the side view of fractured sample, and Fig. 1(c) shows the fractured sample whose layers separate completely. From these photographs, it can be clearly observed that the main failure mode of 2D C/C composites in bending is the matrix pyrolytic carbon delamination induced by interlaminar shear stress. Besides the fiber bundles punctured vertically in the cloth are not dense enough, this phenomenon mainly implies the interfacial bonding strength of the composites is not high enough. Delamination failure occurred in the matrix when the composites were subjected to bend loading, and the damage extended along the matrix carbon layer until two layers separated completely.
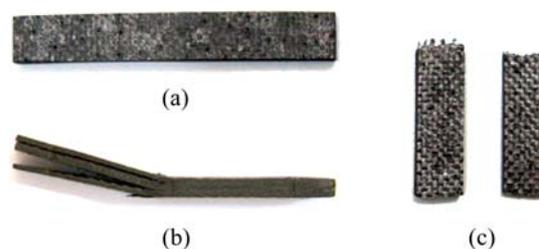

Fig. 1 Photographs of samples before (a) and after (b, c) flexural loading

3.2 Dynamic-static testing

The fatigue tests were stopped after 10^4, 10^5, and 10^6 cycles, respectively. And the surviving samples were destroyed to obtain residual strength and modulus. The results are shown in Figs. 2 and 3. It can be seen obviously that, the residual flexural strength of the fatigued samples is much higher than that of the original samples after being loaded under either 0.8 or 0.9 stress level.

The residual strength of the fatigued samples is increased after cyclic loading and would increase continuously with the increase of fatigue loading cycles. Comparing the results of low cycle fatigue, it can be observed that the strengthening effect with stress level of 0.9 is superior to that with stress level of 0.8. The residual strength of the samples at 10^4 fatigue cycles is increased by 25% with stress level of 0.8, while 35% with stress level of 0.9. The same result is also obtained at 10^5 fatigue cycles. However, the result changes at 10^6

cycles. The residual strength with stress level of 0.9 is lower than that with stress level of 0.8. In this study, the maximum increment of flexural strength is about 51% of the original strength. All the phenomena above show that 2D carbon cloth laminate C/C composites have an excellent fatigue resistance.
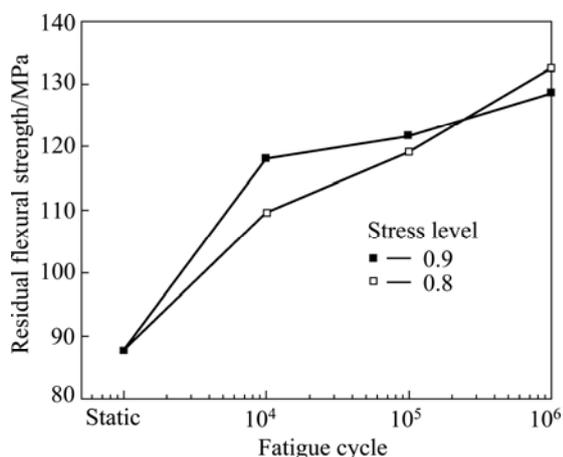
Fig. 2 Residual flexural strength at different fatigue cycles with different applied stress levels of C/C composites
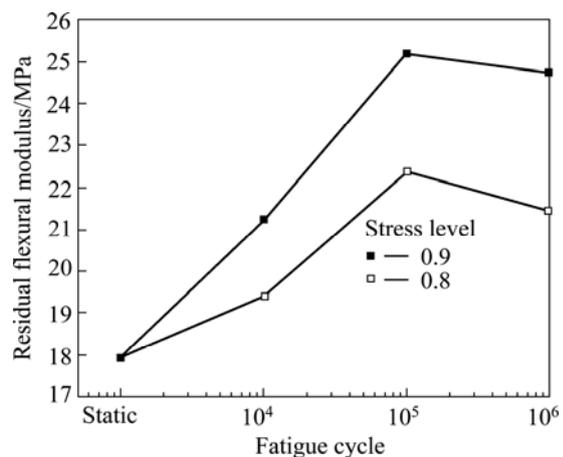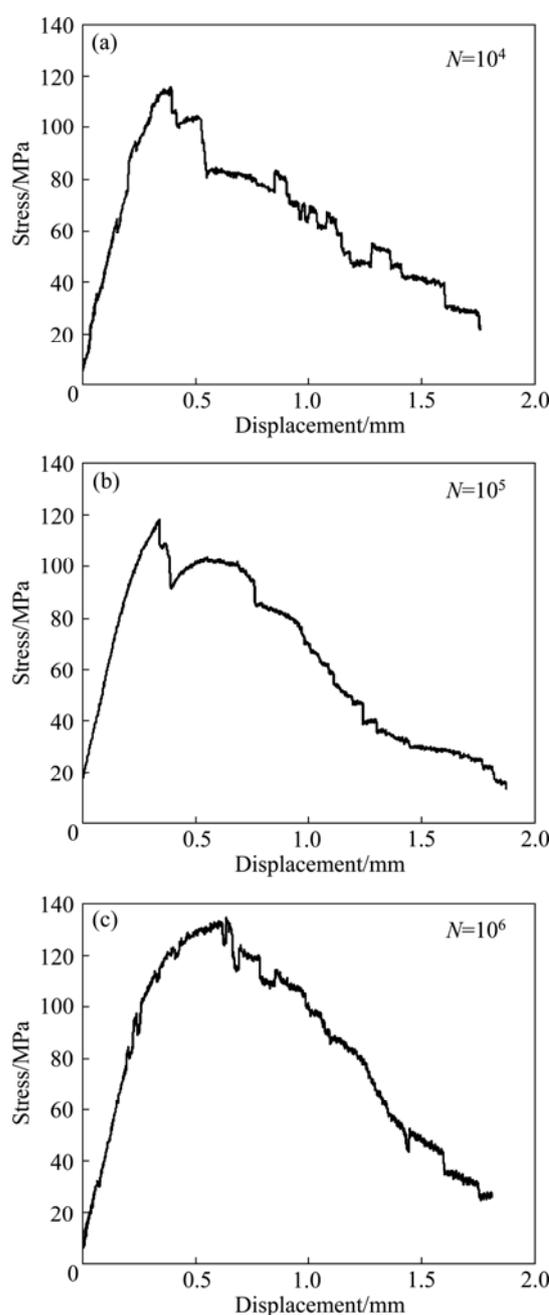

Fig. 3 Flexural modulus at different fatigue cycles with different applied stress levels of C/C composites

Figure 3 shows the flexural modulus at different fatigue cycles with two different applied stresses. It can be seen obviously that the whole development trends of the two curves are similar, and the residual flexural modulus is much higher than the static flexural modulus. The modulus increases continuously within 10^5 cycles, but decreases with the increase of fatigue cycles to 10^6. This is different from the results of strength. So the improvement in modulus by cyclic loading can be considered to be limited. That is to say, the modulus will increase by virtue of cyclic loading, but decrease beyond a certain limit. The maximum increase amplitude of residual modulus is about 20% of the original modulus with stress level of 0.8, while 38% with stress level of 0.9. The flexural modulus under stress level of 0.9 is

higher than that under stress level of 0.8 after the same fatigue cycles. This implies that high stress level is more effective for the increase of flexural modulus.

3.3 Stress—displacement relation

Figure 4 shows the flexural stress—displacement curves of surviving samples at various fatigue cycles with stress level of 0.9. The bear load capacity of the original sample decreases rapidly as soon as beyond the limit load, implying that the crack growth is rapid after the first fracture and causes the overall failure directly. That is a typical brittle fracture. In Fig. 4, it is observed that the stress of the fatigued sample after 10^4 cycles has


Fig. 4 Flexural stress—displacement curves of C/C composites at different fatigue cycles

a stepped decrease with the increase of displacement. This phenomenon shows the crack extends slowly and involves deflection, which causes much more energy is needed to the propagation of damage crack. So the carrying capacity of composites improves. The curve of fatigued sample after 10^5 cycles shows that the sample keeps high load−carrying capacity after failure. And the sample after 10^6 cycles could bear high stress in a long time. By the observation of the whole fatigue tests of C/C composites, the cyclic loading gets the mode of fracture evolving from brittle to pseudo-plastic mode.

3.4 Composites microstructure

C/C composites consist of carbon fiber reinforcement and carbon matrix. The fracture mode is related to the strength retention of fiber and the interface bonding between the fiber and the matrix. Figure 5 presents the flexural fracture morphology of C/C composites after 10^4 fatigue cycles at stress level of 0.8. It can be seen that the fracture section is smooth, which exhibits typical brittle fracture morphology. The carbon fibers fractured are still tightly wrapped in the matrix, indicating that the bonding between the carbon fibers and the matrix is strong. The strong interface causes that the stress can not be relaxed in the interface, and some of the micro-crack tips perpendicular to the fibers may be a direct impact on the carbon fibers. By the external load, the stress can not be relaxed if there is no interface debonding between the fibers and matrix. When the energy concentrated at the crack tip is large enough, the
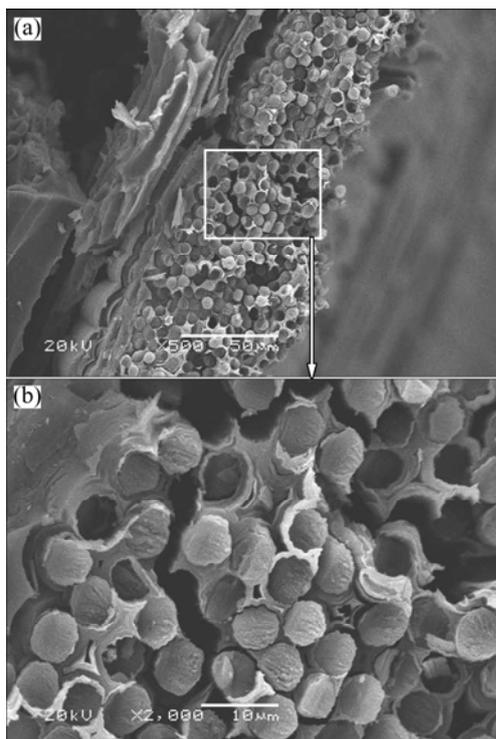
crack could go through the carbon fibers and continue expanding, causing the chain-destruction of the fibers around, showing a similar brittle failure mode. It also can be inferred that, the stronger the interface bonding between carbon fiber and the matrix is, the more brittle the material is.

For comparison, the fracture morphology of composite after 10^6 fatigue cycles is presented in Fig. 6. It can be seen that the fibers are pulled-out, which indicates that the fiber bundle/matrix interface and the fiber/matrix interface are weakened under fatigue loading.
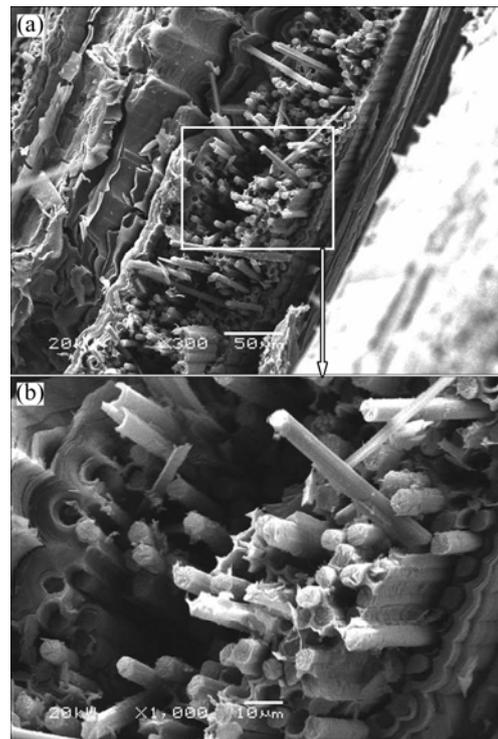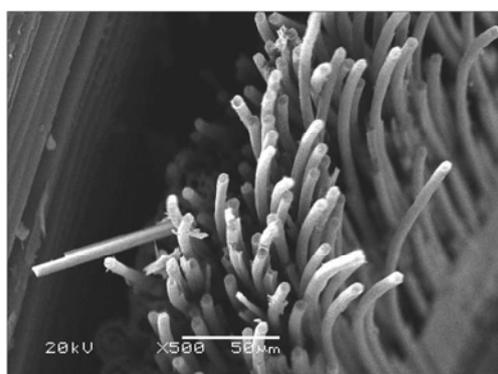

Fig. 6 Flexural fracture pattern of C/C composites after 10^6 cycles at applied stress of 0.8

Figure 7 shows the fracture morphology of sample after 10^4 cycles at stress level of 0.9 in flexural mode. Although there are some relatively flat sections, the phenomenon of carbon fibers debonding and pulling-out is more significant, compared with that of the sample after the same cycles at applied stress of 0.8. Because the pulling-out of carbon fibers will absorb energy, the damage process manifests as intermittent and periodic one. And the fibers debonding and pulling-out can consume some failure energy caused by external load, which can explain the phenomenon that after 10^4 cycles, the residual flexural strength at stress level of 0.9 is higher than that at stress level of 0.8.

Figure 8 presents the fracture morphology of sample after 10^6 cycles at stress level of 0.9. Plenty of carbon fibers and fiber bundles are pulled out. All the fibers bend in one direction and basically break in the place

Fig. 5 Flexural fracture pattern of C/C composites after 10^4 cycles at applied stress of 0.8
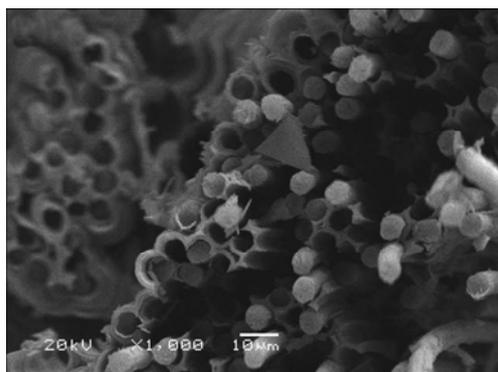
Fig. 7 Flexural fracture pattern of C/C composites after 10^4 cycles at applied stress of 0.9
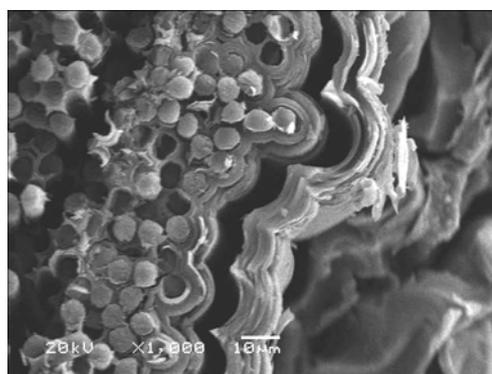

Fig. 8 Flexural fracture pattern of C/C composites after 10^6 cycles at applied stress of 0.9

with a maximum deformation. It indicates that the fibers break factually under tensile effect of external load, producing an enhancement effect on the composites. It is precisely because the role of carbon fiber bearing, the applied load does not decline rapidly. So the composites show higher fracture energy. This can also be demonstrated from the flexural stress一load curve shown in Fig. 4. So it can be concluded that, the proper interfacial bonding and high strength retention of fibers will cause fibers to give full play of the enhanced role, and the composites will exhibit better mechanical properties.

The fracture of composite materials is essentially the matrix crack propagation process. And the propagation pathway is related to the interfacial bonding. From the SEM images we can observe that there is not only the pulling-out of fiber bundles, but also the pulling-out of fibers in fiber bundles. It indicates that the fiber bundle/matrix interface and the fiber/matrix interface are weakened under fatigue loading. The crack along interface will cause the debonding between the matrix and fiber; meanwhile the debonding will lead to the relative sliding between the matrix and fiber. The sliding will absorb a considerable portion of energy, which can delay the fracture, playing a toughening role.

As shown in Fig. 9, the matrix carbon layer between the carbon cloth laminations separates from the matrix carbon layer which wraps the carbon fiber tightly. Such a separation of matrix carbon layers will adversely affect the properties of composites. However, the experimental results show that the properties of the fatigued samples are more improved compared with the original samples, which means that the change of material internal structure by fatigue loading will enhance the carrying capacity eventually. These two aspects are contradictory. The gap can only be compensated by the reinforcement of C/C composites (carbon fibers). The strengthening effect of carbon fibers is far greater than the adverse effect of separation between the carbon layers.

Fig. 9 Fracture pattern of C/C composites after fatigue loading

In Fig. 9 we also observed that the deposition of pyrolytic carbon is typical layered deposition. The matrix carbon layer separation indicates that the interfacial bonding of matrix pyrolytic carbon is weak. Moreover, the bonding strength is varied. Close to the fibers, the interfacial bonding between matrix and fiber is strong, while weak in the outer layer.

4 Conclusions

1) 2D carbon cloth laminate C/C composites have excellent fatigue resistance.

2) The mechanical properties of the fatigued samples with both stress levels are significantly improved compared to the original samples. The strength increases continuously with the increase of fatigue loading cycles, while the modulus increases within 10^5 cycles and then decreases, showing the improvement is limited.

3) High stress level is more effective to increase the modulus. And for the increase of flexural strength, high stress level is more effective only in low cycles.

4) The fatigue loading changes the interface state, weakens the bonding between the matrix and the fiber, and then affects the damage propagation pathway, and increases the energy consumption. So the properties of

C/C composites are improved.

5) The deposition of pyrolytic carbon is typical layered deposition, and the interfacial bonding strength is varied in C/C composites.

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应力水平对二维炭/炭复合材料疲劳行为的影响

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摘 要：采用等温化学气相浸渗法制备二维炭布叠层炭/炭复合材料。进行了两种应力水平下的弯曲疲劳测试，测定剩余弯曲强度及模量，以表征不同应力水平对炭/炭复合材料疲劳行为的作用。结果表明：疲劳加载后试样的剩余弯曲强度及模量均有提高，在高应力水平下剩余弯曲模量的增强作用更显著，但在疲劳循环超过 10^5 次后，低应力水平下的剩余强度更高。分析了炭/炭复合材料的疲劳强化与损伤机理。疲劳加载会弱化基体与纤维之间的结合，改变损伤扩展路径，从而改善炭/炭复合材料的性能。

关键词：炭/炭复合材料；疲劳行为；应力水平；剩余强度

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