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Original Article

Influence of boron nitride nanotubes on the damage evolution of SiC_f/SiC composites

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A B S T R A C T

As-grown and BN-coated boron nitride nanotubes (BNNTs) were incorporated into SiC_f/SiC composites to produce nanotube-based hierarchical composites. In-depth studies on damage evolution reveal that early damage development are delayed owing to the restriction effects on crack propagations from as-grown and BN-coated BNNTs. Moreover, this delay effect is more pronounced from BN-coated BNNTs because BN-coated BNNTs/matrix interfacial bonding strength is low. Final failure of composites with as-grown BNNTs still comes much earlier compared with virgin composite due to strong fibers/matrix bonding enhanced by as-grown BNNTs. This premature final failure is remedied in large part in composites with BN-coated BNNTs because fibers/matrix bonding enhanced by as-grown BNNTs is weaken after the deposition of an interphase on nanotube surface. Additionally, the type, the number and the released energy level of damage mechanisms during the whole damage evolution after the incorporation of as-grown and BN-coated BNNTs were also discussed elaborately compared with virgin composite.

1. Introduction

Conventional silicon carbide (SiC) fiber reinforced SiC matrix (SiC_f/SiC) composites have exerted a significant impact on the aerospace industry over the past several decades. By virtue of their outstanding properties, such as low weight, good high-temperature strength, excellent thermal, chemical and environmental stability, they have been considered as the most promising candidate for structural applications in hot section aero-engine components [1–3]. The incorporation of SiC fibers and a proper design of fibers/matrix interface via depositing an interphase on fiber surface enable improved fracture toughness and damage tolerance of the macroscopic composite. It is attributed to matrix crack arresting and deflection occurring at fibers/matrix interface [3]. Nevertheless, poor damage resistance of matrix at micron scale among fibers remains a major challenge to further optimize the matrix dominated properties of SiC_f/SiC composites. On one hand, the threshold stress or strain for damage initiation of matrix is rather low. It will lead to the premature damage initiation of matrix under stress, at last deteriorating the fatigue limit and service lifetime of composites. On the other hand, cracks in matrix at micron scale among fibers propagate easily without being hindered, thus inducing the premature failure of matrix and eventually the composite. This issue puts a restriction in some extent on further application of SiC_f/SiC composites where enduring service is required emphatically.

Recently, nanotube-based hierarchical composites consisting of nano- and micro-scale reinforcements have drawn tremendous attention from composites science community, which offers a promising approach to solve above-mentioned problem. It is anticipated that by introducing nanoscale reinforcements such as carbon nanotubes (CNTs) with extraordinary mechanical properties into matrix, toughening in matrix at micron scale among fibers can be gained and damage resistance or tolerance of the local matrix can be improved thanks to crack arresting and deflection from nanotubes. Consequently, the existing limitations associated with the matrix dominated properties can be alleviated [4]. A lot of works have been done on CNTs-based hierarchical composites and the exhilarating results have been obtained. The matrix dominated properties of hierarchical composites, such as fracture strength, fracture toughness and interlaminar shear strength,

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have been improved [4–8]. As the analog of CNTs, boron nitride nanotubes (BNNTs) also have high tensile modulus and tensile strength like CNTs, which endows BNNTs a great potential application as nanoscale reinforcements like CNTs [9]. What's more, due to better oxidation resistance up to 900 °C [10], BNNTs are more suitable than CNTs as nanoscale reinforcements in those composites such as SiC_f/SiC that are employed at high temperature in oxidizing environment.

However, not like CNTs-based ones, researches on BNNTs-based hierarchical composites are still in embryonic stage and receive little attention except from our previous study about BNNTs-based SiC_f/SiC hierarchical composites [9,11–13]. According to the existing results reported [9,13], it is confirmed that BNNTs can arrest and deflect cracks at nanoscale level by debonding at nanotube/matrix interface as fibers do at micro-scale level. It gives rise to increased toughness and improved damage resistance of matrix. More essentially speaking, it is ascribed to the fact that BNNTs play a crucial role in controlling damage initiation and evolution in hierarchical composites [14]. While almost all studies have been focused on macroscopic properties, little attention has been paid to the effect of BNNTs on damage process in hierarchical composites. So in-depth investigation on damage initiation and evolution of BNNTs-based hierarchical composites is urgently needed. It will be beneficial to comprehend the function of BNNTs in hierarchical composites.

In this research, we report, for the first time, the influence of BNNTs on damage initiation and evolution of SiC_f/SiC composites. Additionally, it is well known that BNNTs/matrix interfacial bonding dictates the debonding and pull-out of nanotubes, and thus affects damage process in hierarchical composites as well [9]. So for comparison, a thin layer of BN interphase was deposited on BNNT surface to optimize the interfacial bonding. In the present study, in situ monitoring of damage process in BNNTs-based SiC_f/SiC hierarchical composites under stress was carried out via acoustic emission (AE) technique simultaneously. To the best of our knowledge, AE investigation on damage process in BNNTs-based SiC_f/SiC hierarchical composites has not yet been reported elsewhere. Direct observation of damage patterns at different stress levels and after final failure by optical microscopy was employed for further confirmation about AE results. Possible damage mechanisms based on all experimental results were discussed as well.

2. Experimental

BNNTs were in situ grown on SiC fiber surface by a simplified ball milling, impregnation and annealing method, which has been reported elaborately in our previous work [15]. To optimize BNNTs/matrix interfacial bonding, BN interphase was deposited on BNNT surface via chemical vapor deposition (CVD) method at 900 °C using BCl_3 (7.5 sccm) and NH_3 (15 sccm) as the source gas under a pressure of 0.5 kPa for 10 min. Then SiC fiber cloths with BNNTs in situ grown were stacked and compressed together with a volume fraction of fibers of about 40%. Polymer impregnation/pyrolysis (PIP) and chemical vapor infiltration (CVI) method were used for SiC matrix densification. The former was aimed at infiltrating matrix into fiber bundles using polycarbosilane (PCS) as ceramic precursor, which was pyrolyzed at 900 °C in Ar atmosphere. The latter was adopted for inter-bundle and interlaminate matrix deposition, which was conducted at 1000 °C using methyltrichlorosilane (MTS, CH_3SiCl_3) and H_2 as the source gas. Finally, virgin SiC_f/SiC composites, as-grown and BN-coated BNNTs-based SiC_f/SiC hierarchical composites were fabricated. These composites are denoted as F, N-F and CN-F, respectively.

To evaluate mechanical properties such as flexural strength, strain at flexural strength and proportional limit stress, three-point bending test was performed on a CRIMS-DDl20 universal testing machine with a span of 30 mm at a cross head speed of 0.1 mm/min. Composites were cut into bar specimens with the dimension of 2.9 mm × 5.0 mm × 45 mm for bending test. AE technique was employed to detect in situ information of damage process in composites during three-point bending test. AE activity, originating from the rapid release of strain energy due to crack formation and propagation inside composites, was real-timely monitored. In this study, AE detection was conducted by using a two-channel MISTRAS acquisition system (Physical Acoustics Corporation) with a sampling rate of 2 MHz. Two nano-30 (Physical Acoustics Corporation) sensors were mounted on the surface of specimens to record AE signals. Vacuum grease was used as a coupling agent. Two pre-amplifiers were also adopted and set with the pre-amplification of 40 dB and band-pass filtering of 20–1200 kHz. To avoid possible noise recording, the threshold was set at 45 dB. The damage patterns in composites at different stress levels and finally failed were examined by optical microscopy. In addition, Hitachi SU8220 field-emission scanning electron microscopy (SEM) and JEM-2100F field emission transmission electron microscopy (TEM) were used to characterize the morphology and microstructure of as-grown and BN-coated BNNTs. The fracture morphology of fibers and nanotubes in composites were also investigated via SEM.

3. Results and discussion

3.1. Morphology and microstructure of BNNTs in situ grown on fiber surface

The morphology and microstructure observation of as-grown and BN-coated BNNTs on fiber surface were conducted by SEM and TEM, as presented in Fig. 1. It can be seen clearly from Fig. 1(a) and (c) that the fiber is covered by BNNTs fully. Moreover, as-grown BNNTs exhibit a unique morphology of the bubble-chain walls, as shown in Fig. 1(b). It results from the stress-induced sequential growth mechanism, which has been discussed in detail in our previous paper [15]. After the deposition of BN interphase for BNNTs/matrix interface optimization, the morphology of the bubble-chain walls disappears. TEM observation inserted in Fig. 1(d) indicates that BNNTs are coated with BN interphase uniformly, of which the thickness is about 20–25 nm. By the way, BNNTs have a multi-walled and bamboo-like structure, which is also ascribed to the unique growth mechanism.

It should be clarified that the aim of depositing an interphase on nanotube surface is to optimize the BNNTs/matrix interfacial bonding or, more precisely speaking, to reduce the interfacial bonding strength. Without an interphase deposited, the BNNTs/matrix interfacial bonding is quite strong due to mechanical interlocking caused by the bubble-chain walls and thermal residual clamping stress on the interface [16–19]. In this case, debondings at BNNTs/matrix interface and subsequently crack deflections are inhibited. After the deposition of BN interphase, the morphology of bubble-chain walls disappears and thermal residual clamping stress is relieved [20]. Thus, the BNNTs/matrix interfacial bonding strength decreases and crack deflections occur easily. Consequently, it will change crack development in matrix. The effect of BNNTs as well as the BNNTs/matrix interface on damage process in hierarchical composites both are investigated in the following sections.

3.2. Mechanical properties and fracture morphologies of composites

As-grown and BN-coated BNNTs were incorporated into SiC_f/SiC composites to produce nanotube-based hierarchical composites. Mechanical properties of these composites were evaluated, as summarized in Table 1. It can be found that the incorporation of as-grown BNNTs brings about a slight decrease in flexural strength of composite while a minor improvement is obtained from BN-coated BNNTs. The strain at flexural strength of composite N-F is undermined largely by 37.9% after in situ growth of BNNTs without an interphase. In contrast, that of composite CN-F only drops slightly by 12.6% after depositing an interphase on BNNT surface. The fracture morphologies of two reinforcements in hierarchical composites, i.e. nanotubes and fibers, were also examined, as exhibited in Figs. 2 and 3. As clarified above, without