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In situ self-sensing of delamination initiation and growth in multi-directional laminates using carbon nanotube interleaves

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ABSTRACT

Self-sensing capability of carbon nanotube buckypapers (BPs), played as in situ sensor on delamination damage in multi-directional laminates, was investigated in this paper. BPs were interleaved into these interfaces of the laminates, where delamination tends to happen firstly under tensile loading. The inserted BPs are porous and conductive, which can benefit infiltration of epoxy resin and give contribution to damage monitoring. Systematic analysis of resistance variation ($\Delta R/R_0\%$) against strain has demonstrated that BPs are sensitive to the initiation and growth of delamination. When delamination initiates, a sudden rise of the $\Delta R/R_0\%$ or the slope of the $\Delta R/R_0\%$-strain curves is observed and corresponding delamination initiation stresses are simultaneously obtained. Moreover, effect of thicker laminates on the sensitivity is also examined and the self-sensing ability of BPs has been further proved. Finally, tensile properties of the BPs interleaved laminates slightly change compared with these of base laminates.

1. Introduction

Due to the one-by-one stacking nature of composite laminates, delamination is a critical damage form, which can drastically reduce the stiffness and consequently degrade the long-term performance [1]. Both static and dynamic loading will cause delamination cracks, and these invisible internal cracks are usually not easy to be detected [2]. Once delamination occurs, it would propagate quickly until structural failure [1,2]. Therefore, real-time identifying delamination initiation and immediately restraining its propagation are necessary to avoid a catastrophic failure as well as extend the service life of composite structures.

Among various damage detection techniques, acoustic emission is one of the most used non-destructive testing methods, which have already been used to real-time monitor the initiation and growth of delamination for some special lay-ups, e.g. $[0^\circ_n/\pm45^\circ_n/90^\circ_n]_s$ [3] and $[\pm\theta]_s$ ($18^\circ < \theta < 44^\circ$) [4–8] under in-plane tensile load. C-scan is another mature method applied to monitor damages in composites [9]. This technique is usually applied to detect some established damages. In fact, both acoustic emission and C-scan techniques play as external sensors, strongly depending on the experience of manipulating personnel and sometimes are not convenient for practical applications [10]. In view of this, embedded sensors have been proposed to monitor damages within composites in a real-time and in situ mode, such as Fiber-Bragg-Grating [11], piezoresistive sensor [12], and newly developed carbon nanotube (CNT) based nano-detector [13]. The former two types can solely provide sensitive function, and they further need special surface treatment which will degrade mechanical properties of structures [11,12]. Due to the super mechanical and electrical properties, CNT has attracted more and more attentions in the field of structural and functional materials [14]. Many efforts have been carried out and have demonstrated that static and dynamic mechanical properties of fiber reinforced plastics can be improved significantly by incorporating a small amount of CNTs [15–18]. Besides, highly conductive CNTs provide an electrical network within insulating matrix which performs as an inherent sensor to identify sensing capabilities in terms of strain variation [19–23] and global damage [24–29]. Actually, composite laminates tend to damage firstly at specific interfaces or within plies under specific loading. Therefore, it is rational to localize nano-sensor at specific site where needs damage measurement. Free-standing CNT Buckypaper (BP) is considered as one promising alternative sensing approach due to its advantages providing both structural and functional contributions to composites [24–29]. Most importantly, it is flexible to be localized at any site where needs to be monitored or reinforced. Zhang et al. [10]. Du et al. [30] and Kravchenko et al. [31] addressed damage self-sensing and structural strengthening functions of CNT networks in unidirectional laminates examined under mode I or mode II loading. It can be seen that previous studies in terms of

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delamination damage identification usually considered unidirectional laminates and several simple loadings [10,30,31], especially mode I or mode II with pre-cracks. Practically, laminates are mainly made up of multi-directional plies and served under specific loading without pre-crack.

Therefore, this research aims at studying the damage self-sensing ability of BP interleaved in various multi-directional laminates without pre-crack, where BP serves as real-time and in situ sensor. Glass fiber/epoxy prepregs were used to prepare laminates with predesigned lay-ups of $[\pm\theta]_S$ and $[45°/0°/\text{-}45°/90°]_S$, in which BPs were inserted into the potential debonding interfaces and then co-cured with the lay-ups. Tensile testing was carried out to obtain stress-strain and resistance variation ($\Delta R/R_0$%)-strain curves, which assesses the self-sensing ability of BPs. Furthermore, the thickness effect of laminate on the BP sensitivity was also examined to prove the sensing ability of the proposed BPs. Finally, ultimate tensile properties were discussed to evaluate the comprehensive effect of BPs on these laminates.

2. Experimental

2.1. Preparation

The used multi-walled CNTs have an average diameter of 10 nm and variable lengths between 5 and 15 μm (Chengdu Organic Chemicals Co., P.R.China). The CNTs were processed as suspension, and then porous BPs were obtained through a positive filtration method, which was detailed in our previous study [17]. The thickness of the BPs was controlled within 35–50 μm and the in-plane conductivity was measured as 13–18 S/cm.

To evaluate the self-sensing capability of BPs on delamination, insulating glass fiber/epoxy prepregs (Shenying Carbon Fiber Bicycle co., P.R.China) were used to fabricate laminates, and thus the $\Delta R/R_0$% corresponding to delamination is solely caused by deformation or damage of BPs. The thickness, weight percentage of epoxy resin and areal density of the prepregs are supplied as 0.13 mm, 33% and 130 g/m^2, respectively. The laminates in this study were set as $[45°_4/0°_4/\text{-}45°_4/BP/90°_4/BP_{1/2}]_S$, $[20°_4/BP/\text{-}20°_4]_S$, $[30°_4/BP/\text{-}30°_4]_S$ and $[40°_4/BP/\text{-}40°_4]_S$ according to previous reports [3–8], which revealed that $[0°_n/\pm 45°_n/90°_n]_S$ and $[\pm\theta_n]_S$ ($18° < \theta < 44°$) lay-ups tended to delaminate at the interfaces of $-45°/90°$, $90°/90°$ and θ/-θ when subjected to axial tension. For the above predesigned laminates, each ply angle was simultaneously chosen as 4 plies to satisfy the thicknesses requirement of tensile testing specimens in accordance with ASTM D3039 standard. The ply stacking sequences and inserted sites of porous BPs are described in Fig. 1a, b and c. The sandwich-like laminates were cured in an autoclave, which involved firstly heating up from room temperature to 85 °C and holding for 40 min, followed by elevating the temperature to 130 °C with a pressure of 0.62 MPa for another 120 min.

2.2. Characterization

Microstructures of the cured laminates were checked using a SEM machine (Philips XL 30 ESEM instrument) firstly to confirm the good wettability. Then, these laminates were cut into standard testing specimens according to ASTM D3039 standard. Two ends of the tensile specimens are coated with conductive adhesive (yellow color in Fig. 1d), and then connected with copper wires and a Keithley 2450 source meter (Tektronix, America), as shown in Fig. 1d. Finally, glass fiber/epoxy end tabs are bonded on the surfaces of the testing specimens. The end tabs not only help to eliminate end-effect, but also separate the circuit connection between specimen and the testing fixture. All the tests were conducted by a testing machine (Zwick Z050TN, Germany), with a gauge length of 50 mm using strain gauge and a cross-head speed of 2 mm/min. Stress, strain and resistance were simultaneously collected during the test. With the increasing of loading, the collected resistance signal would change significantly if delamination occurred. At this time, testing was stopped to polish side-sections of the specimens, followed by observing them with a SEM machine to confirm whether delamination occurred. Other specimens were tested to their ultimate fracture to evaluate the comprehensive effect of the BP interleaves on laminates. Some literature reported that thicker laminates tend to delaminate more easily than thinner laminates with same lay-up angles [3–5,32]. Thus, thicker laminates, $[\pm\theta_n]_S$ (θ = 20°, 30°, 40°) and $[45°_n/0°_n/\text{-}45°_n/90°_n]_S$ (where n is set as 6), were also prepared to check the monitoring sensitivity of the proposed BPs.

Finally, ultimate tensile properties of both base and interleaved laminates were also examined to evaluate the effect of BPs on the structural performance of laminates. Five to six specimens were tested in each case to ensure the repeatability of the results.

3. Results and discussion

3.1. Morphology of the laminates

Fig. 2 gives side-sections of the prepared laminates, i.e., $[30°_4/BP/\text{-}30°_4]_S$ and $[45°_4/0°_4/\text{-}45°_4/BP/90°_4/BP_{1/2}]_S$ (Chosen as representatives). Fig. 2a and b shows that BPs form into thin BP/epoxy interleaves after curing, in which, CNTs distribute homogeneously through the thickness direction of the interlayers (shown in Fig. 2c and d). Fig. 2d indicates that the BP interleaves are infiltrated well with

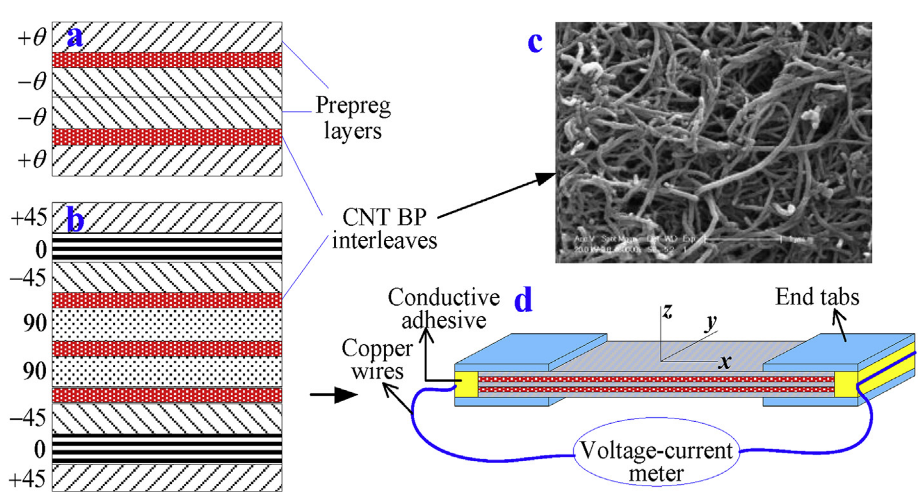

Fig. 1. Laminated sketch of the BP interleaves and prepreg layers, (a) $[\pm\theta_n]_S$ (θ = 20°, 30° and 40°), (b) $[45°_n/0°_n/\text{-}45°_n/90°_n]_S$, (c) SEM image of BP, and (d) testing specimen.