Increasing the interlaminar fracture toughness and thermal conductivity of carbon fiber/epoxy composites interleaved with carbon nanotube/polyimide composite films

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Abstract: Carbon fiber/epoxy (CF/EP) composites are widely used in the aerospace industry, but their interlaminar properties and out-of-plane thermal conductivity are poor because of the lack of CF reinforcement in the interlaminar area. We prepared carbon nanotube/polyimide (CNT/PI) composite films and used them to improve the interlaminar fracture toughness (ILFT) and thermal conductivity of laminates of CF/EP and CNT/PI. Interleaving CNT/PI films with unidirectional CF/EP prepregs increased the mode I (the pre-cracked laminate failure is governed by peel forces) and mode II (the crack is propagated by shear stresses) ILFT of CF/EP laminates by 260% and 220%, respectively, which is attributed to the reinforcement effect of the CNTs and the plastic deformation of PI film. In addition, the out-of-plane thermal conductivity of the laminates is improved by introducing CNT/PI films because of their intrinsic high thermal conductivity and the continuous conductive network of CNTs. This toughening method provides an effective strategy for improving the thermal conductivity and mechanical properties of CF/EP laminates simultaneously.

Key words: CNT/BOH film; ILFT; Thermal conductivity; CF/EP laminates; Thermal conductive network

1 Introduction

Carbon fiber/epoxy (CF/EP) laminates have been widely used in aerospace, defense and automotive industries because of their excellent specific strength and stiffness[1–3]. However, owing to the lack of CF reinforcement in the interlaminar area, the out-of-plane thermal conductivity and interlaminar properties are poor, limiting their applications in many fields[4–5]. Moreover, to meet the requirement of advanced applications, the development of high-performance and structure-function integration are trending research topics for carbon fiber reinforced polymers[6–7]. It is important to enhance the interlaminar fracture toughness (ILFT) and thermal conductivity of the laminates without scarifying the other static mechanical properties[8–9].

To simultaneously achieve high-performance structural composites and satisfy the heat dissipation requirement, introducing nano materials into CF/EP composites has bcome the subject of focus[10–13]. In particular, carbon nanotubes (CNTs) as a functional second phase has been extensively investigated[14–19]. For instance, Quan et al.[20] introduced 1.0% CNTs into the epoxy matrix, and the mode I ILFT (G_{IC}) of CF/EP composites was improved by 25%. Prasad et al.[21] used CNTs and poly(ether sulfone) together as tougheners in epoxy matrix for enhancing the toughness of CF/EP laminates. Yao et al.[22] deposited CNTs on carbon fiber surface to enhance the interfacial adhesion, the interlaminar shear stress (ILSS) and G_{IC} of modified laminates were 32% and 95% higher than that of the control sample. Although the research for adding nano materials into matrix resins has reached greater heights, the unsatisfactory dispersion of nanosized second phase and increased viscosity of modified matrix resin remain insurmountable problems.

The interleaving method is a kind of effective and simple way to improve the ILFT without increasing viscosity of matrix resin. Li et al.[23] fabricated continuous and randomly distributed CNTs by float-

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ing catalytic chemical vapor deposition method to improve the ILFFT of CF/EP laminates. The maximum G_{IC} under tensile loading is twice that of the control sample. Khan et al.[24] investigated the effects of bucky paper on ILFT of CF/EP laminates and found 31% and 104% enhancement in ILSS and G_{IIC}, respectively. Ou et al.[25] demonstrated improvements of 60% in G_{IC} by interleaving CNTs veils in CF/EP laminates. Shin et al.[26] prepared CNTs/epoxy films through three-roll milling and ultrasonication method to toughen CF/EP laminates, and the G_{IIC} of unidirectional CF/EP laminates increased by 126.7% after interleaving 3% (mass fraction) CNTs/epoxy film. However, inserting pure CNTs layers or CNTs/epoxy film into the carbon fiber/epoxy laminates is prone to cause stress concentration, limiting the further improvement of ILFT. Moreover, Kaynan et al.[27] reported 2-fold increment in mixed-mode I/II ILFT by introducing CNTs/polyvinyl butyral nanofibrous interleaves. Although the nanofibrous interleaves can improve the ILFT effectively, the high equipment cost still limits their commercial application. In addition, most researches unfortunately focused on the enhancement of ILFT only and did not pay enough attention to the thermal conductivity of CF/EP laminates.

In our previous work[28], hydroxyl-functionalized polyimide BPADA-ODA/HAB (BOH) was synthesized as toughener for tetrafunctional epoxy resin and showed excellent toughening effect, which is attributed to the nano-sized phase separation and the proper decrement of crosslinking density of epoxy resin. In this work, CNT/BOH hybrid films were prepared as interleaves to improve the ILFT and thermal conductivity of CF/EP laminates. The effects of CNTs content in CNT/BOH films on the mode I and mode II ILFT, flexural strength and through-thickness thermal conductivity were investigated. The experimental results show that interleaving the laminates with CNT/BOH films increase G_{IC} and G_{IIC} values by 260% and 220%, respectively. In addition, the through-thickness thermal conductivity of CF/EP laminates are improved by 32% through the introduction of CNT/BOH films.

2 Experimental

2.1 Material

Multiwall carbon nanotubes (MWCNTs, purity of 95%, diameter of 10-20 nm, length of 10-30 μm) were purchased from Chengdu Organic Chemistry Co. Ltd., China. BPADA-ODA/HAB (BOH) were synthesized in our laboratory. 1-Methyl-2-pyrrolidinone (NMP) was purchased from Aladdin Chemical Corporation. The unidirectional carbon fiber reinforced tetrafunctional epoxy resin prepregs were manufactured by Tianjin Hansort Co., Ltd, the specifications of prepregs are shown in Table 1.

2.2 Preparation of CNT/BOH interleaves

CNTs were purified with HCl and acidified by mixture reagent of concentrated sulfuric acid/nitric acid to introduce carboxyl groups, the details of which are presented in Fig. S1. Then the acidified CNTs were dispersed in NMP using an ultrasonic probe for 20 min. After that, BOH powders were added to form mixture, in which the solid content is 1%. The mixture was ultrasonically treated and casted on clean glass plate by metal roller, then dried in the oven at 100 °C for 48 h to remove solvent. After that, the CNT/BOH films with thickness of 7 μm were peeled off glass plate by immersed in boiled distilled water. Films with different CNTs content were obtained by changing the CNTs loading and denoted as BOH-*x*%CNT, in which *x* is the mass percentage of CNTs in CNT/BOH film.

2.3 Fabrication of CF/EP laminates

For the control specimen, 32 plies of CF/EP prepregs with size of 260 mm × 210 mm were laminated. A poly tetra fluoroethylene (PTFE) film with

Table 1 Specifications of unidirectional carbon fiber/epoxy prepregs

Parameter	Specifications
Carbon fiber	T700
Resin content/%	38±2
Area density/(g/m^2)	150±3
Thickness of prepreg/mm	0.11
Epoxy resin	Hansort$^{@}$6320
T_g of epoxy resin/°C	210
Tensile strength of epoxy resin/MPa	75
Longitudinal tensile strength of	1 900

thickness of 13 μm was placed between the 15th and 16th layer of the prepregs to initiate the cracks for delamination in both mode I and mode II test. For the interleaved specimens, the CNT/BOH film was inserted subsequent to the initial crack tip from PTFE film. After the stacking procedure, the prepregs were cured by hot-pressing them at 150 °C for 2 h and post-cured at 180 °C for 2 h under 0.1 MPa pressure. Finally, laminates with thickness of 4 mm were obtained. As for flexural strength and thermal conductivity tests, CNT/BOH films were inserted between each layer of CF/EP prepregs, and the average thickness of laminates was 2 and 1 mm, respectively (Fig. 1(a)).

2.4 Characterization

The G_{IC} of the laminates were measured through double cantilever beams tests (DCB) according to ASTM D5528. As presented in Fig. 1(b), DCB specimens with length (L) of 140 mm and width (b) of 20 mm were machined from the hot-pressed laminate, with initial crack length a_0= 40 mm. A pair of hinges were bonded on both sides of DCB specimen. A scale bar was painted on specimen edge which was sprayed with white primer in order to easily detect the delamination propagation during tests. DCB specimens were performed through a universal testing machine (Shimadzu AG-IS) with the crosshead displacement rate of 1 mm/min. Digital microscope was placed near the specimen edge in order to record the crack propagation. G_{IC} was calculated as following:

$$G_I=\frac{nP\delta}{2ba} \tag{1}$$

where δ is load point displacement, P is max load, a is delamination length, b is width of sample, n is the parameter of Compliance Calibration (CC) method, which is obtained from the slope of the fit line from the least squares plot of $\log(\delta_i/P_i)$ versus $\log(a_i)$.

G_{IIC} was obtained using 3-point end-notched flexure (3-ENF) test according to ASTM 7905. As shown in Fig. 1(c), samples with length of 140 mm

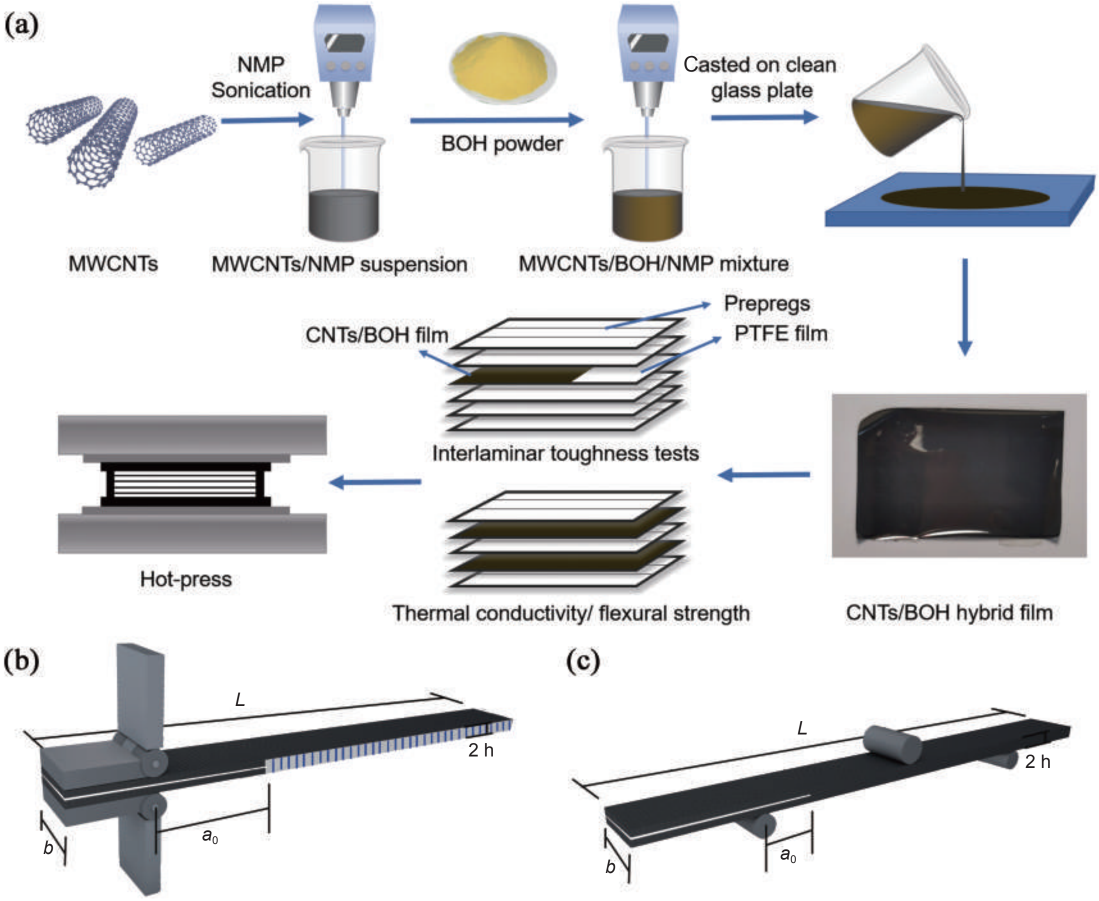

Fig. 1 (a) Schematic diagram of the fabrication process. Schematic diagram of (b) DCB samples and (c) ENF samples and width of 20 mm were manufactured from the laminate. 3-ENF specimens were tested at 0.5 mm/min displacement rate using universal testing machine, which is same as DCB tests. The G_{IIC} was calculated as follows:

$$G_{\mathrm{II}}=\frac{9P^2a_0^2}{16b^2E_{\mathrm{f}}h^3} \tag{2}$$

where P is the max force, a_0 is initial delamination length, b is width of specimen, E_{f} is flexural modulus, h is specimen half-thickness. Flexural strength testing was performed according to ASTM D790 standards at crosshead displacement rate of 1 mm/min, with specimen size of 70 mm × 13 mm × 2 mm.

Through-thickness thermal conductivity was measured through a laser flash analysis machine (Netzscz LFA 467) according to GB/T 22588-2008. Samples were square with an average size of 10 mm. The thickness was 1 mm. To obtain the through-thickness thermal diffusivity (α), an energy pulse was exerted at one side of specimen, and temperature on the backside was measured. The thermal conductivity (λ) was subsequently calculated using the following equation:

$$\lambda=\alpha\times C_{\mathrm{p}}\times\rho \tag{3}$$

where C_{p} is specific heat, ρ is the density of samples, the detailed density, specific heat capacity, and porosity of laminates are shown in Table 2. Scanning electron microscopy (Bruker JSM-7900F) was used for investigating the surface morphologies of laminates.

Table 2　Density, specific heat capacity, and porosity of laminates for thermal conductivity testing

Interleaf	Density/(g/cm^3)	Cp/ J/(g·K)	Porosity/%
Control	1.667	0.892	0.26
BOH	1.624	0.945	0.21
BOH-0.1%CNT	1.624	0.949	0.61
BOH-0.3%CNT	1.552	0.958	1.40
BOH-0.5%CNT	1.662	0.898	2.00
BOH-1.0%CNT	1.675	0.886	3.20
BOH-2.0%CNT	1.669	0.888	4.00

3　Results and discussions

3.1　G_{IC} of CF/EP laminates

In general, the G_{I} values at the non-liner point ($G_{\mathrm{IC\text{-}NL}}$) and average value of G_{I} during the stable crack propagation ($G_{\mathrm{IC\text{-}Prop}}$) are used to describe the interlaminar fracture behavior of laminates. Typical load-displacement curves and R-curves of the control sample and toughened laminates are displayed in Fig. 2. The load of the control laminates decreases continuously with increasing opening displacement. On the other hand, laminates toughened with CNT/BOH film exhibit an increase of load after initial crack, and then the load decreases gradually. Compared with the control sample, the toughened laminates show higher fracture load and longer opening displacement when the crack propagated near the end. R-curves indicate the relationship between the G_{I} values and delamination length in DCB tests. As shown in Fig. 2(b), the control sample presented a relatively flat R-curve owing to the moderate fiber bridging against the crack propagation. While the R-curves of toughened laminates first rose gradually and then remained stable with the crack propagation, which is attributed to the introduction of additional energy dissipation mechanisms from the CNTs and BOH component. Interleaving CNT/BOH film results

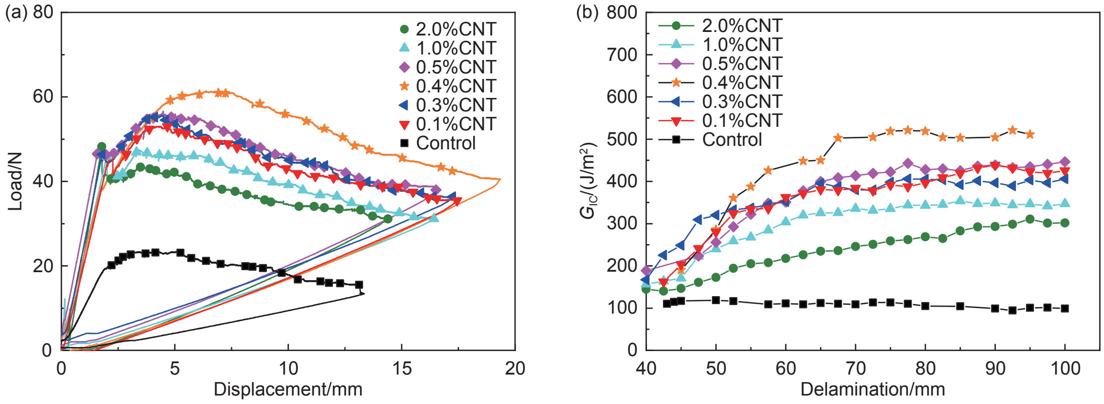

Fig. 2　(a) Representative mode I load-displacement curves and (b) R-curves of specimens with and without CNT/BOH films in a significant enhancement of both $G_{\text{IC-NL}}$ and $G_{\text{IC-Prop}}$ for carbon fiber/epoxy laminates. For instance, $G_{\text{IC-prop}}$ increased from 140 to 505 J/m^2 by interleaving with BOH-0.4%CNT films, enhanced by 260%. However, as the CNTs loading in BOH film further reached up to 1.0%, the $G_{\text{IC-Prop}}$ decreased to 338 J/m^2. This is probably related to the inherent agglomeration characteristic of CNTs due to the Van der Waals force[29].

Typical SEM images of the fracture surfaces of the laminates after DCB tests are displayed in Fig. 3. The control specimen presented smooth carbon fiber surface and brittle fracture morphology of the epoxy resin, indicating typical adhesive failure. In comparison, the fracture surfaces of the CNT/BOH film interleaved laminates present lotus-like characteristics due to the plastic deformation of BOH films, which is beneficial for energy dissipation. In addition, CNTs are pulled out from the epoxy resin matrix, triggering CNTs bridge and crack deflection, which suppress crack propagation. However, obvious agglomeration of CNTs was observed when the CNT loading was higher than 0.5% (Fig. 3(f) and Fig. S2), which is

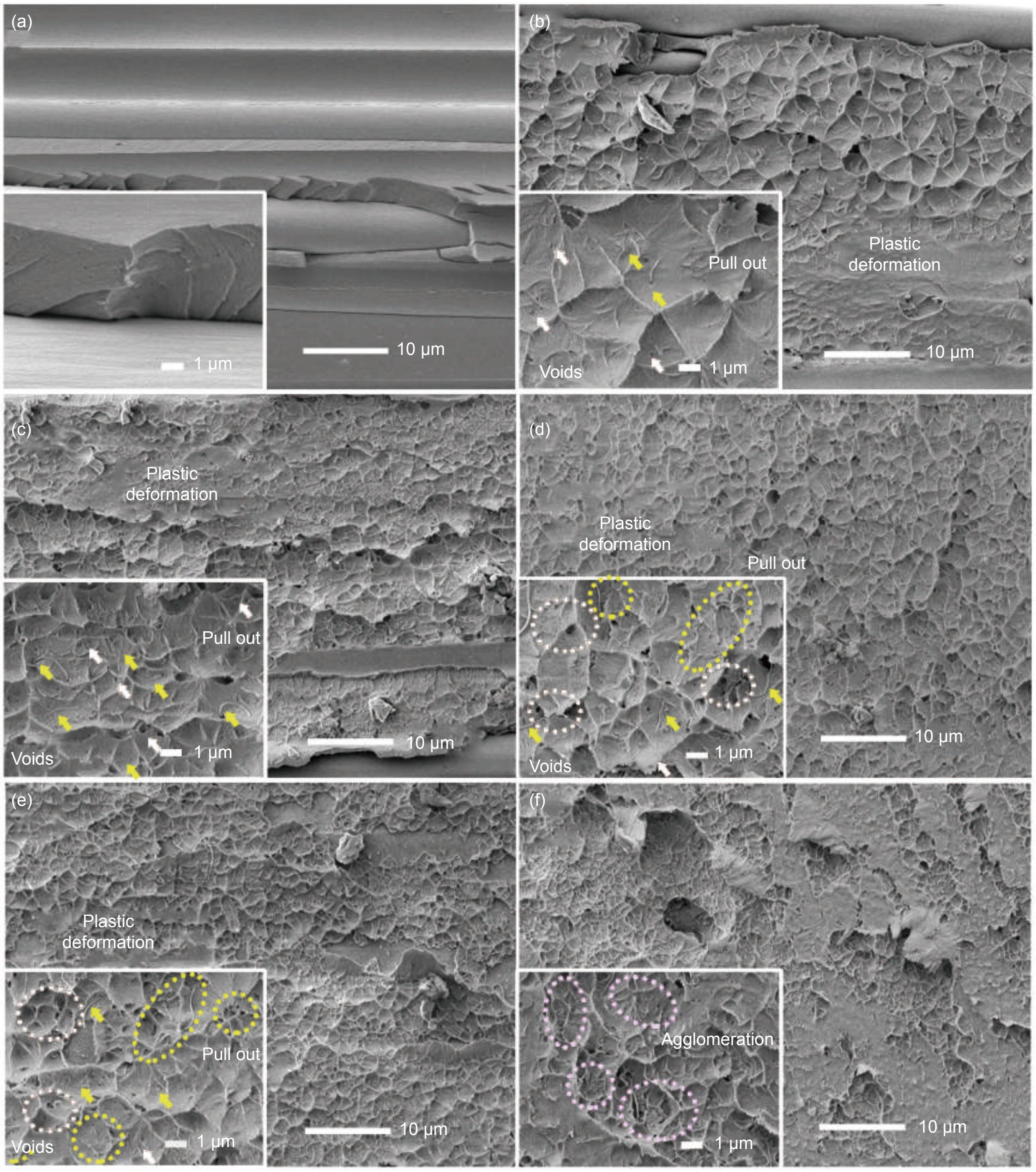

Fig. 3 SEM images of the fracture surface under mode I loading: (a) control, (b) BOH-0.1%CNT, (c) BOH-0.3%CNT, (d) BOH-0.4%CNT, (e) BOH-0.5%CNT, (f) BOH-1.0%CNT as interleaves

prone to stress concentration and can reduce mechanical properties.

3.2 G_{IIC} of CF/EP laminates

The load versus displacement curves and G_{IIC} values of ENF samples are shown in Fig. 4. All the specimens exhibit typical brittle behavior at the crack tip. Laminates interleaved with CNT/BOH films show higher load and displacement at the break point compared with those of the control sample. As shown in Fig. 4(b), G_{IIC} is improved after introducing the CNT/BOH films in CF/EP laminates. A significant improvement of G_{IIC} (2 708 J/m^2) is obtained by interleaving BOH-0.4%CNT film, which is 2.2 times higher than that of the control laminate.

Fracture morphologies of the control and toughened laminates after ENF tests are presented in Fig. 5. Smooth carbon fiber surfaces and brittle facture morphology of epoxy resin parallel to crack growth direction were observed in the control sample, indicating characteristic adhesive failure of composites.

In CNT/BOH toughened laminates, obvious pulling-out and cavities after CNTs debonding are visible, which consume extra fracture energy during crack growth. In addition, shear deformation effect of BOH plastic phase were deepened by introducing CNTs into the BOH films, in which the CNTs acted as “pins” and induced crack deflection. However, as the CNT loading were further increased to 1.0%, agglomeration of CNTs was observed in the interlayer region and interface between CF and EP (Fig. 5(f) and Fig. S3), which is not beneficial for the enhancement of ILFT.

3.3 Flexural strength of CF/EP laminates

As is known that the interleaving method could improve the interlaminar properties but always scarifies the longitudinal mechanical properties due to the decreasing carbon fiber volume fraction of laminates[30–31]. However, as shown in Fig. 6, the flexural strength of the CNT/BOH films interleaved laminates is slightly higher than that of the control sample. Especially flexural strength of 1 530.04 MPa is obtained by introducing BOH-0.5%CNT film, which is an improvement of 8.3% compared with that of the control laminates. Besides carbon fiber volume fraction, the shear properties of the matrix and the interfacial adhesion of CFs and matrix are important factors for the flexural strength of CF/EP laminates[32–33]. In this work, the introduction of CNTs could improve both the fiber-matrix adhesion and the shear properties of the epoxy matrix in their vicinity by increasing the fiber surface area[34]. When the content of CNTs in BOH film was increased to 1.0%, the average flexural strength of laminates decreased compared with that of the laminates interleaved with BOH-0.5%CNT film. This is due to the poor interfacial adhesion between CF and EP caused by the agglomeration of CNTs. In addition, large-scale CNTs agglomeration may cause stress concentration, decreasing the mechanical properties of composites. Compared with other interlaminar toughening methods, the CNT/BOH film is an effective interleaf which

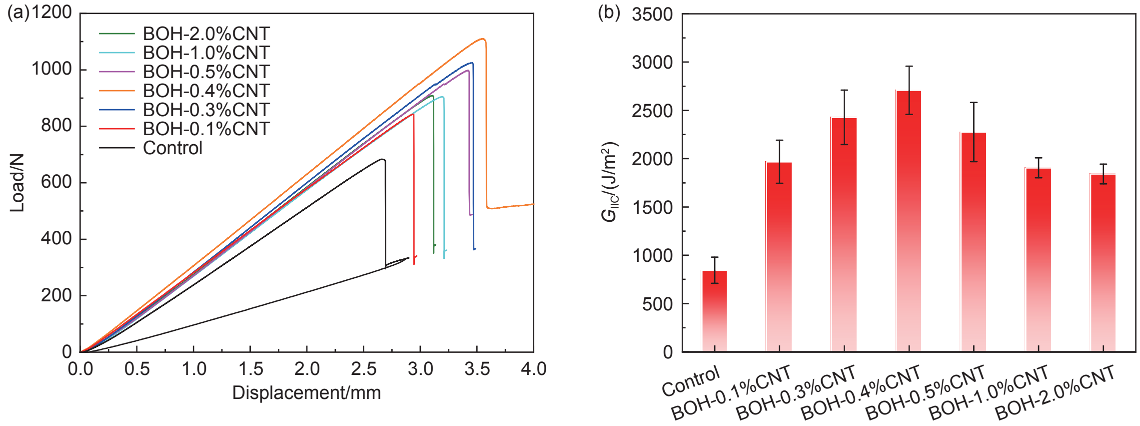

Fig. 4 (a) Typical mode II load-displacement curves and (b) G_{IIC} values of samples with and without CNT/BOH films

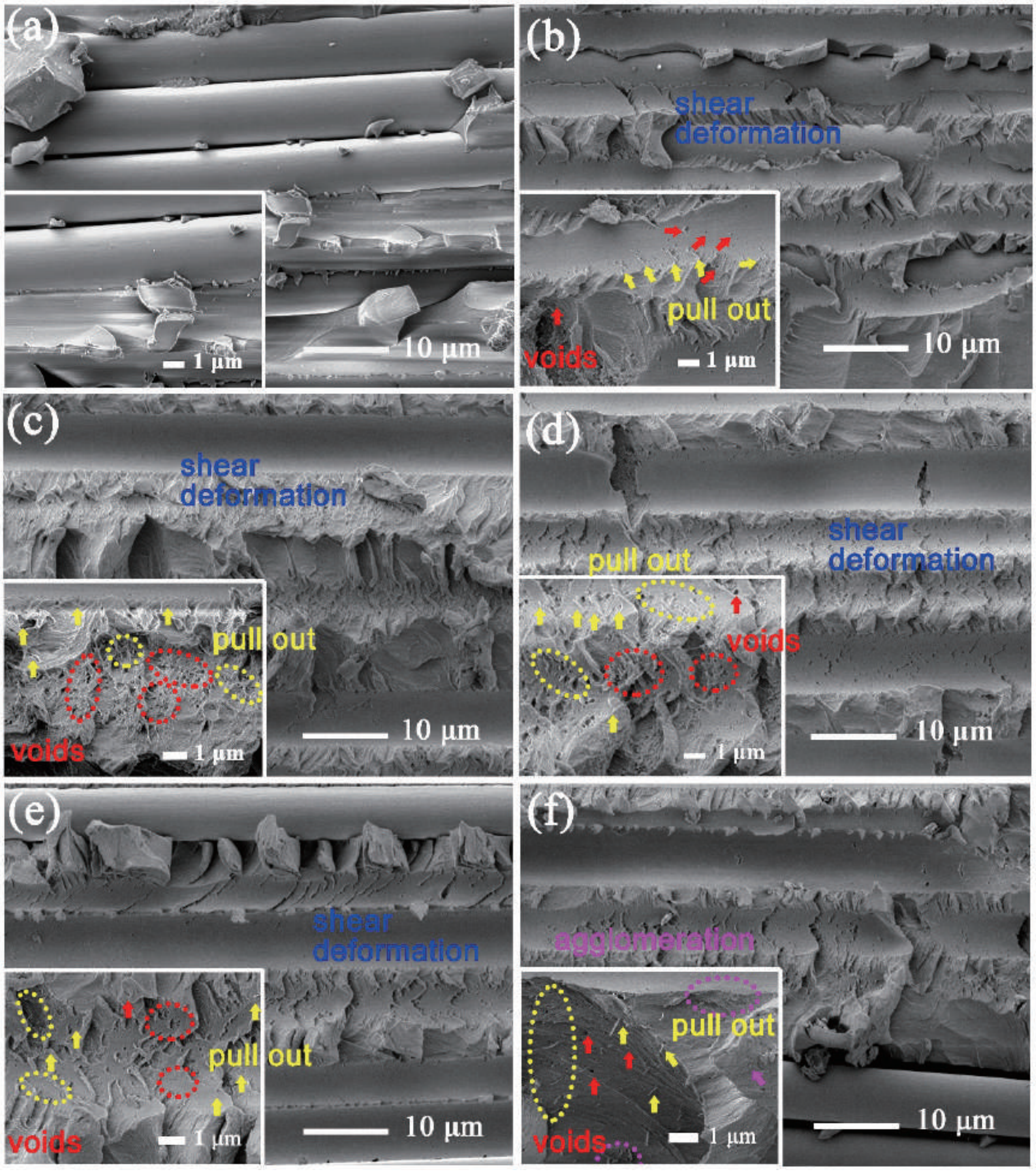

Fig. 5 SEM images of the fracture surface under Mode II loading: (a) control, (b) BOH-0.1%CNT, (c) BOH-0.3%CNT, (d) BOH-0.4%CNT, (e) BOH-0.5%CNT, (f) BOH-1.0%CNT as interleaves

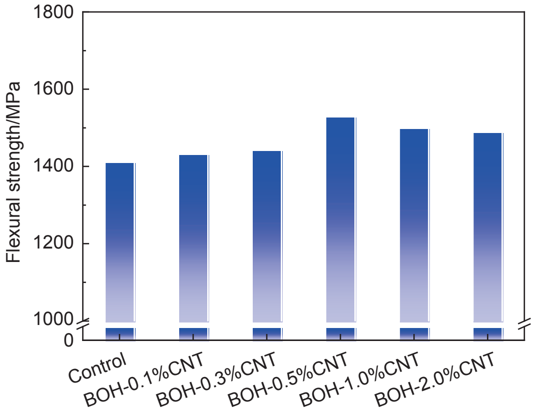

Fig. 6 Flexural strength of laminates with and without CNT/BOH films

can improve interlaminar toughness and mechanical properties of CF/EP laminates at the same time (Table S1).

3.4 Thermal conductivity of carbon fiber/epoxy laminates

Fig. 7 shows the correlation between the vertical thermal conductivity of CNT/BOH film interleaved laminates and the CNTs content. The thermal conductivity of the control laminate is 0.84 W/m·K. The lower through-thickness thermal conductivity of pure BOH films interleaved laminates is because of the fact

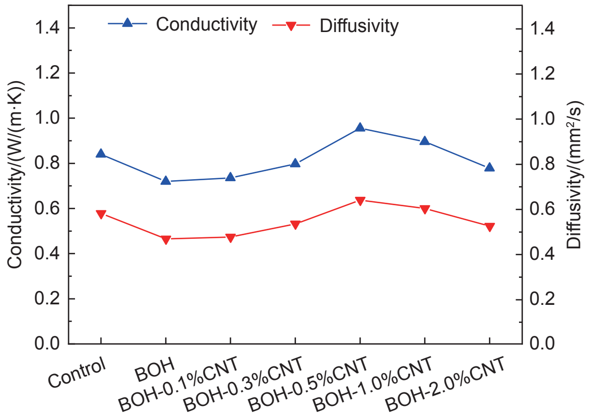

Fig. 7 Thermal conductivity and diffusivity of CF/EP laminates interleaved with CNT/BOH films with different CNT contents

that the BOH film and epoxy resin are thermal insulating materials. As shown in Fig. S4, the insertion of BOH film formed a resin enrichment area between the carbon fiber layers, resulting in a decrease in the thermal conductivity. But when the CNT content reached 0.5%, the thermal conductivity of laminates slightly increased to 0.956 W/m·K, which is 32% higher than that of the laminates interleaved with pure BOH film. This improvement can be attributed to the effective thermal conductive path created by the CNTs between adjacent carbon fiber layers. CNTs possess high intrinsic thermal conductivity (3 000 W/m·K)[35–36], and heat can be transferred more effective when CNTs form continuous conductive network in the through-thickness direction as shown in laminates interleaved with BOH-0.5%CNT films. However, when CNT content is increased further, the thermal conductivity is slightly decreased because of the aggregation of CNTs.

4 Conclusion

In this work, carbon nanotubes/co-polyimide (CNT/BOH) hybrid films were prepared as interleaves to improve the ILFT and thermal conductivity of CF/EP laminates. The experimental results show that the G_{IC} and G_{IIC} values of CF/EP laminates interleaved with CNT/BOH films are increased by 260% and 220%, respectively. This improvement is attributed to the plastic deformation of the BOH phase and the reinforcement provided by CNTs. Also, the flexural strength of CF/EP laminates is improved by 8.3%. Furthermore, the through-thickness thermal conductivity of CF/EP laminates is improved by introducing CNT/BOH films due to the high intrinsic thermal conductivity and the formation of continuous conductive network of CNTs. In summary, this toughening strategy provides an effective way for the high-performance and structure-function integration applications of CF/EP composites.

Date availability statement

The data that support the findings of this study are openly available in Science Data Bank at https://www.doi.org/10.57760/sciencedb.j00125.00018 or https://doi.org/10.57760/sciencedb.j00125.00018.

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