CARBON NANOTUBE FILM ENGINEERED FIBER REINFORCED POLYMER COMPOSITES

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ABSTRACT

Due to their excellent specific strength and modulus, fiber reinforced polymer composites (FRPCs) have been widely used in nowadays industry, especially in these weigh-sensitive structures such as satellites, aircrafts, as well as wind blades. Enhancing interlaminate performance, developing advanced FRPCs with integrated outstanding structural and functional properties, as well as innovating low cost manufacturing technologies are now some of the hot topics in the composites community. To develop an out-of-autoclave composite curing technology with lower cost, less energy-consummation and high efficiency, carbon nanotube (CNT) film fabricated by floating catalyst chemical vapor deposition method was adopted as the resistive heater to cure the glass fiber reinforced composite by its Joule heating. CNTs films were incorporated into the FRPCs either by interleaving between the lamina or coating them directly onto the composite surface. It was found that CNT film was of high porosity and possessed fast heating rate and superb thermal stability. Compared to the conventional oven curing method, both the energy consummation and processing cycle of this newly developed curing method were greatly decreased. In addition, the cure degree and mechanical property of the composites cured by CNT Joule heating were found similar to those of the composites cured by conventional oven method.

1 INTRODUCTION

Fiber reinforced polymer (FRP) composites have been increasingly used in many areas, particularly in weight- and environment-sensitive structures such as aircrafts, land vehicles, and wind blades owing to their excellent specific strength/stiffness. FRP composites that require outstanding structural performance are typically fabricated by curing the fiber/resin preform in an oven with designed pressure and temperature [1]. During this process, considerable energy is required first to heat the oven, then the air in it, and finally the composite surrounded by the hot air. As the oven is much larger than the composite part itself, this curing process is time- and energy-consuming and inefficient. Reducing the cost and increasing the efficiency of the composite curing process has been of paramount importance in recent years. New curing methods such as microwave curing, heat blanket curing, and resistive embedded heating curing have been developed [2-11].

CNTs possess an unprecedented combination of superb mechanical, electrical, and thermal properties [13-15], making them not only ideal materials for constructing multifunction composites [16-18], but also promising candidates for resistive heating [19-22]. When an electrical current passes through CNTs, they heat up quickly [23], and the heat dissipates to its surroundings through conduction and radiation [24, 25]. Many studies have reported that composite materials can be cured using the resistive heating of CNTs. For example, by dispersing 0.4 wt% CNTs in the epoxy matrix, Mas and co-workers [26] found that the composite could be cured through resistive heating of the dispersed CNTs. As the CNTs were uniformly distributed in the composites, the heat was evenly distributed throughout the whole matrix, minimizing thermal gradients in the sample. A total of 4.5 kJ of electrical energy was consumed to cure a piece 13×9.5×9 mm^3 (1.3 g) in size, while the oven roughly consumed about 3 MJ to cure a sample of the same size. Some efforts have been made to improve electrical conductivity, which is important for resistive heating. Davis et al. [27]

demonstrated that the formation of a certain degree of CNT-rich domain and the aggregation at low CNT content could improve electrical conductivity. However, the electrical conductivity is below 100 S/m at this low CNT content and is inappropriate for resistive heating [28]. Evenly dispersing high content CNTs in the matrix materials is still known to be a serious challenge, and it is even more difficult to disperse CNTs evenly in the fiber-reinforced composites. Further research efforts are needed to overcome this challenge.

Macroscale CNT film can also be used as the heating element, which circumvents the aforementioned challenge of CNT dispersion. Continuous CNT film can currently be fabricated using several different methods, such as by rolling down a pre-grown vertically aligned CNT array [29-31]. Lee and co-workers [29] demonstrated that polymer-based laminated composites can be cured using this film as a surface heater, and found that this curing process had a degree of cure comparable with that of a conventional oven heating process. More importantly, this curing process is energy saving, requiring input power of about 30 W to cure a composite of 40×50×2 mm^3 in size, while the oven curing process requires over 1 kW to cure similar composites. Alternatively, the macroscale CNT film can be made directly through the floating catalyst chemical vapor deposition (FCCVD) method [32], where the carbon sources in the FCCVD process directly convert to a CNT film in a single step. The electrothermal properties of this type of CNT film were investigated by Koziol and co-workers [33], and found that the film could be electrically heated very quickly. By inserting these films between the layers of CFRP laminate, Nguyen et al. [34] demonstrated that the composites could be cured by the resistive heating of CNT films. The storage modulus of the composites, however, was 30% lower than that of the autoclave-cured samples.

In this study, we developed a new low cost, time- and energy-saving out-of-autoclave curing process for constructing high performance composites. The macroscale continuous CNT films made using the FCCVD method are used as either the surface or embedded heating element for glass fiber reinforced polymer (GFRP) composite curing. The degree of cure, energy consumption, and mechanical properties of the obtained composites are evaluated and compared with those of the oven cured samples. The CNT film enriches the composite's functionalities, such as electrostatic discharging, electromagnetic interference shielding, lightning protecting, and deicing. In this study, deicing of the laminates based on the resistive heating of CNT film is investigated.

2 EXPERIMENTAL SECTION

2.1 CNT film fabrication

CNT film was made using the FCCVD method, which was detailed in our previous publication [35]. Fig. 1a shows the schematics of CNT film fabrication. The feedstock, which contained about 96.5 wt% ethanol, 1.9 wt% ferrocene and 1.6 wt% thiophene, was injected into the a hot CVD furnace (~1150°C) along with the carrier gas consisting of hydrogen and argon (ca. 1:1 of volume). Ethanol, ferrocene, and thiophene are used as the carbon source, catalyst precursor, and promoter for CNT syntheses, respectively. The feedstock broke down and reacted to form CNTs in the high temperature furnace, which entangled into a sock-like aerogel. This CNT aerogel was then pulled out and continuously collected by a rotating roller, resulting in a CNT sponge with high porosity, as shown in Fig. 1b. A densified CNT film was obtained by spraying ethanol on the surface of the sponge, which could then be easily peeled off the roller. The size and thickness of the film can typically be determined by the size of the roller and the connecting time. In this study, the size of the film was about 1 m^2 (Fig. 1c), large enough to fabricate a large film heater.

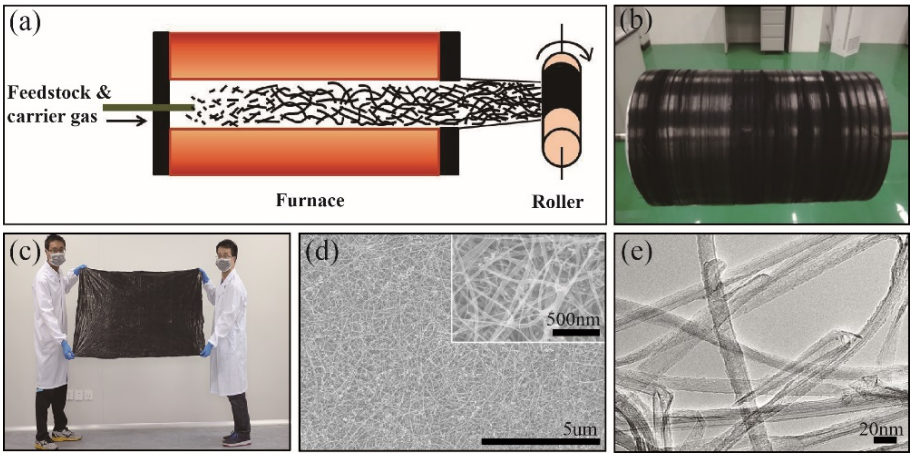

Fig.1. FCCVD CNT film: (a) schematics of film fabrication, (b) CNT sponge wound on a collecting roller, (c) a 1 m^2 size CNT film, (d) SEM morphology of CNT film, and (e) TEM morphology of CNTs.

2.2 Composite fabrication

Glass fiber prepreg with an areal density of 224 g/m^2, supplied by Weihai Guangwei Composites Co. Ltd, China, was used in this study. The prepreg consisted of 63 wt% plain woven E-glass fiber fabrics and 37 wt% epoxy matrix, and the weight ratio of the epoxy resin and amines hardener was about 95:5. Layers of prepreg 200×200 mm in size were stacked up in one direction by hand. To prepare the CNT film heater, the film was first cut into 260×200 mm pieces. Two 400-mm-long, 1-mm-in-diameter copper wires were used as electrodes. The two opposite boundaries of the CNT film were then wound around the copper wire until the final film size reached 200×200 mm. To reduce the contact resistance between the electrodes and CNT film, uniform silver paste was painted on the wire surface before the winding process. Fig. 2a shows a photograph of CNT film heater.

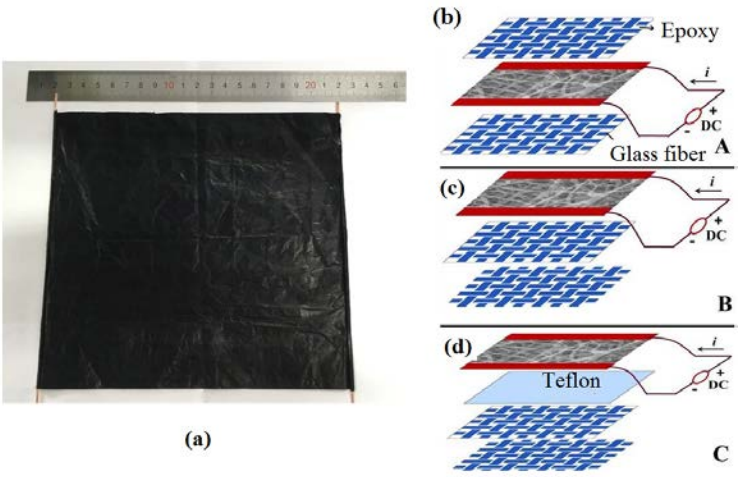

Fig.2: (a) Photograph of CNT film heater, (b-d) diagrams of CNT film/glass fiber hybrid laminate.

Three types of composites were made in this study, referred to as samples A, B and C, respectively. For sample A, the CNT film was placed in the middle of the uncured laminates. For sample B, the CNT film was placed directly onto the surface layer of the uncured laminates, while for sample C a thin layer of Teflon film was inserted between the CNT film and uncured laminates. The CNT film in sample A and B served not only as the heater and but also as a surface conductive layer in the final composites, while in sample C the film served only as the heater and could be peeled off after curing for reuse. The CNT film/glass-fiber hybrid laminate was then embedded in a vacuumed bag, with the CNT film side facing the mold. Two thermocouples were embedded in the vacuumed bag and

connected to the top and bottom surfaces to monitor the curing temperature. The two copper wire electrodes were connected to a DC power supply (ATTEN, TPR3020S). The laminate was then cured by the resistive heating of the CNT film under the vacuum condition, hereafter referred to as the C-heating. To evaluate the efficiency of this newly developed C-heating method, the controlled samples were also made by curing the similar CNT film/glass-fiber hybrid laminate in an oven with the same curing temperature profile, hereafter referred to as the O-heating method. The cured laminate was then demolded and cut using a water-saw (SYJ-400, Shenyang Kejing Instrument Co., Ltd) to prepare specimens for testing.

2.3 Characterization

The thickness was measured with a micrometer (NSCING, 0.001 mm). The morphology of the CNT film and individual CNTs were characterized by scanning electron microscopy (SEM, Hitachi S4800, Japan) and transmission electron microscopy (TEM, Tecnai G2 F20 S-TWIN, Japan). The pore size and specific surface area of the CNT films were measured through Brunauer-Emmett-Teller and Barret-Joyner-Halenda methods. The tensile modulus and strength of the film were tested on Instron 3350, and the composites were evaluated according to the GB/T 1445 standard using a universal testing machine (CMT5105, MTS Systems (China) Co., Ltd).

The glass transition temperature (Tg) and storage modulus of the composites were obtained through three-point-bending testing according to the ASTM D4065 standard, using a DMA tester (NETZSCH 242E, Germany). The sample for this testing was 50 mm long, 10 mm wide, and 2 mm thick, and the test was performed at a 1-Hz frequency with a ramp rate of 5°C/min from 30°C to 250°C. The temperature corresponding to the loss modulus peak was set as Tg.

The surface resistance was measured with a digital four-point probe tester (ST-2258C, Suzhou Jingge Electronic Co., Ltd). The electrical conductivity was measured with a commercial resistance meter (Victor VC890D). The sample size for these measurements was 35 mm long, 12.7 mm wide, and 1.2 mm thick. To reduce the contact resistance between the probes and specimen, the surfaces of the sample ends were polished using sandpaper and then painted with silver paste before testing. The in-plane electrical resistivity ρ was calculated by $\rho=R\times S/L$, where R is the tested resistance, S is the sectional area and L is the length. The electrical conductivity σ equaled $1/\rho$. All the measurements were performed on at least five specimens. The electrical power was obtained through formula $P=V\times I$, where V is the applied voltage and I is the current that passes through the CNT film [36, 37].

3 RESULTS AND DISCUSSION

3.1 CNT film and its resistive heating behavior

The thickness of CNT film used in this study is about 6.6 um. The average areal weight of the CNT film is about 4.99 g/m^2, much lighter than metal and carbon fiber sheet heaters. Fig. 1d shows the SEM image of the CNT film, illustrating that the long CNTs in the film are randomly distributed and entangled with each other, and the film is of high porosity. The pore diameter ranges from 20 to 40 nm, and the specific surface area is about 125.7 m^2/g, favorable for the resin to penetrate through the film during the curing process. The CNT diameter ranges from about 9 to 20 nm (Fig. 1e). The surface resistance is about 2.0 Ω/□. The tensile strength and modulus of CNT films were found to about 207.4± 46.2 MPa and 2.41±0.29 GPa, respectively. And the failure strain is about 30 %.

The thermal stability and electrothermal property of CNT film are also critical characteristics for their application in composites curing. The thermal stability of CNT film in both air and an inert atmosphere were thus investigated. The thermogravimetric analysis (TGA) curves shown in Fig. 3a demonstrate that the CNT film would not degrade within the composites' processing temperature range, which is below 200°C. The weight loss below 500°C in both atmospheres was attributed to the evaporation of H_2O and the oxidation of amorphous carbon. The sharp decrease in air atmosphere between 500°C and 700°C was attributed to the degradation of the graphite walls of CNTs. Fig. 3b shows the variation of temperature of a 200×200 mm^2 size CNT film during the increase of applied voltage. Initially, the film temperature increased slowly with the voltage, possibly because major of the generated heat was dissipated into the surrounding air. When the voltage was over about 6 V

(488.3 W/m^2), the film temperature correspondingly increased linearly. The cyclic electro-thermal behavior of 100×100 mm^2 CNT film in air was also investigated. In each cycle, a voltage of 8 V (3024.4 W/m^2) was applied to the CNT film for 10 min and then powered off for 8 min. The room temperature was around 25.8°C. As shown in Fig. 3c, the temperature increased rapidly when the electric was powered on, and then kept stable when the heat generation rate was equal to the heat dissipation rate. The enlarged electro-thermal curve in Fig. 3d shows that it took only 23 s to increase the film temperature from room temperature to 150°C. The infrared thermal image in Fig. 3d indicates the outstanding temperature uniformity of the heated CNT film. Due to the higher heat dissipation rate at the edge area, the temperature of these areas was lower than that of the middle. Upon power off, the film cooled down very quickly. This outstanding cyclic electrothermal performance makes CNT films desirable for long-term applications.

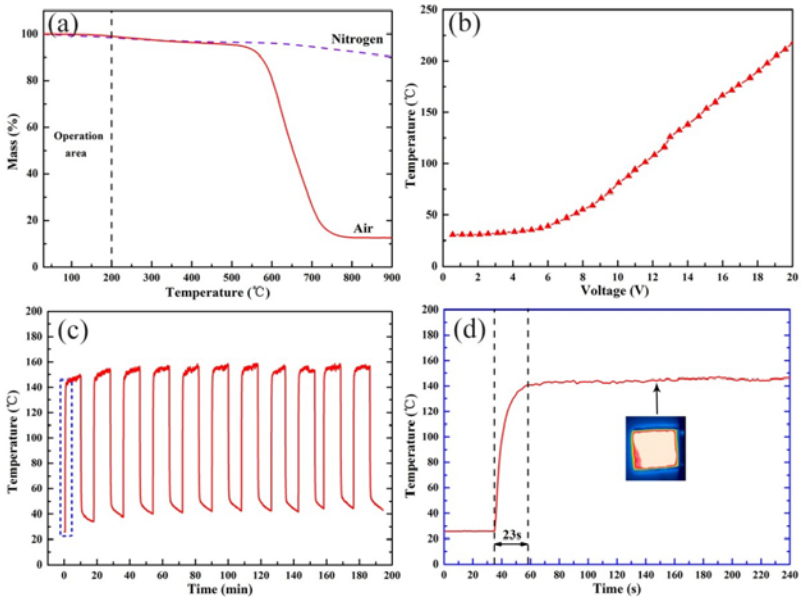

Fig.3. Thermal and electrothermal behavior of CNT films: (a) thermogravimetric curve, (b) variation of temperature with increasing voltage, (c) cyclic electrothermal behavior, and (d) its enlargement. (The insert picture was the thermal infrared image.)

3.2 In-situ curing of GFRP composite

The composites were cured by the resistive heating of CNT films. Fig. 4 demonstrates the relationships of surface temperature and input power during the curing process of sample-C composite consisting of one layer of CNT film and six layers of prepreg. As mentioned in the experimental section, two thermocouples were connected to the top and bottom surfaces of the composites to measure their temperature throughout the curing process. The prepreg supplier suggested an optimized curing process for the composite of 80°C for 0.5 h and then 120°C for 2 h. To realize this, the DC power supply connected to the CNT film through the copper wires was powered on to provide 11.5 V. The temperature of the composite was gradually increased and reached 80°C (Stage I) after 32 min. After 30 min holding at 80°C (Stage II), the DC power supply was increased to 13 V. The temperature reached about 120°C (Stage III) after 20 min and was kept stable for 2 h (Stage IV). The DC power supply was then powered off and the sample cooled down in air (Stage V). The increasing temperature rate of the CNT film during this curing process was much slower than that of the free CNT film, as a portion of the heat of the CNT film was transferred to the resin and mold. The temperature increasing rate could be increased by inserting a thermal insulating layer between the film and mold. The temperatures of both sides of the composite were almost the same, with the largest temperature difference being less than 5.9°C in Stage III, illustrating the outstanding temperature uniformity in the thickness direction of the composite.

The specific electric energy consumption of GFRP composites was about 1175.2 kJ, which was obtained by calculating the area under the in-situ input power curve. Thus, considering that the mass the composite sample is 151.48 g, the specific energy consumption is about 7.76 kJ/g, which is much lower than that of the oven curing process, discussed as follows.

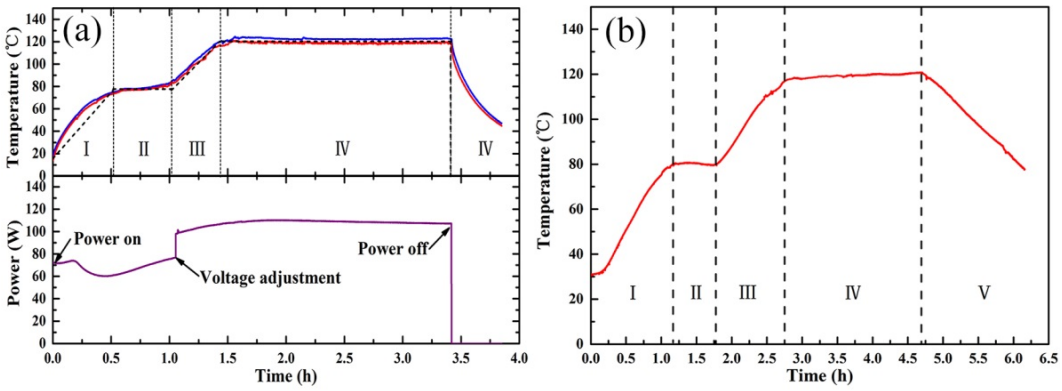

Fig.4. (a) Variation of the temperature (red line for top surface and blue line for bottom surface) and electric power during the CNT-film based curing process (Sample-C), (b) temperature variation during the oven curing process.

Fig. 4b shows the temperature cycle of the O-heating process. The process took much more time during the first and third stages than the C-heating process. As mentioned, the oven itself was first heated in the O-heating process. The air within it was then warmed up, and the composite part was finally heated by the hot air around it. It took about 70 min to heat the composite part from room temperature to 80°C, and about 60 min to heat it from 80°C to 120°C, while it took only 32 and 20 min respectively during the C-heating process. The electric energy consumption during the O-heating process was recorded by an ammeter, and the average energy consumed was found to be 55.34 kJ/g, which is over seven times that of the C-heating process.

3.3 Mechanical property of composite

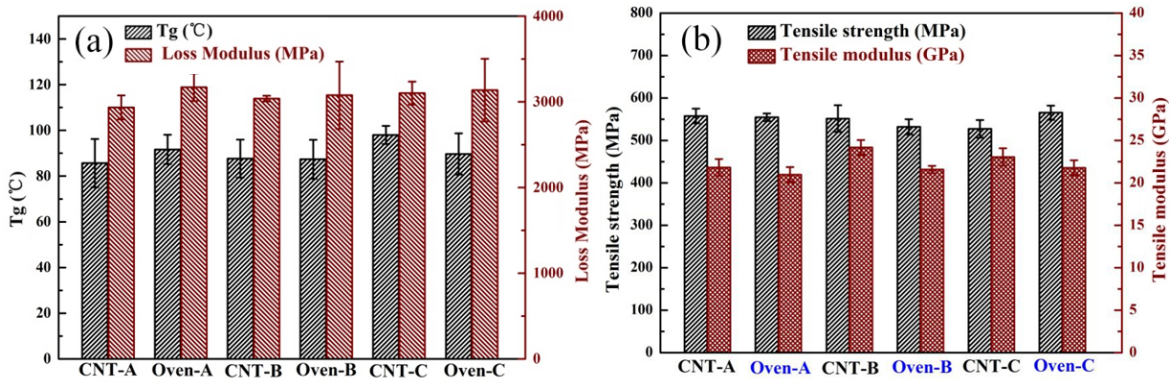

Fig.5: (a) T_g and loss modulus and (b) tensile strength and modulus of composites made from C-heating and O-heating curing processes.

The glass transition temperature (T_g) of the resin increases with the increase of cross-linking [38]. The higher T_g of composites means a higher curing degree [39]. The temperature corresponding to the peak of loss modulus was set as the T_g [40]. As Fig. 6a demonstrates, the T_g and loss modulus of the composites made from the CNT-heating and Oven-heating processes show no obvious distinction, also implying a similar curing degree of these two curing methods.

The tensile mechanical properties of the composites made from the different method are characterized and compared. The tensile strength and modulus of these composites are compared in Fig. 5b. It can be found that the average tensile strength of these four group composites is in the range

of 540-560 MPa, and the average tensile modulus is in the range of 22.2-23.9 GPa. This implies that the mechanical properties of the composites made by C-heating process are comparable with these of composites made by Oven-heating process.

3.4 Electrical property of composite and its deicing performance

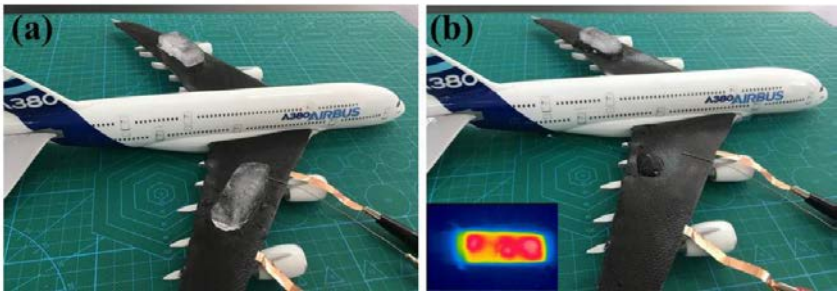

Fig.6: CNT film resistive heating based deicing of GFRP composites.

GFRP composites are now widely used in the wind blade industry, and deicing of the composite blades is one of the key challenges for their application in icing temperature areas. In addition to being used as the heating element for curing GFRP, electrically conductive CNT film can increase the conductivity of the GFRP composites. The electrical conductivity of pristine CNT film is about 4.38×10^4 S/m, much higher than that of films made by either solution infiltration [41, 42] or an array of rolling down CNTs [30, 31]. The resistive heating of the film can be used as the heating source for composite deicing. To demonstrate this, a 60×30 mm CNT film was placed in the middle of a six layer GRRP composites. The temperature and humidity of the experiment were 18.1°C and 70%, respectively. A cube of ice weighing about 10.5 g was placed on the surface of the wing, as shown in Fig. 6a. The model was placed in a freezer at -20°C for several hours until the ice completely froze onto the composites, and 5 V of electric power was then applied to the composites. The ice started to slide on the wing under its gravity after about 300 s, as shown in Fig. 6a. After 1300 s, the whole ice was melted into water (Fig. 6b). Considering that CNT film is much lighter than the metal meshes or foils, incorporating CNT films on the surface of fiber-reinforced composites provides an energy-saving and lightweighting strategy of de-icing without degrading the structural performance of composites.

4 CONCLUSION

High efficiency out-of-oven curing is one of the key technologies for realizing the low cost fabrication of high performance composites. In this study, we demonstrate a highly efficient, energy-saving out-of-oven curing process based on the resistive heating of a macroscale thin CNT film, made using the FCCVD method. Compared with other methods, the FCCVD method can be scaled up for the large-scale production of continuous CNT films. The film can be heated very quickly when connected to an electrical power source. In terms of its thickness, surface resistance, and temperature, the film has shown outstanding uniformity, making it a suitable heating element for composite curing.

By applying the CNT film either to the surface or in the middle of uncured GFRP composite, the hybrid composite can be heated and cured by the resistive heating of the CNT film. The efficiency of this new curing process has been evaluated, and the degree of cure, loss storage, and tensile properties of the composite resulting from the out-of-oven process have been found to be almost the same as composites cured through the traditional oven heating process. This new curing process also saves time and energy. The energy consumption is only one seventh that of the oven curing process, and thus provides a promising strategy for reducing the cost of composites. The CNT film also enriches the functionality of GFRP composites. A deicing test found that the resistive heating of the CNT film was able to remove the ice on the composite surface very quickly.

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