Highly aligned carbon nanotube web for structural health monitoring applications

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Abstract
Herein, we present a novel approach for damage sensing in adhesively bonded joints using a carbon nanotube single layer web (CNT-SLW) which marks a significant departure from the direct mixing approach of CNTs with epoxy adhesives. In this work, very thin highly aligned CNT-SLWs (thickness ~ 2.5 μm) with aerial density of 2.0 μg/cm² were horizontally drawn from vertically aligned CNTs forests and positioned over an adhesive film and placed between two non-conductive composite adherents, followed by the application of heat and pressure to form the joint. These joints were subjected to quasi-static loading to investigate the influences of CNT-SLW orientation on the damage sensing performance.

Keywords: Carbon nanotubes, Structural health monitoring, damage sensing and mechanical testing

1. Introduction

Over the past two decades, carbon nanotubes (CNTs) have attracted considerable attention due to their unique optical, mechanical, and electrothermal properties. [1]. Consequently, CNTs have emerged as promisingly attractive nanostructured materials for in-situ real time damage sensing in advanced lightweight non-metallic structures. Much of the initial work has been focused on integrating a randomly oriented percolating network of nanotubes in a polymer matrix to create conductive nanocomposites [2]. Interestingly, in composites, the nanoscale percolating network of CNTs enables the monitoring of crack initiation and propagation at a scale at which most non-destructive techniques are ineffective [3]. However, achieving adequate dispersion of CNTs in an epoxy/polymer matrix remains a challenge due to strong agglomeration tendencies of CNTs.

In this work, we report the use of a highly-aligned carbon nanotube single layer web (CNT-SLW), of negligible weight, to monitor the state of damage in joints. By their very nature, CNT webs exhibit a very uniform and dispersed network, mitigating the agglomeration challenges encountered when dispersing in bulk resin. These joints were subjected to quasi-static tests to investigate the influence of CNT-SLW orientation on the damage sensing performance.
2. EXPERIMENTAL

2.1. Materials

To prepare the adherent plies for single lap joints, exceptionally versatile hot melt, woven glass fibre epoxy prepregs (Gurit SE84LV) were used. Cytec FM300 film adhesive with polyester carrier was used in this work. FM300 is a high-temperature epoxy adhesive. Copper foil (Alfa Aesar, 0.025mm thick, annealed, uncoated, 99.8%) was used for the electrical buses. A very thin, highly-aligned CNT-SLW (thickness ~ 2.5 μm) with aerial density of 2.0 μg/cm² was used in this study. The fine continuous CNT webs, with controlled alignment, were horizontally drawn from a few micrograms of vertically aligned CNTs forests (each individual CNTs dia. ~10 nm and length~ 300 micron) grown in- house, on silicon substrates, using a CVD (Chemical vapor deposition) method.

2.2. CNT-SLW embedded joint preparation

To fabricate the bonded joints with embedded CNT-SLW, glass fiber laminates were manufactured and cut into rectangular strips of size 100 mm x 25 mm. The CNT-SLW (R// ~ 917±140Ω) was placed over one side of the FM300 film adhesive (25 x 25 mm), where the tacky surface facilitated the positioning of the CNT webs. The CNT-SLW web and film adhesive assembly was subsequently placed between two adherents with overlap area of 25 x 25 mm followed by the application of heat and pressure to form the joint. To measure the resistance during real time damage sensing, the CNT-SLW (specimens with transverse (⊥) and longitudinal (//) webs to the loading direction were prepared) were placed in such a way, that both ends of the web extended beyond the adhesively bonded joint area to permit connection to the electrical copper buses. The construction of the joint is shown in Figure 1.

![Figure 1](image_url). Schematic sketch of lap joint embedded with CNT-SLW+ adhesive film as sensor.

3. Characterization

A Zwick/Roell tensile testing machine (Model: Z100) with a 100kN load cell was used for quasi-static mechanical testing of the lap joints under displacement control. An Agilent 34450A digital multimeter was used to measure the resistance change across the joints. To quantify the CNT-SLW sensor response to deformation, ∆R/R₀ was plotted against displacement, where R₀ is the resistance at zero displacement and ∆R is the instantaneous change of resistance. To gain further insight into the CNT-SLW sensing mechanism, microstructural analysis of the fractured joints was performed using FESEM (JEOL: JSM 6500 F).
4. Results and Discussion

Figures 2a&b show the mechanical performance of the joint, through the interface shear stress, of representative specimens with three types of adhesively bonded joints. The equilibrium curves of stress versus displacement indicate that there was no appreciable change in the joints’ structural performance. In Figure 3, the capability of a CNT-SLW to detect damage initiation and growth in the joints is evaluated and its sensitivity shown to be significantly influenced by the orientation of the CNT-SLW with respect to the loading direction.

![Figure 2](image1.png)

**Figure 2.** (a&b) Stress vs displacement curve for CNT-SLW embedded the adhesively bonded joints.

![Figure 3](image2.png)

**Figure 3.** Relative change in electrical resistances of CNT-SLW embedded joint under applied load and the graphs presents the comparison of damage sensing with different orientation of web with respect to load direction

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The $\Delta R/R_o$ of CNT-SLW parallel (//) to the load direction registered the onset of damage initiation by a sudden step-wise increase in resistance, at an end displacement of 0.16 mm. The rapidly increasing changes in resistance indicate the formation of multiple micro-cracks (cumulative damage) coalescing into macro-scale damage features leading to catastrophic failure. In contrast, the CNT-SLW (⊥) to the load direction showed only 20 % increase in relative resistance ($\Delta R/R_o$) close to failure. Morphological studies were performed on the fractured surfaces of the joints after failure to gain further insight into the behaviour of CNT-SLW transverse (⊥) and parallel (//) to the load direction (Figure 4a&b). Figure 4a show the formation of transverse micro cracks to the load direction and the CNT-SLW parallel to the load can be seen to interact with these cracks. This explain the large increase in resistance from initial damage to failure. In contrast, the CNT-SLW (⊥) to the load direction (Figure 4b), seems to detached from the matrix during deformation and losing the alignment. Due to the high aspect ratio of CNTs, the broken contacts/or detached CNTs again formed an ‘electrical bridge’ with adjacent CNTs that resulted in very low increase in resistance during continuous deformation. This confirms very limited deformation or load transfer to CNTs when they are oriented transverse to applied load. It is also anticipated that flaws/cracks are aligned in the direction of CNT-SLW alignment. On the other hand, the significant shift of resistance for CNT-SLW parallel (//) to load direction confirms the formation of transverse cracks and therefore the damage is easily detected.

![Figure 4. (a) SEM image of fractured surface with CNT-SLW parallel (//) and (b) transverse (⊥) to load direction](image)

**Figure 4.** (a) SEM image of fractured surface with CNT-SLW parallel (//) and (b) transverse (⊥) to load direction

5. Conclusion

In this work, we presented a novel CNT-based sensor for damage detection in joints using a thin and highly aligned carbon nanotube single layer web (CNT-SLW). CNT-SLW damage sensors exhibited extremely high sensitivity to damage initiation and evolution. CNT-SLW may be effectively utilized for in-situ structural health monitoring of safety-critical joints.

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