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Di Leonardo, S., Nistal, A., Catalanotti, G., Hawkins, S. C., & Falzon, B. G. (2019). Mode I interlaminar fracture toughness of thin-ply laminates with CNT webs at the crack interface. *Composite Structures*, 225, [111178]. <https://doi.org/10.1016/j.compstruct.2019.111178>

Published in:
Composite Structures

Document Version:
Peer reviewed version

Queen's University Belfast - Research Portal:
[Link to publication record in Queen's University Belfast Research Portal](#)

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Accepted Manuscript

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PII: S0263-8223(18)34582-3
DOI: <https://doi.org/10.1016/j.compstruct.2019.111178>
Article Number: 111178
Reference: COST 111178

To appear in: *Composite Structures*

Received Date: 18 December 2018
Revised Date: 10 April 2019
Accepted Date: 18 June 2019

Please cite this article as: Di Leonardo, S., Nistal, A., Catalanotti, G., Hawkins, S.C., Falzon, B.G., Mode I interlaminar fracture toughness of thin-ply laminates with CNT webs at the crack interface, *Composite Structures* (2019), doi: <https://doi.org/10.1016/j.compstruct.2019.111178>

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Mode I interlaminar fracture toughness of thin-ply laminates with CNT webs at the crack interface

Sofia Di Leonardo^a, Andrés Nistal^b, Giuseppe Catalanotti^{a,*}, Stephen C. Hawkins^{a,c}, Brian G. Falzon^{a,†}

^a Advanced Composites Research Group (ACRG), School of Mechanical and Aerospace Engineering, Queen's University Belfast, Belfast BT9 5AH, UK

^b Institute for Materials Discovery, University College London, Roberts Building, London WC1E 7JE, UK

^c Department of Materials Science and Engineering, Monash University, Clayton, Victoria 3800, Australia

Abstract

Pristine and functionalized carbon nanotube (CNT) webs were introduced at the interlaminar region of two different thin-ply carbon fibre reinforced polymer systems (NTPT 402 prepreg, and TeXtreme/SE84LV). The Mode I interlaminar fracture toughness (ILFT) for the NTPT 402 material was substantially lower. In contrast when SE84LV resin was used, the resistance curves were virtually unchanged and with no depreciation in the steady state value of the ILFT. This demonstrates that a careful choice of the resin is essential to permit the embedding of CNT webs in thin-ply laminates without compromising structural performance and opens a new way towards the development of multifunctional thin-ply structures.

Keywords: A. Thin-ply laminates; B. Carbon nanotube (CNT) webs; C. Interlaminar fracture toughness

1. Introduction

Carbon nanotube (CNT) webs have recently been proposed as a promising route to achieving multifunctional carbon fibre reinforced polymer (CFRP) laminates [1–3]. The highly conductive CNT webs are drawn directly from specially grown CNT forests produced by chemical vapour deposition (CVD) [4]. The webs can be interleaved with plies of carbon fibre (CF) to tune the thermal and electrical properties of the laminate with negligible weight penalty.

Despite their negligible weight and thickness, the introduction of CNT webs may affect the interlaminar fracture toughness (ILFT) behaviour. In standardised mode I ILFT tests (ASTM D5528 [5]), using a double cantilever beam (DCB) made from unidirectional composite plies, webs may hinder the nesting between the carbon fibres of different plies, reducing the bridging effect and consequently the steady-state ILFT. Moreover, it has been observed that the interaction of CNTs with some epoxy resins has a major influence on ILFT. Bhanushali and Bradford [6], working with woven glass fibre reinforced polymer (GFRP) composites, found that ILFT was either marginally degraded or enhanced, depending on the orientation and number of layers of the CNT webs.

Chitwan et al. [7] observed a substantial reduction (- 65%) of the mode I ILFT when using CNT webs with Solvay 977-2 epoxy resin composite system while a small *increase* was observed with the Gurit SE84LV resin. Care,

* Corresponding author. Tel.: +44 (0)28 9097 4502.

E-mail address: g.catalanotti@qub.ac.uk

† Corresponding author. Tel.: +44 (0)28 9097 5640.

E-mail address: b.falzon@qub.ac.uk

therefore, needs to be taken when selecting the resin, because the reduction of the fracture toughness can be considerable and unacceptable for structural applications.

Nistal et al [8,9] demonstrated a way of improving the interaction between the resin and CNT web without affecting the orientation or the microstructure of the CNTs, important in underpinning the development of CNT web-based multifunctionality. This was achieved through a single step gas-phase amino functionalisation which enhanced ILFT of conventional SE84LV CFRP by 24% with respect to pristine CNT webs.

These preliminary investigations [6,7] have considered the use of CNT webs in conventional composite laminates. However it is important to study how the use of CNT webs may affect the ILFT of thin-ply laminates because, by combining these two non-conventional reinforcements, new opportunities may arise, and risks identified, in the development of multifunctional composite structures [10].

Conventional composite laminates have a ply thickness typically between 100 and 200 μm , while thin-ply can be as low as 15 μm . The reduction of the ply thickness results in the suppression of all subcritical damage mechanisms associated with the matrix, such as matrix cracking and delamination [11–14]. Although a reduced ply thickness may also result in a reduction in ILFT [15] (due to a reduction of the bridging effect caused by less waviness of the fibre in a thin ply), it renders the laminate less prone to both matrix cracking induced delamination (MCID) and free edge delamination (FED) [16]. This is because the lower ply thickness causes a lower interlaminar stress concentration at the adjacent interface, delaying the premature matrix cracking and delamination.

A combination of the conventional CF ply, thin CF ply and CNT web in an epoxy matrix offers the potential to create a truly hierarchical composite structure which optimises the advantages of each component, introduces additional through-thickness characteristics such as thermal and electrical conductivity and mitigates any weaknesses [13]. Such flexibility also underpins the development of additional functionalities such as structural health monitoring, anti-icing/de-icing and lightning strike protection within the composite structure [10].

We now report a study of the ILFT when CNT webs are combined with thin-ply CFRP and on progress towards the development of practical multifunctional hierarchical systems. Two different thin-ply materials (a UD prepreg and UD dry fibre), and both pristine and amino-functionalized CNT webs are utilized to explore the effect of resin-CNT interaction and sample fabrication.

2. Materials and methods

2.1. Materials

Two thin-ply material systems were used in this work: i) NTPT 402, a UD prepreg (MR70 CF) of 300 mm width provided by North Thin Ply Technology, with a nominal thickness of 15 μm , a CF areal weight of 15 gsm, and a resin content of 38%wt; and ii) UTS50S UD, a ‘spread-tow-tape’ of dry carbon fibre carried on a backing strip, supplied by TeXtreme, with a CF areal weight of 32 gsm and a width of 25 mm. Thin plies and CNT webs were only introduced in the middle plane of the laminate (crack plane), while for the outer parts the Gurit HMC SE84LV unidirectional prepreg, with a nominal thickness of 0.35 mm, was used. This also provided the resin to infuse the dry TeXtreme ply and included CNT web, whereas the NTPT 402 prepreg product provided the resin to infuse the CNT web included in those samples.

Directly spinnable CNTs were produced by a CVD process as reported elsewhere [15]. Highly aligned CNT forests were grown on a silicon wafer bearing a catalytic iron layer. Using a gas mixture of acetylene and hydrogen diluted in helium, a temperature of 690 °C and a dwell time of only 12 minutes, 300 µm long CNTs with an average of 6 walls and 10 nm diameter were obtained. CNTs were directly drawn and wound (10 layers) onto aluminium frames to produce 70 x 70 mm² webs with an aerial density of 0.2 g/m². Finally, the CNT webs were stored in a desiccator cabinet (relative humidity ≤ 10 %) until used.

Amino functionalized CNT webs were produced in a gas-phase reaction following a procedure recently developed and reported. In summary, a 10-layer CNT web is held under static vacuum (-100 kPa) in the presence of 50 mg of ethylenediamine (≥ 99.5%; bp = 118 °C; Sigma-Aldrich). The system was heated to 180 °C and dwelled for 2 h. This procedure was found to produce a high concentration of chemisorbed (i.e. non-covalently bound) amine without disrupting the delicate highly aligned CNT web and hence is very suitable for studying advanced functionalities as noted above.

2.2. Specimen preparation

Composite panels were manufactured according to several different layup patterns depending on the material used and were always symmetric about the mid- (i.e. crack) plane. Thus, from the outer face of the panel to the crack plane, 6 plies of Gurit HMC SE84LV and 3 plies of thin-ply prepreg NTPT *or* 1 ply of dry thin-ply TeXtreme were used, with the remainder of the layup reflecting symmetry (see Fig. 1). For crack initiation, a polytetrafluoroethylene (PTFE) film (13 µm thick) was included in the crack plane during layup, and, for experiments, a single pristine or amino-treated 10 layer CNT web was also included (Fig 1). The DCB specimens were manufactured as per the ASTM D5528 [5] standard requirements. The CNT webs were laid down in the transverse direction (90°), perpendicular to the fibre and parallel to the crack front. It has been observed that, when placed in parallel to the carbon fibres, CNT web contributes to their interlocking and hence facilitate fibre bridging; in contrast, when the CNT web is placed perpendicularly to the fibre, it inhibits or prevents the interlocking thereby reducing also fibre bridging [7]. It should be noted however that fibre bridging is an artificial toughening mechanism that occurs when testing DCB specimens (since plies at the crack interface are aligned along the 0° direction) and that in real structures it does not normally occur since delamination tends to originate and propagate between two plies with different orientations. Therefore, laying down the CNT webs transverse to the crack direction ensures that no bridging occurs and allows for a conservative value of the ILFT to be obtained. However, it is recognised that there is an interest in determining the effect of the CNT-web orientation, number of web layers, and layup configuration, and this work is currently being undertaken.

●	6 ply Gurit HMC SE84LV
●	3 ply NTPT 402 prepreg or 1 ply TeXtreme dry
●	10 layer CNT web
●	3 ply NTPT 402 prepreg or 1 ply TeXtreme dry
●	6 ply Gurit HMC SE84LV

Figure 1: Layup of the specimens investigated.

Preliminary experiments with NTPT 402 prepreg indicated a degradation in ILFT performance in the presence of CNT web, raising the possibility that the 402 resin was failing to penetrate the web effectively. Therefore a set of samples was also prepared using acetone to improve the mobility of the 402 resin and ensure the CNT web was fully wetted and embedded. This methodology has been tested on other, high viscosity, resins and found to be satisfactory. This was not necessary for the SE84LV (ie low viscosity) resin. Thus each 10 layer CNT web was placed dry onto one prepreg panel still on its backing sheet and exposed to an acetone-saturated atmosphere for 1 minute. Absorption of acetone vapour rapidly expanded and diluted the top-most film of resin, causing it to wet and incorporate the CNT web. The panel was removed from the acetone environment and placed under dynamic vacuum for 30 minutes to eliminate the absorbed solvent vapour and the CNT web pre-preg was then incorporated into the layup as usual.

Assembled composite panels were placed on a mould-release treated aluminium tool, covered with a sheet of release film (non-perforated), vacuum bagged and debulked (-1 atm) for 1 h and finally cured in an autoclave. The recommended cure cycle used is illustrated (Fig 2a). An extended cycle was tested (Fig 2b) to provide more time (45 min) at the temperature of minimum viscosity (80 °C), and vacuum was maintained throughout in order to facilitate CNT web impregnation.

Figure 2: (a) recommended cure cycle; (b) extended cycle.

Table 1. Code label used to identify the samples.

Variable	Label	Description
Thin ply / Resin	T	TeXtreme / SE84LV
	N	NTPT 402
Process	R	Recommended cycle (Fig. 2a)
	X	Extended cycle (Fig. 2b)
CNT web	C	Control specimen without CNT web
	P	Pristine CNT web
	A	Acetone treated pristine CNT web
	F	Functionalized CNT web

After curing, specimens of nominal thickness (h) 3.5 mm, length (L) 130 mm, width (b) 20 mm, and initial crack length (a_0) 50 mm according to the ASTM D5528-13 DCB standard test method [5] (Fig. 3), were cut to size using a diamond saw. Hinges were attached, using a bicomponent epoxy adhesive, to each arm of the specimen and one specimen edge painted with a matt white paint and a graduated scale to allow monitoring of the crack length during the test.

The different configurations of process, treatment and material tested are identified using the code labels presented in Table 1. As an example, NAR indicates specimens with NTPT 402 prepreg and resin at the crack interface with pristine CNT web treated with Acetone to improve resin penetration, and processed by the Recommended cure cycle (Fig 2a). Correspondingly, TFR indicates specimens that in the thin-ply region are made with the TeXtreme fibre and SE84LV resin (from the Gurit prepreg), with Functionalized CNT webs, cured with the Recommended curing cycle.

2.3. Experiments

Double Cantilever Beam (DCB) tests were performed to measure the Mode I ILFT [5].

As noted, all specimens have a pre-crack of 50 mm (the distance between the crack tip and the load introduction point). However this value is affected by the specifics of the manufacturing of the laminate and positioning of the hinges. Using Mohr's analogy, a corrected value of the initial crack length, a_0 , was obtained, as:

$$a_0 = \sqrt[3]{\frac{3\delta_i EI}{2P_i}} \quad (1)$$

where E is the Young's modulus of the laminate (approximated as the Young's modulus of the Gurit laminate since the contribution of the thin plies and CNT webs may be neglected), I is the moment of inertia of one arm, $I = b(h/2)^3/12$, with b and h the width and the thickness respectively of the sample (Fig. 3). δ_i and P_i are the i -th displacement and load, measured during the loading of the laminate prior to crack propagation, and estimated from the initial linear part of the load vs. displacement curve. Tests were conducted on an Instron 5564 universal testing machine equipped with a 2 kN load cell, at a crosshead speed of 1 mm/min.

Figure 3: Double Cantilever Beam specimen.

A USB 2.0 Digital Microscope camera synchronized with the universal testing machine, was used to record crack propagation at three second intervals and the images post-analysed. The fracture toughness for the initial and steady state crack propagation, G_c^i and G_c^{ss} , respectively, was estimated from Modified Beam Theory:

$$G_c = \frac{3P\delta F}{2b(a + |\Delta|)} \quad (4)$$

where a is the measured crack length, Δ is the correction coefficient of a and F is the correction coefficient of δ [5].

2.4. Post-test analyses

A Hitachi FlexSEM 1000 Scanning Electron Microscope was used to observe the morphology of the fractured surfaces, and of the cross section of the specimens.

3. Results and discussion

3.1. Effect of CNT webs on NTPT 402 prepreg

Resistance curves of the calculated interlaminar fracture toughness (G_c) as a function of the crack length (Δa), for the NTPT 402 thin-ply specimens with pristine CNT webs and recommended processing exhibit (Fig. 4) a *reduction* in respectively the fracture initiation (G_c^i) and steady state (G_c^{ss}) values of 44% and 68%.

Figure 4: Resistance curves $G_c(\Delta a)$ of the NRP and NRC samples.

The interlaminar fracture toughness when a CNT web is incorporated has been reported previously by Bhanushali and Bradford [6] to vary significantly and both positively and negatively with the number of web layers used and their orientation in Glass FRP. A dramatic effect on ILFT was recently reported by Chitwan et al [7] who observed a 65% decline when using the aerospace grade toughened epoxy 977-2 from Solvay, and was independent of the amount of webs used.

In order to ensure that the loss in fracture toughness was not a result of failure to fully embed the nanotubes in the resin, the curing cycle of the composite was modified to extend the period of low viscosity (Figure 2b, samples NXP) or else to temporarily reduce the viscosity of the surface resin using acetone vapour (samples NRA).

All configurations showed a reduction of 44% and 67% for the initiation and steady state value of the interlaminar fracture toughness, respectively (Fig. 5). No change was noted for the different sample types tested, indicating that the solvent treatment itself is not deleterious, and that poor contact of the resin with the CNT web is not the cause of the degradation in performance. This result was further confirmed by similarity of the SEM images of untreated (NRP) and treated (NXP, NRA) samples which showed no substantial difference as a result of these treatments.

Figure 5: Summary of the experimental results obtained for the NTPT 402 thin-ply prepreg.

The dominant cause of weakening for the Solvay 977-2 resin in the presence of CNT web was identified by Chitwan et al [7] as the poor interfacial strength between the CNTs and the particular epoxy formulation. The precise nature of this interface failure was not clarified, however it is evident that in NTPT 402, another example of interface failure is observed. The planar web morphology and nano-scale size and spacing of the CNTs ensures an effectively uninterrupted fracture plane through the material, as seen in the SEM of the fracture surfaces (Fig. 6), where the scalloped morphology (as observed in Fig. 6(a)) is largely or completely eliminated by the planar structure of the CNT-web (Fig. 6(b-d)).

Figure 6: Fracture surfaces for the (a) NRC, (b) NRP, (c) NXP, and (d) NRA samples.

Figure 8: Summary of the results obtained for the TeXtreme/SE84LV.

From a comparison of the control specimens, NRC and TRC, the ILFT of the latter is substantially lower. This is reflected in the different fracture morphology, with the TRC (Fig. 9(a)) showing a smoother fracture surface and evidently poorer CF/resin interaction than the NRC (Fig. 6(a)). The NTPT 402 and the SE84LV are different toughened resins, for which limited data is available, therefore limiting the interpretation of the fracture behaviour.

The fracture toughness of the NTPT specimens containing CNT-web is substantially lower than the control specimens and this is attributed to a poor resin/CNT interfacial strength and the planar web morphology. A similar fracture behaviour was previously reported for the Solvay 977-2 toughened resin [7]. The roughness of the NRC specimens (Fig. 6(a)) arises from matrix fracture and carbon fibre-matrix debonding, and so the fracture surfaces exhibit a fibre-scale characteristic size. The planar CNT-web, by having a weak resin interfacial strength, promotes the onset and propagation of a planar crack through the structure of the web. The fracture morphology therefore reflects the scale and structure of the CNT-web. Although the CNT modified specimens appear rougher (Fig. 6(b-d)), the roughness is of a finer scale than observed for the control specimens (Fig. 6(a)).

The lack of negative effect due to the insertion of CNT-web into the SE84LV resin indicates the resin/CNT interfacial strength is at least as strong as the bulk resin and perhaps also the carbon fibre/resin interface. As observed in Fig. 9(b), the fracture plane crosses, but does not substantially follow, the CNT-web plane. As a result, the macroscopic fracture morphology of the control (Fig. 9(a)) and CNT-web modified (Fig. 9(b)) specimens is similar.

Figure 9: Fracture surfaces for the (a) TRC and (b) TRP samples.

3.3. Discussion

The experimental results obtained for the NTPT 402 resin clearly show that an incompatibility exists between CNT web and the NTPT 402 resin, as was previously observed for the Solvay 977-2 resin [7]. This effect is not intrinsic to CNT web but rather to the particular resin formulation. Scope exists to modify the CNT web, for example by amino-functionalization, to overcome this detriment, indeed it is essential to understand the fundamental causes of this effect.

For the TeXtreme/SE84LV specimens, experimental results obtained in a previous investigation have measured a steady state value of the interlaminar fracture toughness of 0.35 N/mm for conventional laminates [7]. Pristine thin-ply tested in this investigation showed a steady state value of the interlaminar fracture toughness of 0.24 N/mm, 31% lower than reported for the conventional laminate. The reduction in interlaminar fracture toughness with decreasing ply thickness is expected (because decreasing the ply thickness decreases the waviness of the fibre and therefore the bridging), and has been reported in other investigations [15]. The introduction of pristine CNT web for this thin-ply material system does not cause any reduction in the interlaminar fracture toughness. This demonstrates that the CNT web can be utilised as an interfacing ‘nano-ply’ within the thin-ply design to improve thermoelectric properties without degrading structural properties. It should be highlighted that, in the case of thin-ply laminates, the number of possible interfaces increases, and consequently the quantity of CNT webs that could be embedded in the laminate.

Finally, it should be observed that the use of functionalized CNT webs for this case does not produce a toughening effect such as has been found for conventional laminates [8]. The Gurit HMC SE84LV system has a small though clear excess of resin which, when combined with amine-treated CNT web alone, is ample to interact with the cross-linker to enhance the CNT-resin interaction. However, the inclusion of the dry thin ply plus the CNT web is thought sufficient to exhaust this excess and limit the extent of any cross-linking interaction. The other effect that is thought to be relevant is that the amine is only chemisorbed onto the CNT surface and the temperature at which it reacts with the SE84LV resin in the range of only 40 to 90 °C [8]. Insertion of the functionalized web in direct contact with the SE84LV would capture the amine. However insertion between dry thin ply, which depends on heating and vacuum to move the SE84LV resin into it and the included CNT web, would result in significant diffusion and reaction of the

amine away from the CNT surface and hence loss of effect. This should be remedied by the use of a Thin-Ply prepreg bearing a compatible resin such as SE84LV.

4. Conclusions

Experiments were conducted on two different thin-ply material systems with the aim of assessing the effect that the introduction of embedded CNT web has on the mode I interlaminar fracture toughness. Experiments have shown that the introduction of CNT webs results in a decrease in ILFT for the NTPT 402 prepreg of about 44% and 68%, for the initial and steady state values, respectively. This degradation of performance is attributable to an incompatibility between the CNT web and the particular resin formulation. Scope exists to improve the performance of CNT web, for example by amino-functionalization. Indeed it is essential to understand the fundamental causes of this effect if the potential offered by CNTs and other nanomaterials to improve the physical and electro-thermal properties of modern composites is to be realized. In contrast, no depreciation of the interlaminar fracture toughness was observed for the SE84LV specimens. This result is particularly important because it demonstrates hierarchical thin-ply/CNT web structures can be obtained without compromising their structural performance.

Acknowledgements

The authors would like to acknowledge the financial support from the EPSRC project MACANTA—Multifunctional hierarchical advanced composite aerostructures utilising the combined properties of different carbon nanotube (CNT) assemblies (EP/N007190/1). The first and third authors would also like to acknowledge Prof. Giuseppe Pitarresi of the University of Palermo, and the financial support provided by the Collegio Universitario ARCES through the Erasmus+ mobility grant.

Data availability

Datasets related to this article may be found at <http://dx.doi.org/10.17632/pfr55wdn72.1>, an open source online data repository hosted at Mendeley Data.

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