IN-SITU MONITORING OF INTERLAMINAR SHEAR DAMAGE IN CARBON FIBRE COMPOSITES

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ABSTRACT

The in-situ damage sensing of carbon/epoxy composites during interlaminar shear testing is investigated. Next to direct monitoring of woven fabric carbon/epoxy reference laminates, the introduction of carbon nanotubes (CNTs) onto these carbon fibre fabrics via a spray coating technique for damage sensing is evaluated. We observed very different sensing behaviour compared to previous studies, which is believed to be more useful for real applications. Through-thickness measurements showed for both reference and CNT modified specimens a continuous increase in electrical resistivity, due to reduced contact areas and conductive pathways. The effect of the introduced CNT network at the interfacial region is also been compared and analysed.

1. INTRODUCTION

The use of carbon fibre reinforced plastics (CFRPs) has kept increasing in the last few years, especially in aerospace, automotive and wind industries due to requirements of weight reduction and durability. However, compared to outstanding in-plane fibre dominated properties, out-of-plane properties such as interlaminar shear strength and fracture toughness are often limiting their performance. More importantly, the various non-visible failure modes such as matrix cracking and delamination also often require monitoring throughout the usage of composite components to fulfil safety requirements.

Interlaminar shear strength (ILSS) is often regarded as a key property and indicator for a composites' performance, particularly in relation to interfacial adhesion. Hence it is important and necessary to monitor the interlaminar shear failure of CFRPs. Due to the intrinsic conductive nature of carbon fibres (CF), previous research studies [1-4] have demonstrated the self-sensing capability of CFRPs under tensile and flexural loadings by electrical resistance measurements, although very limited works can be found on damage sensing of interlaminar shear failure [5]. In the case of insulating glass fibre composites, conductive fillers such as carbon nanotubes (CNTs) have been introduced into the polymer matrix to provide a percolating network for damage sensing in nano-engineered composites under shear loading conditions [6-7]. For instance, Thostenson and Chou [7] observed that the electrical resistivity increased several orders at the point of shear failure, indicating abrupt delamination without progressive damage accumulation. In fact, all previously reported damage sensing studies showed no obvious changes in resistivity until final rupture of the specimen [1-6], which provides little inside nor useful health monitoring information for such shear loading conditions.

To best of the authors' knowledge, this paper for the first time demonstrates the capability to monitor damage progression in CFRP and CF/CNT nano-engineered composites during interlaminar shear testing. An innovative spray coating technique is used to apply the CNTs directly at interfacial regions in carbon fibre composites for ILSS damage sensing. This spray coating technique overcomes dispersion issues of nanoparticles [8, 9] and deposits CNT percolated networks at the carbon fibre surface where it can be utilized as an integrated sensor to detect damage during the test. The effect of these CNTs is evaluated by comparing sensing signals from carbon fibre reference specimens and CNT modified specimens.

2. EXPERIMENTAL

2.1 Materials

The composite system employed consists of high strength 2 × 2 twill carbon fibre fabrics, with an areal weight of 286 g/m^2, and RTM6-2 two component aerospace grade epoxy resin, both from Hexcel®. Carbon nanotubes are multiwalled and supplied by Nanocyl S.A. (Product No. NC7000).

2.2 Sample preparation

Ten plies of carbon fibre fabrics were used to produce the panels. In case of the CNT modified panels, nine out of ten plies were spray coated with CNTs. The amount of CNTs sprayed on each ply was 5 mg. The concentration of 0.08 wt.% was calculated based on total amount of CNTs deposited, ignoring the limited amount of loss during the spraying process. Detailed spray coating procedures can be found elsewhere [10]. Standard vacuum assisted resin transfer moulding (VARTM) process has been applied, with a curing cycle of 140 °C for 1.5 hrs followed by 2 hrs post-curing at 180 °C. The mould was under vacuum (-1 bar) throughout, with no evidence of CNT migration with resin flow as the resin remained clear at outlet. The cured panels were cut into specimen with the dimension of 40 × 13 × 3 mm and polished on both sides before testing.

2.3 Short beam shear testing

For ILSS evaluation, short beam strength (SBS) tests were performed in accordance with ASTM D2344, using an Instron 5566 universal testing machine with a crosshead speed of 1 mm/min. An illustration of testing set-up can be found in Fig. 1.

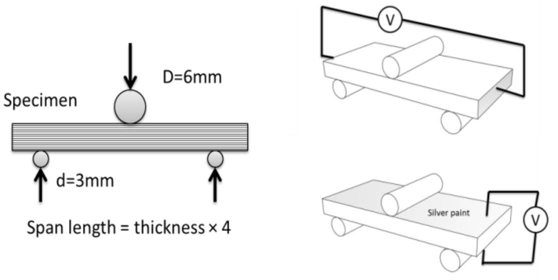


Fig. 1: Illustration of in-situ damage sensing setup for SBS test

2.4 In-situ damage sensing

For ILSS specimen, both in-plane and through-thickness electrical measurements were conducted for the sensing tests, as shown in Fig. 1. Thin copper wire was positioned at both ends of the specimen for in-plane measurements, while for through-thickness tests, conductive silver paint (from RS Components) was applied on top and bottom surface of the specimen, while the copper wire was attached using silver-loaded epoxy adhesive. A digital multi-meter (Agilent 34401A) was used to measure the volumetric resistance of each specimen using a two probe direct current (DC) measurement set-up.

2.5 Fractography

The fracture surface of the tested specimen was examined by scanning electron microscopy (SEM) using a FEI Inspect-F. Images at different magnifications were taken.

3. RESULTS AND DISCUSSION

3.1 Morphology

The cross-sections of tested specimen were examined using SEM (Fig. 2). A clear interlaminar shear failure mode was observed, together with evidence of translaminar cracking between fabric plies. The fracture surface of the CNT modified specimen shows more morphological features rather than the typical smooth fracture surface of neat epoxy based specimen. No obvious CNT agglomerations were observed in these fracture surfaces.

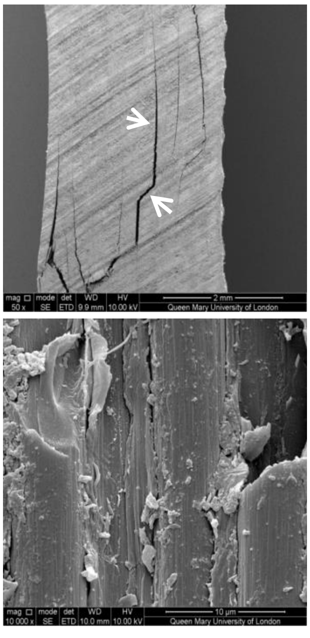


Fig. 2: SEM images of interlaminar shear failure after SBS tests of CNT modified CFRP. Interlaminar shear failure mode was observed, together with some evidence of translaminar cracking between fabric plies as indicated by the arrows

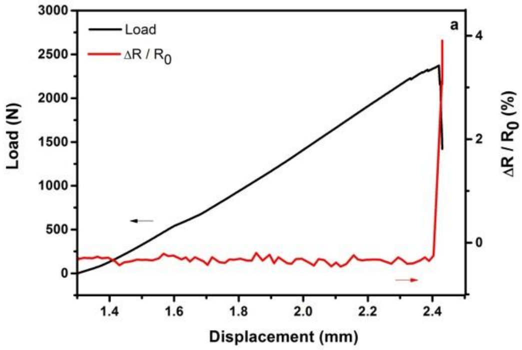
3.2 Interlaminar shear strength

For all specimens, linear mechanical behaviour is observed with a sudden force drop in the load-deflection curves at the end of the test, confirming that clear interlaminar shear failure is taking place rather than a combination of failure modes. The average ILSS values calculated from the load-displacement curves for reference and CNT deposited specimen are 51.7 (± 1.8) MPa and 48.8 (± 2.2) MPa, respectively. As these values are within the experimental error, no obvious effect of the CNT coating on the ILSS of the carbon/epoxy composites was found. The two main reasons for this are believed to be: i) the extremely low CNT concentration (0.08 wt.%) within the composites; and ii) the fact that the deposited CNTs are mainly deposited at ply level rather than individual fibre level. As interlaminar shear failure may occur at fibre level, CNT modification at ply level may therefore not lead to significant improvements in ILSS. Therefore the obtained mechanical reinforcement effects of the CNTs here are less obvious as for our previous mode-I interlaminar fracture toughness studies where delamination occurred mainly at the ply level [10].

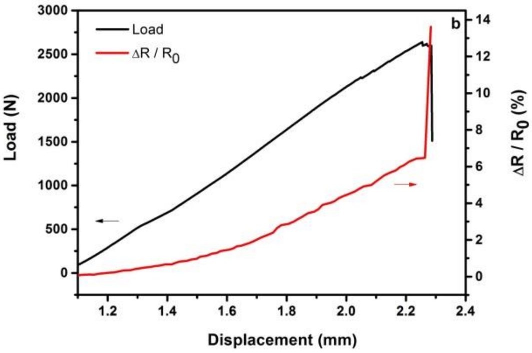
3.3 In-situ damage sensing

The *in-situ* damage sensing results for reference specimens are presented in Fig. 3, for both in-plane (a) and through-thickness (b) measurements. As mentioned earlier, the electrical conducting nature of carbon fibres allows the reference specimen to be directly used for such an electrical damage sensing method. However, due to the insulating layers of epoxy resin between the carbon fibre fabric plies, the conductivity is completely relying on interconnects between individual carbon fibres or tows. Therefore, a direct sensing method based on carbon fibre without CNTs to improve network formation may lead to more scattered signals and less reliable [10]. CNT modified specimen may improve small-scale tunnelling effects, leading to an improvement in sensitivity of the electrical damage sensing method.

For the in-plane measurement, the electrical sensing signal of the unmodified CFRP laminate showed some unstable readings during SBS testing, with a slight reduction in resistance being even observed with increased loading. This small reduction was attributed to the deformation of the specimen, which may result from a reduction in gap distance between conductive carbon fibres as a result of the compressive load applied in the three-point bending experiment. This was found consistent with other research works [5- 7, 11]. No obvious resistance change was observed with increasing load, due to the dominating conductive character of the carbon fibre phase. At failure the sensing signal shows a sudden increase in resistivity, which is due to a break-down of interlaminar conductive pathways as well as fibre breakage. However, once a composite structure has reached the fibre breakage stage, the component is too near the end of its life-time for this kind of damage sensing to be useful.



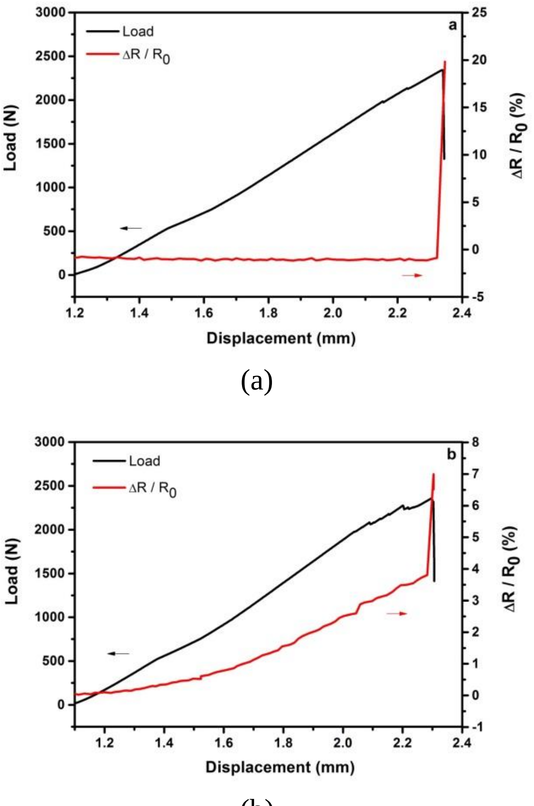
(a)



(b)

Fig. 3: Typical load-displacement curves together with in-situ damage sensing results for CFRP reference specimen for: (a) in-plane electrical measurement; (b) through-thickness electrical measurement. With increasing load applied to the specimen, the electrical sensing signals remained fairly stable until the final failure for in-plane measurement, while for through-thickness measurements, a progressive sensing signal was obtained to indicate the internal damaged

In the case of through-thickness measurements, unlike previously reported results [5-6] where the sensing signal remained unchanged until final failure, here the measured electrical sensing signals increased continuously with load. At final failure of the specimen, also a sudden jump is observed in resistivity, however, this sudden jump is preceded by progressive damage accumulation, making this a more interesting experiment from a structural health monitoring (SHM) point of view. It is also worth noting that the observed relative resistivity change was much greater for the through-thickness measurements (14%) than for the in-plane measurements (4%) (see Fig. 3a and 3b).



(a)

(b)

Fig. 4: Typical load-displacement curves and in-situ damage sensing results for CNT coated CFRP specimen for: (a) in-plane electrical measurement; (b) through-thickness electrical measurement. Similar to CFRP reference specimen, progressive electrical sensing signals were obtained from through-thickness measurements

The sensing results of CNT spray-coated specimen are presented in Fig. 4, for both in-plane (a) and through-thickness (b) electrical measurements. Due to the extremely limited amount of CNTs introduced in the laminates, no obvious difference in electrical resistance was observed between reference and CNT-modified specimens. The electrical resistance measured in-plane for reference and CNT-modified laminates are 0.25 and 0.24 Ohm, respectively, while out-of-plane values are 1.04 and 0.97 Ohm, respectively. Typical load-displacement curves for the ILSS tests are shown in the graphs, together with normalized resistance change values as sensing signals. For in-plane measurements, the sensing signals remain constant without any obvious change until final failure of the specimen where a sudden jump in electrical resistance was found. Similar to the reference CFRP specimens, this sudden jump was mainly due to breakage of conductive carbon fibres, which dominate the overall electrical properties. In the case of through-thickness measurements, the sensing signal continuously increased with loading similar to the reference specimen, with another sudden increment being observed at final failure of the specimen.

For both reference and CNT deposited CFRP specimen, the in-plane sensing signals remained fairly unaffected with increasing load until final failure of the specimen, while through-thickness sensing signals progressively changed with increasing loading. These finding are in agreement with expected SBS failure modes as in-plane resistivity is dominated by the conductive carbon fibres, while the out-of-plane measurements are matrix and/or interface dominated. These finding are different to previous ILSS damage sensing works [5,6] where through-thickness resistivity was not affected by loading. In order to monitor the structural health condition of composites components, here the through-thickness method is better than the in-plane method, as it provides a possibility to in-situ monitor the internal deformation and damage before catastrophic failure. Moreover, it agrees with expected failure modes in such a test, which should be either dominated by matrix cracking or interfacial debonding, rather than fibre breakage.

In order to confirm the obtained *in-situ* sensing results, particularly with respect to the observed progressive damage as recorded via the through-thickness method, acoustic emission (AE) was performed separately on a reference specimen during SBS testing. Fig. 5 shows the cumulative AE hits as a function of time together with the load curve. It can be clearly seen that with increasing load during the SBS test, the specimen experiences progressive damage, with the AE hits following a similar trend as previously recorded by through-thickness electrical resistivity measurements (see Fig 3b).

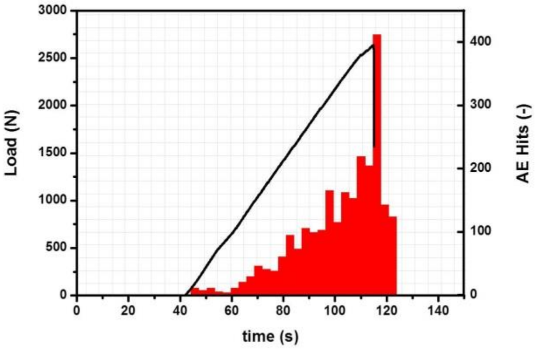


Fig. 5: Acoustic emission hits with load curve, confirming progressive damage during ILSS testing

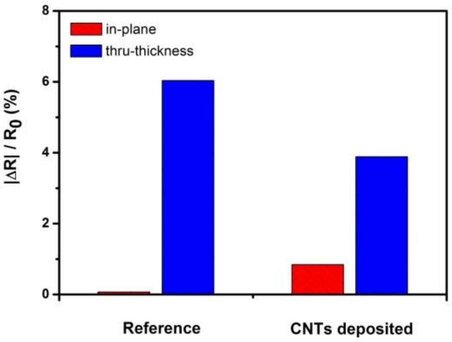


Fig. 6: Comparison of maximum sensing signals before failure between reference and CNT coated specimen

To evaluate the effect of the introduction of CNTs on the sensing properties of the specimen, the average values of the maximum sensing signals before failure are presented in Fig. 6. Compared to the reference CFRP specimen, the maximum sensing signals slightly improved with the introduction of a CNT network for the in-plane method, while surprisingly the sensitivity of the through-thickness signals was slightly reduced by 35%. The increment in in-plane sensitivity is believed to be due to the additional conductive pathways for electrons and is not solely based on conducting carbon fibres but also on the deposited CNT network. Hence the interruption of the in-plane conductive network will result in a greater resistance change at the final point of failure. On the other hand, the conduction mechanism in the case of through-thickness measurements seems to be dominated by physical contacts between carbon fibres, while the addition of CNTs between carbon fibre plies does little to improve the damage sensing behaviour. In SBS testing the specimen are subjected to flexural loading combined with high compressive contact stresses, which may maintain electrical conductive pathways even in plain CFRP laminates This is in contrast to our previous mode-I delamination studies, where obviously such contact stresses are absent and CNTs greatly enhanced the sensitivity towards interlaminar failure.

4. CONCLUSIONS

In-situ electrical damage sensing studies on CFRP and CNT modified CFRP laminates subjected to interlaminar shear loading conditions were presented. CNTs were deposited onto carbon fibre fabric preforms by a spray coating technique. Both in-plane and through-thickness electrical resistivity measurements were applied to monitor damage progression during the test.

Based on the electrical sensing signals recorded during the ILSS test, the through-thickness sensing method was found to be more effective for health monitoring under this loading condition as a gradual increase in electrical resistivity is recorded upon loading, while only a sudden increase in resistivity is recorded for the in-plane method at final failure of the specimen. These findings are in agreement with expected ILSS failure modes, being either (out-of-plane) matrix cracking and/or interfacial debonding rather than (in-plane) fibre breakage. The current method of CNT spray-coated fabrics has shown great potential to detect interlaminar cracking in laminates composites. However, for failure modes like matrix cracking or interfacial debonding at the fibre rather than ply level, percolating networks of CNTs dispersed in the matrix [7] or chemically grown CNTs onto carbon fibre surfaces [12] could be more beneficial. Although further works need to be devoted to optimize the efficiency of the CNT networks for ILSS damage sensing in CFRP (e.g. higher amount of CNTs or alternative deposition methods), this study for the first time revealed the potential of through-thickness electrical measurements to detect and monitor damage in CFRP under interlaminar shear loading conditions.

References:

1. **Wang S, Chung DDL.** Self-sensing of flexural strain and damage in carbon fiber polymer-matrix composite by electrical resistance measurement. Carbon. **44**/13 (2006):2739-2751.
2. **Abry JC, Bochard S, Chateauminois A, Salvia M, Giraud G.** In situ detection of damage in CFRP laminates by electrical resistance measurements. Comp. Scie and Technol. **59**/6 (1999), 925-935.
3. **Wen J, Xia Z, Choy F**. Damage detection of carbon fiber reinforced polymer composites via electrical resistance measurement. Comp. Part B-Engin. **42**/1 (2011), 77-86.
4. **Schulte K, Baron C**. Load and failure analyses of cfrp laminates by means of electrical-resistivity measurements. Compos Sci Technol. **36**/1 (1989), 63-76.
5. **Takeda T, Shindo Y, Fukuzaki T, Narita F**. Short beam interlaminar shear behavior and electrical resistance-based damage self-sensing of woven carbon/epoxy composite laminates in a cryogenic environment. J Compos Mater. **48**/1 (2014), 119-128.
6. **Boeger L, Wichmann MHG, Meyer LO, Schulte K.** Load and health monitoring in glass fibre reinforced composites with an electrically conductive nanocomposite epoxy matrix. Compos Sci Technol. **68**/7-8 (2008), 1886-1894.
7. **Thostenson ET, Chou T-W.** Carbon nanotube networks: Sensing of distributed strain and damage for life prediction and self healing. Adv Mater. **18**/21 (2006), 2837-.
8. **Inam F, Peijs T.** Transmission light microscopy of carbon nanotubes-epoxy nanocomposites involving different dispersion methods. Adv. Compos. Lett. **15**/1 (2006), 7-13.
9. **Li X, Huang Y**. Influence of acid treatment on dispersion of carbon nanotubes in PBO matrix. Adv. Comp. Lett. **14**/6 (2005), 201-206.
10. **Zhang H, Liu Y, Kuwata M, Bilotti E, Peijs T.** Improved fracture toughness and integrated damage sensing capability by spray coated CNTs on carbon fibre prepreg. Compos Pt A-Appl Sci Manuf. **70** (2015), 102-110.
11. **Wang DJ, Chung DDL.** Through-thickness piezoresistivity in a carbon fiber polymer-matrix structural composite for electrical-resistance-based through-thickness strain sensing. Carbon. **60** (2013), 129-138.
12. **Zhang J, Zhuang R, Liu J, Maeder E, Heinrich G, Gao S.** Functional interphases with multi-walled carbon nanotubes in glass fibre/epoxy composites. Carbon. **48**/8 (2010), 2273-2281.