DAMAGE DETECTION IN CARBON FIBRE CROSS-PLY LAMINATES BY AID OF CARBON NANOTUBE DOPED RESIN

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ABSTRACT

The possibility to use resistance variations of carbon nanotube (CNT) doped carbon fibre reinforced plastics (CFRP) to characterise the level of microstructural damage in the form of transverse cracking is experimentally investigated in the current work. The resistance of CNT-doped and unmodified CFRP was measured after they had been subjected to stepwise increased tensile loading. Both increase and decrease resistance was observed which made it difficult to make any firm statement that the changes we observed were due to damage. The conductivity along carbon fibres is dominating electrical conductivity of the studied cross-ply laminate. Any disturbance of conductive CNT based nanocomposite matrix by transverse cracks in 90-layer therefore caused too small changes of resistance to be measured with current experimental setup.

1. INTRODUCTION

Among the many types of polymeric nanocomposites that were intensely studied in the last decade, composites based on CNT have attracted significant attention [1]. One interesting feature of these nanocomposites is that they become electrically conductive even if very small amount of nanotubes is added, i.e. the percolation threshold is attained for low fractions of CNTs. Another important characteristic of these materials is their piezoresistivity i.e. the electrical resistivity varies with applied strain. Both these features were found very useful since they offer a new way for integrating deformation and damage sensing ability in fibre reinforced polymer composite materials [2-4].

In previous work [2] it was shown that a glass fibre composite (GFRP) can be made electrically conductive by using CNT-doped epoxy resin as matrix. This conductivity can be used for deformation sensing purpose and characterization of micro-damage [2]. The resistance changes upon loading of a cross-ply GFRP is illustrated in Fig. 1. The curve was obtained by loading-unloading experiments where the maximum strain level was incrementally increased after each cycle. The resistance changes seen in Fig. 1 originate from three different mechanisms; a) geometrical changes of the specimen b) piezoresistive material response and c) accumulation of micro-damage at strain levels above 0.3%. While the damage detection possibility offered by CNT is firmly established for the case of GFRP it is less established for CFRP. In the case of GFRP, the electric conduction takes place solely in the CNT-doped matrix material since glass fibres are not conductive. Therefore transverse cracks in the 90-layer significantly alter the pathway for electrical transport within the material and cause changes in electrical resistance. For the case of CFRP the situation is very different. A majority of the electrical conduction takes place in the carbon fibres (mainly along the fibre direction) and one can not expect that resistance of cross-ply carbon fibre composites should exhibit the same sensitivity towards transverse cracks .

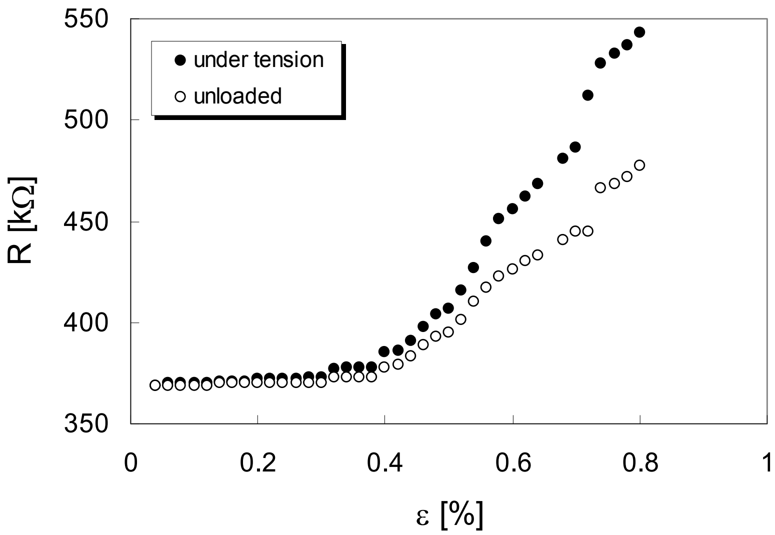

Fig. 1: GFRP specimen resistance extremes during loading – unloading experiments (from [2])

The main objective of the current work is to investigate the possibility to use resistance variations of CNT doped carbon fibre composites to characterise the level of microstructural damage in the form of transverse cracking. Results from an experimental investigation on CNT-doped carbon fibre cross-ply laminates are presented. The extent of transverse cracking is correlated with registered resistance changes and with degradation in mechanical properties. The results are analyzed in the light of data available from similar studies in glass and carbon [5] fibre cross-ply laminates.

2. MATERIALS AND SPECIMEN PREPARATION

CNT-doped nanocomposites with 0.5 wt% CNT were prepared and used as matrix in the CNT-doped continuous fibre composites. An anhydride cured epoxy system Araldite LY556/Aradur HY 917/Accelerator DY 070 mixed in the ratio (by weight) 100/90/1.5, is used as resin for the nanocomposites. The CNTs used were as-produced multi walled carbon nanotubes (MWCNT) supplied by Arkema, France.

Preparation of CNT nanocomposites was performed by dispersion of CNT in the base resin in water cooled steel containers. A Sonics VC 505 tip sonicator equipped with a 13 mm standard tip was used to promote dispersion. Sonication was performed in repeated 30 seconds interval (sonication for 30 seconds followed by a period rest for 30 seconds) and lasted in total for 4 hours. A Heidolph RZR 2650, laboratory mixer was used for mixing of hardener and accelerator with the base and MWCNT suspension. Accelerator and hardener were kept at ambient room temperature. The mixed resin was degassed at a pressure of 150 mbar for 10 minutes.

A unidirectional Non Crimp Fabric (NCF), Devold T320-C05-A, produced by AMT Devold and based on Toray 12k T700S carbon fibres were used to prepare cross-ply samples with $[0/90_2]_s$ lay-up. Three such laminates were produced and denoted as REF-TC2-CF1, CNT-TC2-CF1 and CNT-TC2-CF2. REF-TC2-CF1 is the reference plate made with neat epoxy resin whereas CNT-TC2-CF1 and CF2 are plates made with CNT-doped epoxy as matrix. The plates REF-TC2-CF1 and CNT- TC2-CF1 were produced by conventional RTM (illustrated in Fig. 2A) whereas CNT-TC2-CF2 was produced by a manual moulding procedure (illustrated in Fig. 2B). The manual moulding is performed by placing and manually impregnate the fibre preform in the mould cavity before closing the mould. Compaction of the fibre and resin preform is made by manually tightening bolts distributed along the perimeter of the mould. Excess resin is evacuated through gates in the upper tool half. This moulding procedure was used because RTM with CNT-doped resin caused inhomogeneous CNT-distribution due to filtering of the nanofiller.

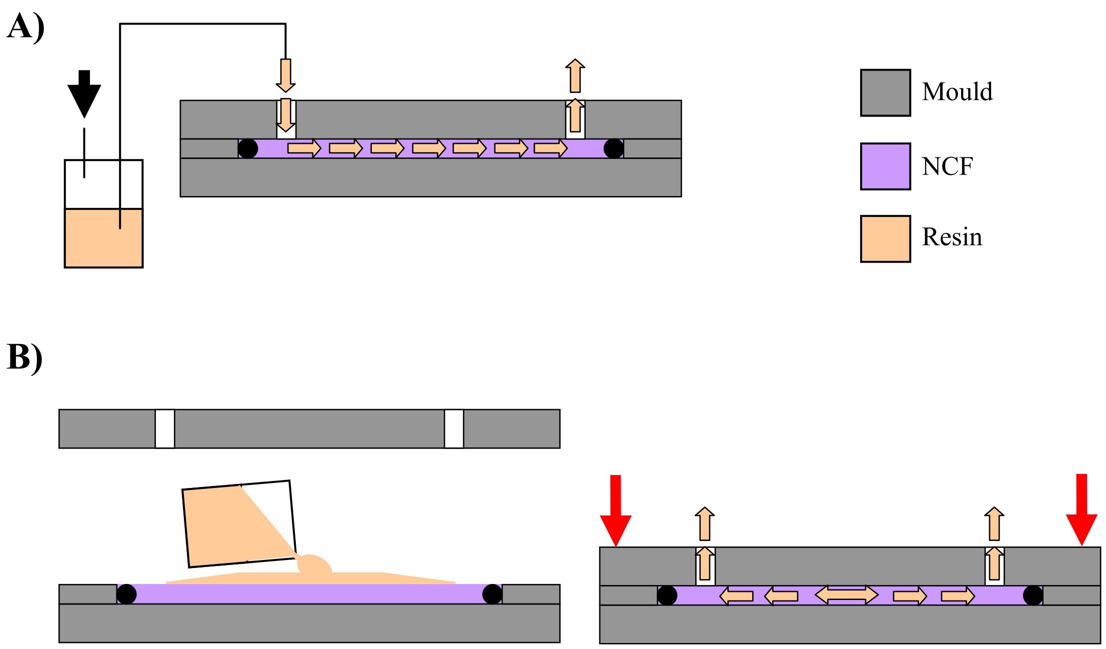

Fig. 2. Manufacturing principles: A) conventional RTM and B) compression moulding in RTM-tool of hand-impregnated pre-form.

In order to reduce filtering and ensure easier resin flow the fibre volume fraction was intentionally kept low, between 40 - 45%. As it turned out the low compaction pressure associated with the low fibre content permitted the outermost 0-degree layers to deform slightly due to the shear forces applied by the flow. All materials were cured for at least 8 hours at 80°C followed by a 4 hours free standing post cure at 140°C. Test specimens have widths of 8-10 mm. Contacts for resistance measurements are produced by painting two contact areas, each separated by a distance of 70 mm, with conductive paint. Wires are attached to the contacts by use of conductive paint and epoxy adhesive. Sand-paper is glued on the specimen at the locations where the extensometer is attached i.e. with a separation of 50 mm (to avoid extensometer sliding and to electrically isolate extensometer from the specimen).

3. EXPERIMENTAL

Mechanical testing was performed using an INSTRON 3366 testing machine with a maximum load capacity of 10kN. The applied strain was measured by a standard Instron extensometer. All measurements of electrical resistance are made using a Keithley 2100 DMM in the two wires resistance measurements mode. Resistance, load and strain are all measured with the same sampling rate of 5 samples/second. Transverse cracking tests are performed according to the loading-and-unloading ramp indicated in Fig. 3. The loading ramp implies that maximum strain increases gradually throughout the test. Between each increasing step is a cycle which reaches a maximum strain of ε=0.3 %. Elastic modulus is determined during this cycle. Indicated in the figure is also the time when resistance of the damaged laminate is measured and when number of transverse cracks is counted by use of optical microscope (specimen was dismounted from the machine for the cracks to be counted and measurements of the resistance). Two specimens were subjected to the tensile loading-unloading sequences where maximum strain reached 0.3% and during which resistance changes were continuously monitored by the DMM. In this test, a GFRP cross-ply laminate with CNT-nanocomposite matrix was tested alongside with CFRP. The specification and manufacturing procedure of the GFRP sample is found in [2].

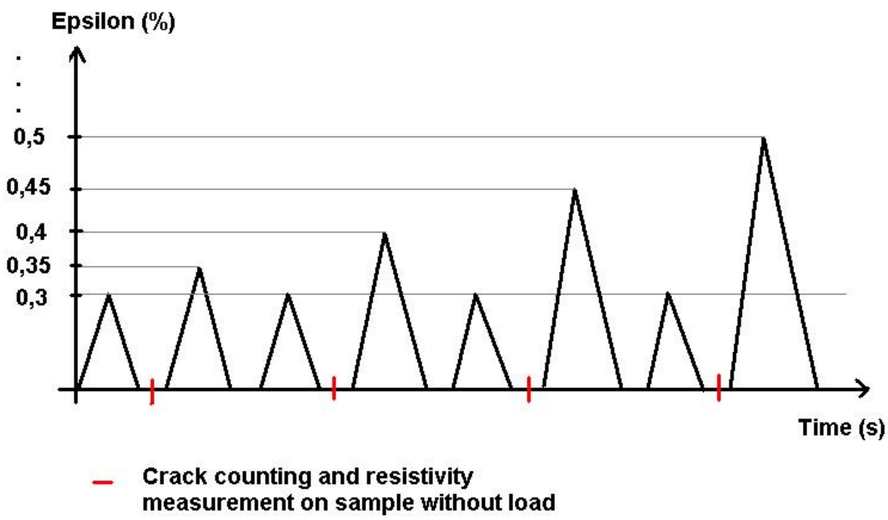

Fig. 3. Principal loading history (applied strain as a function of time)

4. RESULTS AND DISCUSSION

Typical results obtained when continuously monitoring resistance changes during a loading-unloading sequence are presented in Fig. 4. (values are normalized with respect to the resistance of sample before any mechanical load was applied). The specimen resistances in an virgin and unloaded state are R_0 = 7.2 Ω, 47 Ω and 547 kΩ for the CNT-doped CFRP, reference CFRP and CNT-doped GFRP respectively.

We notice that resistance follows the same general shape of the curve for all specimens, regardless if the resin contains CNT or not. This is in line with previous knowledge that the resistance of carbon fibre composites are sensitive to strain [5]. The magnitudes of relative resistance variations vary between different samples. It can be noticed that the largest relative change is reported for the CNT-doped CFRP whereas the other two are similar in magnitude. Too few specimens were tested to support any definite conclusion that CNT enhances sensitivity of CFRP to strain.

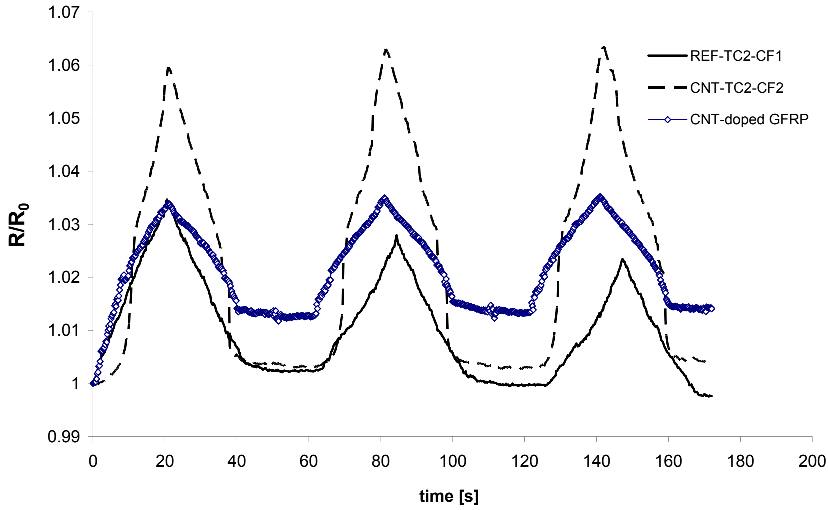

Fig. 4. Relative resistance changes during tensile loading-unloading for various material configurations

An interesting observation is that a permanent increase in relative resistance is observed after the first loading cycle of the GFRP specimen. This could be an indication that permanent damage in the form of cracks could be developed during the first loading cycle. In summary we may conclude that the observed resistance changes upon tensile loading …

- is generally governed by the carbon fibres in case of CFRP.
- is only to a minor extent depending on CNT-nanocomposite for CFRP.
- is solely a consequence of CNT-nanocomposite electrical conductivity for the case of CNT-doped GFRP.

Six specimens were tested and analyzed according to the loading schedule shown in Fig. 3. Resistance over the gauge length was measured for all specimens after each loading cycle. The number of transverse cracks over a fixed length of the specimen was counted after each loading step in the loading-unloading cycle. The results are presented in Fig. 5. From this graph we can say that CNT-based specimen have onset of cracking at lower strains than in reference specimens. First cracks appear at strain of 0.35-0.40% for CNT-based specimens whereas the corresponding interval for the reference material is 0.4-0.6%. The crack density at a given strain is generally higher in CNT-doped as compared to reference specimens.

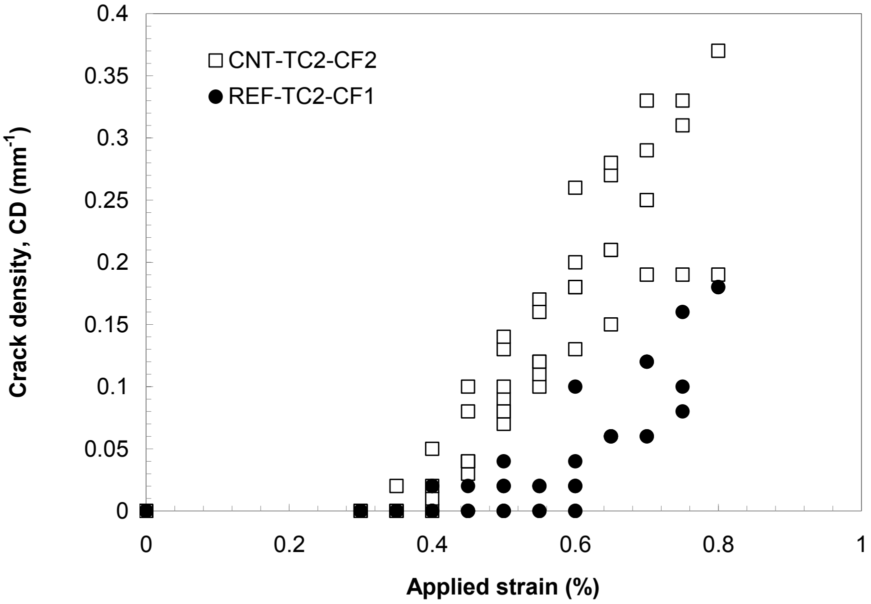

Fig. 5. Transverse Crack Density (CD) vs. strain

Results presented in Fig. 6 shows that stiffness of both materials starts to degenerate as the strain increases. Due to certain experimental scatter it is difficult to estimate a single valued onset strain where the stiffness starts to decrease. It appears that stiffness of both materials have decreased once ε = 0.4% is exceeded. Above this strain the stiffness gradually continues to decrease. It is evident from the results that CNT specimens are subject to a stronger modulus decrease, in particular at strains higher than ε = 0.6 %. In other words, for the same applied strain we have a stronger decrease of the young modulus in CNT specimens than in reference specimens (this is of course due to higher crack density observed in CNT doped composites). An unsatisfactory fact is that the magnitude of the stiffness decrease in CNT-doped specimens is higher than what one expects for the current cross-ply laminate configuration. By using the simplest form of ply discount model, assuming that the 90°-layer completely ceases to contribute to the stiffness once a transverse crack appears and it fails, one can estimate the maximum theoretical degradation due to damage in the transverse layer. With the current material configuration such analyses yields a theoretical minimum value of E/E_0 = 0.88. The low values observed in Fig. 6 can hence not be caused by damage and cracking in the transverse layer only. The values can only be explained by certain damage in the longitudinal layers. This phenomenon in NCF cross-ply laminates has been observed in another study of similar CFRP [6]. By this we conclude that we did not completely manage to promote and isolate the desired type of transverse cracking in our test. Transverse cracking took place alongside with other type of damage e.g. delaminations, at relatively low strain levels. Delaminations have a profound impact on the stiffness of the material and explain the comparably large stiffness reduction observed in particular for CNT-doped specimen in Fig. 6. It is reasonable to assume that the curvature and misalignment of the 0°-layer can induce premature delamination, preferably starting at specimen edges, which severely accelerate the stiffness degradation of these materials.

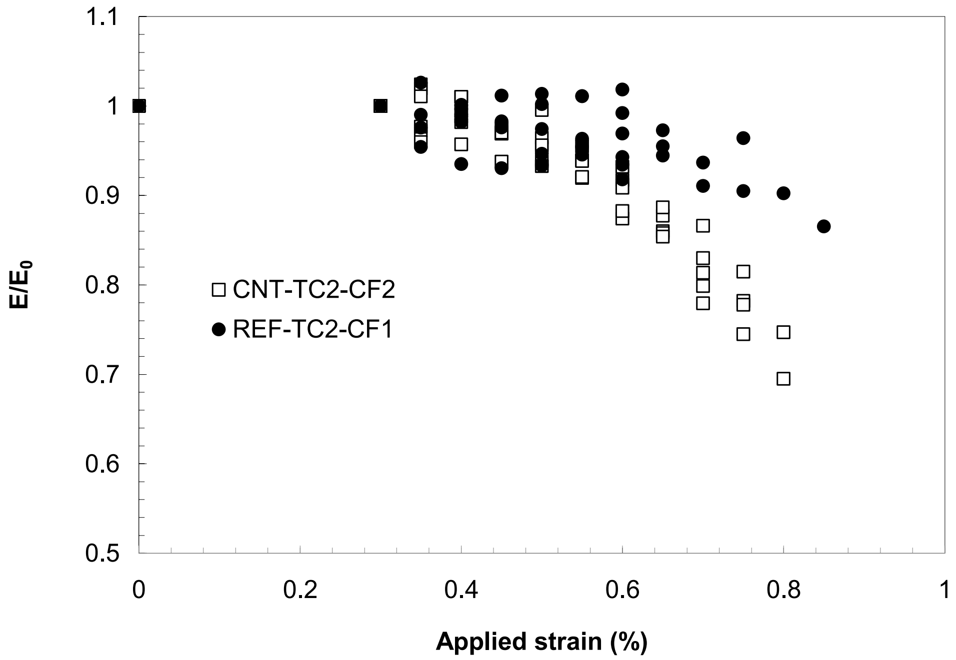

Fig. 6 E/E_0 vs. strain

The actual resistance, *R* in an unloaded case was compared to the resistance R_0 of the pristine specimen. Previous work on CNT-doped glass fibre composites showed that resistance of the specimen increased with increasing load level due to damage in the material [2], a similar trend could be expected for the current CFRP materials. The outcome of the present tests was surprising. Resistance in materials without CNT had in general a tendency to decrease with increasing load level, see Table 1. We have not a satisfactory explanation for this behaviour at the moment. The results for CNT doped materials are equally difficult to explain since both an increase and a decrease in resistance is observed. An experimental uncertainty is that we observed a tendency that the registered resistance decrease over time for some reason when we measure resistance of the damage specimens. We have waited until resistance values stabilized before registering the actual values. The major objective and ambition of this work was to investigate whether resistivity and resistance changes in CNT-doped CFRP can be used as a measure of damage accumulation. The most general statement with respect to this is that our results do not provide a solid foundation for such a conclusion.

Table 1. Resistance changes of damaged laminates

Material	Sample ID	Resistance change after loading to $\varepsilon \approx 0.6$ %
REF-TC2-CF1	2-22	-5%
REF-TC2-CF1	2-23	-0.5%
REF-TC2-CF1	2-24	-7%
CNT-TC2-CF2	2-30	+30%
CNT-TC2-CF2	2-32	-20%
CNT-TC2-CF2	2-33	+3%

5. SUMMARY

The major objective of the work was to experimentally establish whether it is possible to use resistance changes in a CNT-doped CFRP cross-ply laminate to follow the damage (transverse cracking) progress. It was not possible to make any statement that the changes we observed were due to damage. Rather large resistance changes, both increase and decrease, was observed for CNT-doped laminates as well as for reference CFRP. A reason why we are somewhat cautious with making strong conclusions is some experimental uncertainties. Fibres in the 0°-layer of the tested CNT-based laminate were curved. A consequence is that a delamination is likely to occur at lower loading levels. This influences the overall rate of damage progression. The reason for the observed initial drift in resistance measurement is unclear and also shed some uncertainty about the interpretation of these results. While continuously measuring resistance during loading-unloading experiments we could conclude that resistance varies with strain level in similar manner for three types of materials tested i.e. CNT-doped GFRP, CNT-doped CFRP and reference CFRP. From this we conclude that resistance changes upon tensile loading are generally governed by the carbon fibres in case of CFRPs. It is only to a very minor extent depending on enhancement of CNT-nanocomposite conductivity for CFRP. This is in contrast to the case of GFRP where resistivity changes are exclusively a consequence of CNT-nanocomposite electrical conductivity.

6. ACKNOWLEDGEMENTS

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