1 General Introduction

Laminated composite materials have been used in many industrial applications. When a composite laminate is subjected to mechanical loading, its thickness changes. Changes in thickness of laminate happen due to Poisson coupling effect. This effect produces high interlaminar stress around free edges which can cause delamination failure [1]. Many researches were performed to calculate the values of interlaminar (IL) strains and stresses analytically and numerically [2, 3]. Real-time monitoring of through-thickness strain (TTS) in composite laminates, particularly at locations away from the free edges of the laminates will improve safety and proper performance of composite structures. Experimental technique to measure TTS is needed. Technique of small strain gages may be used to measure TTS at free edges. However it is not possible to determine the TTS at locations away from the free edges. Technique of fiber optics may be used to measure in-plane strains and transverse strain in laminates [4]. However, the diameter of the fiber is still large (52 micrometers, which is more than 6 times the diameter of a glass fiber, and about half the thickness of ply) and the interference due to the insertion of the optical fiber may not give accurate strain values. Technique of Moire interferometry was used to measure the TTS at the edge of the laminates [5-7]. However, it cannot be used to determine the TTS at locations away from the laminate edge. The exceptional electrical, thermal, physical and mechanical properties of carbon nanotubes (CNTs) combined with their small size and high aspect ratios make them excellent nanoscale reinforcement for advanced composite structures [8-10]. The incorporation of the CNTs into the resin makes the resin to be electrically conductive and the combined material can be used as sensor. Recently attempts have been made to embed CNTs into polymers to monitor strain and subsequently failure in polymer matrix composites (PMCs) using electrical resistance measurement (ERM) [11]. Fiedler et al. [12] first reported the ability of CNTs as conductive additive for damage sensing in composites. Boger et al. [13] added CNTs and carbon blacks in the glass/epoxy composites and found that there is correspondence between the change in in-plane strain and the change in through-thickness electrical resistance (TTER). Thostenson et al. [14-15] dispersed CNTs into epoxy to make glass/epoxy/CNTs composite specimens and showed that cracking in tensile specimens corresponds with the increase in in-plane electrical resistance. Nofar et al. [16] incorporated CNTs into epoxy to fabricate glass/epoxy/CNTs composite specimens and found that damage in the specimens is detected by significant change in in-plane electrical resistance during tensile and fatigue testing. They also reported that electrical resistance change (ERC) is more sensitive than strain gage measurements for damage monitoring. Gao et al. [17] deposited CNT onto glass fiber surfaces and showed that epoxy composites made using this fiber system may be used for in-situ sensing of strain and damage. Alexopoulos et al. [18] developed fibers consisting of CNTs in a sheath of polymer matrix. These fibers are then embedded inside the glass/polymer structure for detecting mechanical deformation using the change in electrical resistance of the special fiber. Hena-Zamal and Hoa [19] embedded CNTs in glass/epoxy composites and indicated that there is correspondence between the change in TTER and damage accumulation during fatigue testing. Naghashpour and Hoa [20] showed that TTER of glass/epoxy/CNTs composite specimen is sensitive to uni-axial load applied along the length and across the thickness of the specimen. The above works illustrate very interesting attempts to monitor strain
and damage in glass/epoxy/CNTs composite specimens by electrical resistance measurements when the specimen is subjected to tensile and fatigue loading. However, it remains to be observed whether TTS can be monitored by measuring electrical resistance of specimen under four-point bending. Here, we investigated the electrical behaviour of glass/epoxy/CNTs composite specimen subjected to four-point bending test. In the present work, Multiwall carbon nanotubes (MWCNTs) were dispersed into epoxy resin. This modified epoxy resin was then incorporated with long glass fiber to make \([0/90/0]\) laminates. The specimens were tested in four-point bending while the through-thickness and in-plane electrical resistances were measured. The experimental results show that the in-plane and through-thickness electrical resistances are effective to track in-plane and through-thickness strains of the specimen under four-point bending load.

2 Experimental Materials and Methods

2.1 Materials

Multiwall carbon nanotubes (MWCNTs), Unidirectional S-glass fibers, and (Epon 862, EPIKURE W) were purchased from Bayer Material Science, ACP Composites and Miller-Stephenson Chemical Companies respectively.

2.2 Fabrication

Composite laminate of \([0_5/90_8/0_5]\) layup sequence having the largest average through-thickness strain was selected for fabrication [20]. In order to make \([0_5/90_8/0_5]\) glass fibers/epoxy composite containing MWCNTs, the epoxy resin and curing agent (26.4 wt %) were first mixed. Then 0.3wt% MWCNTs were added into epoxy matrix (the percolation threshold of the MWCNTs in epoxy was determined experimentally to be 0.1883 wt% [21]). The mixture was processed using calendering approach (EXAKT 80E, EXAKT Technologies Inc) to disperse the MWCNTs within the epoxy matrix. The modified epoxy was then heated up to 70°C for 20 min in vacuum oven to remove air bubbles. Eighteen layers of unidirectional S-glass fibers were wetted with the modified epoxy by hand lay-up method. The plate was cured using an Autoclave.

2.3 Arrangement of electrical connections and strain gage

The ASTM standard D7264-D7264M-07 was utilized for performing four-point bending tests on the \([0_5/90_8/0_5]\) composite plate. The fabricated plate was cut into strips of dimensions (250 mm × 25 mm × 2.16 mm). Six conductive electrical contact points made from silver-epoxy paste were mounted on the top (T) and bottom (B) surfaces of the \([0_5/90_8/0_5]\) specimen. Then electrical wires were attached with silver-epoxy paste on both surfaces of the specimen to make electrodes for electrical resistance measurement. For measuring ATTS, a small strain gage (1.9 mm long) was bonded on the thickness section of the sample. The specifications of specimen and arrangements of electrical connections and bonded strain gage on the specimen are schematically illustrated in Fig.2.

2.4 Electrical resistance measurement

The electrical resistance measurement (ERM) was used to monitor the state of electrically conductive specimen. The electrical resistance measurement adopts the material itself as a sensor and it does not need expensive equipment. ERM was performed by two-probe method using Agilent digital Multimeter (34401A). Electrical Resistance Change (ERC) is expressed by:

\[
ERC = \frac{\Delta R}{R_i} = \frac{R_f - R_i}{R_i}
\]

(1)

Where \(R_i\) and \(R_f\) are the electrical resistance before and while loading respectively.

Fig.1.SEM of laminate section
2.4. Average through-thickness strain (ATTS) measurement

Average through-thickness strain measurement was performed using conventional metallic strain gage (L1E-350K-PC06-LE) from MFL Company [22]. The length of the copper wire in the strain gage is 0.74 mm. However there is a backing required for bonding with the substrate. The length of the backing is 1.9 mm (the thickness of the sample was about 2.16 mm, which is adequate). The accuracy of these gages is 3%, as provided from the manufacturer. As such the strain gage only measures the strain over the 90° layers (which is about 0.96 mm thick), and not over the whole thickness of the composite specimen as shown in Fig.2.

3. Four-point bending test while monitoring strain and electrical resistances

To investigate the effect of a four-point bending load on electrical resistances and strain, the specimens were tested in four-point bending using 100 KN-MTS mechanical testing machine.

Four-point bending tests were done for six specimens with a crosshead displacement of 0.127 A. A schematic of the four-point bending set-up and experimental details are illustrated schematically in Fig.3. The specimens were loaded under four-point bending while in-plane and through-thickness electrical resistances and through-thickness strain were measured simultaneously. The applied loads, displacement, strain and electrical resistances in real-time were recorded using MTS machine, strain gage conditioner (2120A), power supply (2111), Agilent machine (34401A) respectively.

4 Results and discussion

The in-plane and through-thickness electrical resistance responses measured from CNT networks dispersed in the [0/90/0/90] specimen are investigated when the specimen is subjected to four-point bending.

Fig.2. Schematic illustration of specimen specification and locations of strain gage and electrodes
The specimen was subjected to four-point bending while in-plane electrical resistances at top and bottom surfaces of the specimen and also TTER were measured simultaneously. Since the aim of this study is to check the possibility of using change in TTER as a measure of ATTS, maximum allowable load that can provide linear (elastic) reign in Load-displacement curve is required. A maximum load of 500 N was experimentally determined for the \([0/90/0]_8\) specimen in order to work on elastic reign.

4.1. Four-point bending load (z direction) while monitoring displacement and electrical resistance (z direction)

Four-point bending load and change in TTER measured at the edge of the specimen versus displacement curves are shown in Fig. 4. The change in TTER was calculated based on Eq. 4. As it can be observed from Fig. 4, the load and change in TTER increase linearly as displacement increases.

Fig. 3. Four-point bending test setup a) Specimen placed on MTS machine with load cell under four point bending. b) Close-up view of Load span, support span, electrodes and strain gage bonded to specimen.

Fig. 4. Four-point bending test results showing load and change in TTER at the edge of the specimen versus displacement curves for the specimen.
4.2. Four-point bending load (z direction) while monitoring strain and resistances (z direction)

Four point bending test was performed while through-thickness strain and electrical resistances along the length and across the thickness of the specimens were measured. The obtained experimental results of ATTS measured for 90ø layers located at the center of the [05/908/05] specimen and TTER at the edge of the specimen are plotted in Fig.5. It is observed from Fig.5 that there is good relation between change in TTER and the magnitude of ATTS measured using strain gage for 900 layers localized at the center of the specimen.

![Fig.5. ATTS measured strain gage and change in TTER-Load curves for the specimen](image)

4.3. Four-point bending load (z direction) while monitoring in-plane resistances (compression side)

It is clear from Fig.6 that the change in in-plane electrical resistance measured on compression side of the specimen decreases with increasing load. This indicates that CNT networks are contracted due to compression.

![Fig.6. change in in-plane electrical resistance (compression side)-Load curves for the specimen](image)

4.4. Four-point bending load (z direction) while monitoring in-plane resistances (tension side) and strain

Fig.7 shows that the change in in-plane electrical resistance measured on tension side of the specimen increases as load increases. This reveals that CNT networks are extended due to tension.

![Fig.7. change in in-plane electrical resistance (tension side)-Load curves for the specimen](image)
It is seen that the top and bottom surfaces of the specimen are in compression and tension respectively when it is tested in four-point bending. These compression and tension sides produce negative and positive change in in-plane electrical resistances respectively. Different electrical behaviour for MWCNT networks dispersed in the specimen is observed when the specimen was subjected to four-point bending. The different signs for the change in electrical resistance are due to variation of MWCNT networks, pezoresistivity of individual MWCNTs as well as a change in intertube tunneling distance between crossing MWCNTs. As such, different signs for the change in electrical resistance are as result of different loading conditions.

4 Conclusions

The ability of using CNT networks in glass fiber/epoxy composite laminates to track in-plane and through-the-thickness strain under four-point bending was investigated. The obtained experimental results demonstrate that in-plane and through-thickness electrical resistance measurements are effective to track the in-plane and through-thickness strains. It can be concluded that the addition of MWCNTs into epoxy in glass/epoxy/MCNTs composite laminates make the combined materials as sensor which can be used as a new method for the measurement of in-plane and through-thickness strains. The method to monitor strain is based on monitoring in-plane and through-thickness electrical resistance changes via the mounted electrodes on both surfaces of the specimen.

5 References

[15] ET. Thostenson and TW. Chou “Real-time in situ sensing of damage evaluation in advanced fiber


