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Damage sensing and mechanical properties of laminate composite based MWCNTs under anticlastic test

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Abstract

Self-sensing composite based MWCNTs is important to diagnose the damage in its early stage and to prevent a fatal fracture that could happen in the structure during the working. This paper investigates the effect of adding different concentrations of MWCNTs to the matrix on the mechanical and electro-mechanical properties of the composite laminates under a different type of testing. MWCNTs were a dispersion in the epoxy resin using ultrasonic technique and then the mixture handed lay-up on the glass fibre composite layers followed by vacuum bagging process. The composite samples, which have various percentages of MWCNTs, have been tested using flexural and anticlastic tests. This type of complex test i.e. (anticlastic) gives more understanding to explore the effect of combined twisting and bending deformation on the strain sensitivity of the glass fibre /MWCNTs reinforced composite. The results indicated that the adding of MWCNTs can improve the mechanical properties of the smart composite. The maximum stresses values were observed at the flexural test while the maximum Young's modulus values were at the anticlastic test. SEM pictures have captured from the broken surface to exam the MWCNTs dispersion inside the resin and between the glass fibre layers. From the SEM images, MWCNTs were distributed well inside the matrix while there are some agglomerations occurs at the 2.0 wt% of MWCNTs. The electrical conductivity increases with increasing the wt% of MWCNTs content and showing a high value $1.8 \times 10^{-3}\,\mathrm{s\,m^{-1}}$ at 2.0 wt% of MWCNTs content. The strain deformation due to the flexural test was one i.e. in (y-direction) only and was higher than both strain values when the sample under the anticlastic test. The results also show that there are two strain sensitivities but in opposite signs which obtained by anticlastic test while one only under flexural test and had a magnitude equal to about half value compare with those for the anticlastic test. Therefore, this type of test gives a deep vision to investigate the behaviour of the smart composite when it deforms to a saddle shape.

1. Introduction

Fibre reinforced polymer to made composites have been used widely in many important applications such as oil, gas, aircraft, marine and military industries because of their high strength to weight ratio, fatigue damage resistance, and high corrosion resistance [1, 2]. However, because the nature of their laminates, the mechanical properties are relatively low in the out of plane zone, practically, in the interlaminar shear strength. In addition, due to discrete layers of unidirectional or woven fibres that impregnated with epoxy, a micro-scale crack could happen during the loading and that could develop to make delamination, interfacial debonding, fibre breakage and matrix cracking as well [3]. Therefore, the better mechanical properties are importance for the interfacial zone to transfer the load easily from between the composite components. Recently, it has become more important to fabricate a new strong multifunctional composite such as smart composites or self-sensing composites, which facilitates to work with structural health monitoring (SHM) system. Strain gauges and optical fibre have used in (SHM) to detect the damage when the composite is stressed. Embedding these types of sensors in composite laminates could lead to creating a pre-crack inside the matrix and an additional interface will

Table 1. Some properties of the materials that have used and as per the supplied manufacturing.

Property	MWCNTs	Epoxy	Glass fibre
Density (g cm^{-3})	2.1	1.15	2.54
Modulus (GPa)	~1200	3	72
Strength (GPa)	~63	0.07	3.4

generate and making the composite weak in use [4]. Many researchers have tried to fabricate these multifunctional composite by embedding nanofillers within the matrix. Carbon nanotubes (CNTs), which were discovered by Iijima [5], are one of these fillers. They have high mechanical, electrical, thermal and flame-retardant properties [6–9]. The using of CNTs even at low concentrations could improve the mechanical properties and strain sensitivity of composite [10]. Kathi *et al* [11] have studied the adding of 0.2% CNT to the epoxy and the results show that there is about 18% improvement in flexural strength over the neat epoxy. Another group [12] has reported that addition of 2 wt% of CNT in CF/epoxy composite resulted in 42% and 6% and increment in flexural strength and flexural modulus, respectively. Fan *et al* [13] investigated adding 2.0 wt% of MWCNTs to reinforce the glass fibre/epoxy composite and found 33% increment in the interlaminar shear strength property. Moreover, CNTs are not only improving the structural properties but also it is considered as a nanosensor to detect the crack from the initiation stage. The main advantage of the self-sensing technique is that all the original mechanical properties of the structure are protected while a micro internal damage can quickly be sensed and monitored before developing [14, 15]. Some other studies have been done about the embedded the CNTs in the matrix to fabricate a self-sensing composite and tested under different testing types such as tensile [16], bending [17], fatigue [18], impact [19]. In fact, carbon fibre/epoxy composite laminates could be damaged during usage because of the different stress and other environmental factors. The generation of damage and its accumulation in composite materials will develop and lead to accelerating the senility process of the materials. Consequently, a serious reduction in the lifetime and the resistance to the harsh conditions lead to catastrophic consequences.

Therefore, form the above literature, the researchers concentrate only on the pure test such as tensile or bending etc, but the anticlastic-bending test is very important to investigate the static failure behaviour of composites when it is as a sheet or plate shape.

In this work, the main aim is to study the effect of the MWCNTs, which mixed with the polymer with different percentages on the mechanical and electro-mechanical behaviour of the composites under various tests. These tests are (1) anticlastic bending test, (2) pure flexural test for comparison. The purpose of these tests is to investigate the effects of the distributed MWCNTs in the matrix of the composite on the reversibility mechanical properties of laminate composites under the above tests. Also, to investigate the damage accumulations in the composites by calculating the piezo-resistivity and the strain sensitivity of the composites under these tests with a specific strain deformation.

2. Methodology

2.1. Materials
The multi-wall carbon nanotubes (MWCNTs) were prepared by CVD method and supplied by US-Research Company, USA. Its purity is >98% and the length is 50 μm. Outer and inner diameter is 15 nm and 5 nm respectively. A commercial glass fibre type Woven Glass Plain and epoxy resin type slow EL2 laminate with its hardener have been supplied from the same company, which is easycomposite company, UK. Some important specifications for above materials are listed in table 1.

2.2. Manufacturing of MWCNTs modified glass fibre composites
Pre-calculated amounts of MWCNTs and epoxy resin were carefully weighted and then mixed together manually in a clear beaker. A high-intensity ultrasonic device type (BR-20MT-10L, 1000 W) has been used to dispersion the MWCNTs within the epoxy for 20 min with pulse mode 45 s on and 15 s off and with amplitude 80%. The beaker was kept in a cooling water container during the sonication process to keep the mixture from the overheating. Once the sonication process complete, the hardener has been added to the mixture with mixing weight ratio of epoxy and hardener 100:30. Based on the British standard BS EN ISO 14125-1998 + A1:2011, the laminate thickness is 2 mm for the samples for the flexural test. For this purpose, 10 layers of the woven glass fibre with MWCNT/epoxy were used. The hand lay-up process had been used to fabricate glass fibre /MWCNTs/epoxy composites. During the manufacturing process, the fabric (300 mm × 300 mm) were saturated with the epoxy resin, which already mixed with the MWCNTs, layer by layer to ensure that the resin

Figure 1. Schematic illustration of glass fibre/MWCNTs/epoxy composite fabrication.
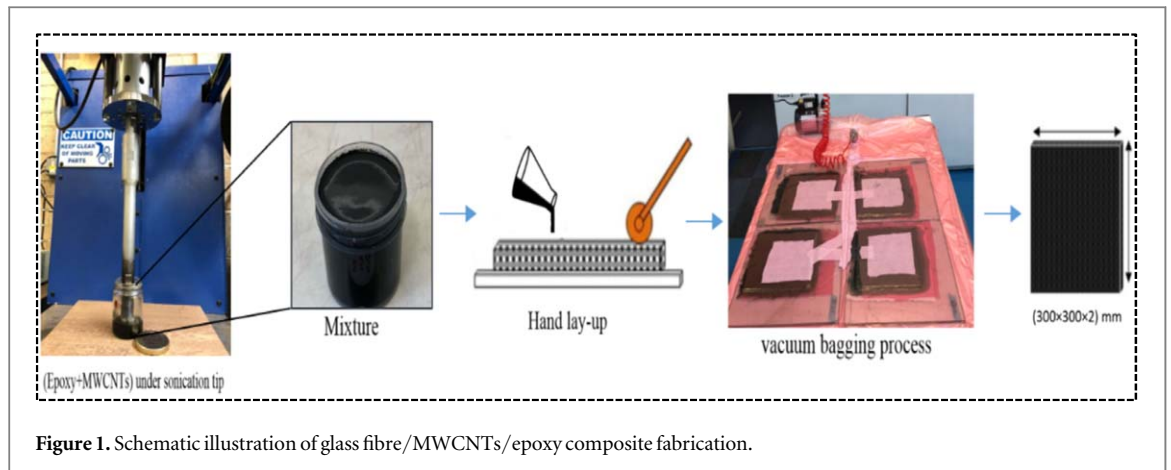
has been distributed homogenously between them. This has been done by using a roller. After complete the hand layup processing, the composites were put in the vacuum bag for 24 h. Finally, the cured laminates were put again in the oven for 15 h at 60 °C for post curing processing. The glass fibre/MWCNTs/epoxy composites had about 50% volume fraction of glass fabric and 50% volume fraction of epoxy resin, these filled with 0.0, 0.5, 1.0, 1.5 and 2.0 wt% of MWCNTs. A brief description for above steps has shown in the figure 1.

2.3. Scanning electron microscope (SEM)

Scanning electron microscope (SEM) type (JEOL JSM-7001F, Japan), which operates at 30 kV and its magnification continuous from 30 to 100,000X, was employed to investigate the MWCNTs dispersion in the matrix and fracture surface mechanism of the composites laminate.

2.4. Electrical properties measurement

Two-point probes technique using an advanced digital multimeter type (Agilent 34401 A) to measure the volume electrical resistance of the specimens. A silver paste has been used to minimize the contact resistance between the surface of the sample and the tip of the probe. Then, the electrical conductivity of the samples was estimated by the following equation:-

$$\sigma = \frac{L}{RA} \qquad (1)$$

Where; R is the resistance and L, A indicates the length and the area of the samples, respectively. The sample dimensions were $150 \times 150 \times 2$ mm. In addition, the strain sensitivity (gauge factor) of the fibre glass/MWCNTs/epoxy was calculated as;

$$GF = \frac{\Delta R}{\varepsilon R_0} \qquad (2)$$

Where; $\triangle R$ refers to the changing in resistance in the composites sample, R0 is the initial resistance without load ε is the flexural strain, which is obtained from the strain gauge. The strain gauge type was (L2A-XX-125LT-350) have been bonded on one side of the specimen surface. The strain gauge resistance 350 Ω and gauge factor equal 2.1.

2.5. Flexural test

The uniaxial flexural mechanical test, which is shown in figure 2(b), was performed by three-point configuration and the sample dimensions were $150 \times 150 \times 2$ mm. The tests were carried out using universal machine type Instron 5582 and at room temperature. The machine was turned under displacement control process and with a crosshead traveling speed 3 mm min^{-1}. For, each composite laminate, at least, three specimens were tested for more accurate. The flexural bending strength σ_b was calculated using the following equation:

$$\sigma_b = \frac{3P_b L}{2Wt^2} \qquad (3)$$

Where, P_b is the bending force, L, W, t are span length, sample width, and sample thickness, respectively. In terms of electromechanical characterization (piezo-resistive), the resistance was recorded during the static flexural testing.
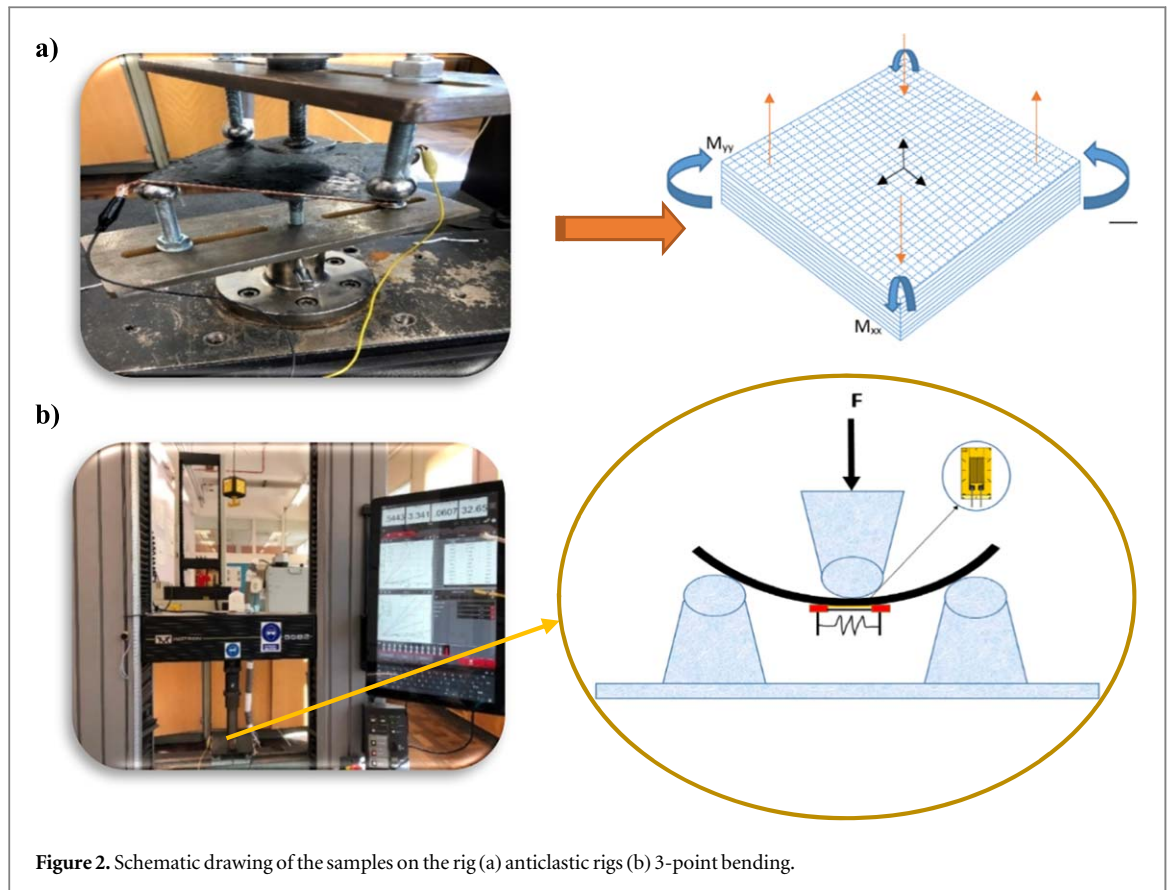
Figure 2. Schematic drawing of the samples on the rig (a) anticlastic rigs (b) 3-point bending.

2.6. Anticlastic test

This test also has been employed to study out-of-plane composites failure. This test has been achieved within the elastic limit zone to avoid sample broken and continue until fracture for monitoring the damage. The thickness of the samples is preferred to be at 2 mm for more accurate. All samples have been cut with the same dimensions, which are 150*150*2 mm. The testing has been conducted in the same universal machine (Instron 5582) which also is described earlier. The rigs were two parts and similar to shape and set as (90°) each other as shown in the figure 2(a). A span between the loading points fitted with 140 mm for both the bottom and top rigs. This span distance has been chosen to ensure that there is no localised bending leads to causes a pure bending in the centre of the plate. The ball bearing on the top of each rod had carefully fitted in the equal height to ensure that the load was equally distributed on each side of the composite plate. The preload was 10 N and then the readings were captured after auto balanced was done. The stress from the anticlastic test can be calculated from the following equation [20]:

$$\sigma_{anticlastic} = \frac{3F}{2t^2} \tag{4}$$

Where F is the load on the corner of the composite plate, t is the sample thickness. After achieving this test, the plate will take an anticlastic shape and that can be obtained by applied an external load on the four edges corner as shown in figure 2(a).

3. Results and discussion

3.1. Electrical Conductivity

Figure 3 shows the DC electrical conductivity of the composites laminate measured as a function of MWCNTs concentration by weight. It can be noted that after adding the MWCNTs to the matrix of the composites, the composite becomes conductive even at a low content of fillers. This is because of MWCNTs, which is considered high conductivity material. In fact, the electric conductivity of the composite (glass fibre/MWCNTs/epoxy) significantly increased from 10^{-14} s m^{-1} for the unmodified composite to 1.8×10^{-3} s m^{-1} at 2.0 wt% of MWCNTs content i.e. around eleven orders of magnitude. In addition, adding more MWCNTs (i.e. above 1.5 wt% of MWCNTs) showed a much smaller increased rate in composites conductivity due to the huge network from CNTs within the polymer were formed [17, 21]. Increasing of the conductivity for the composites
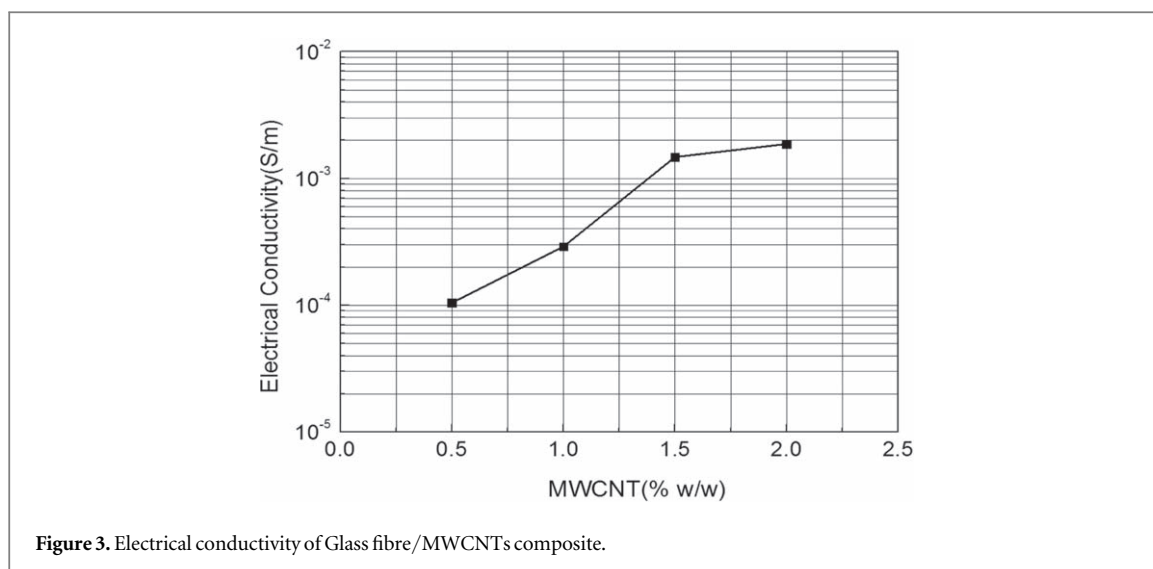
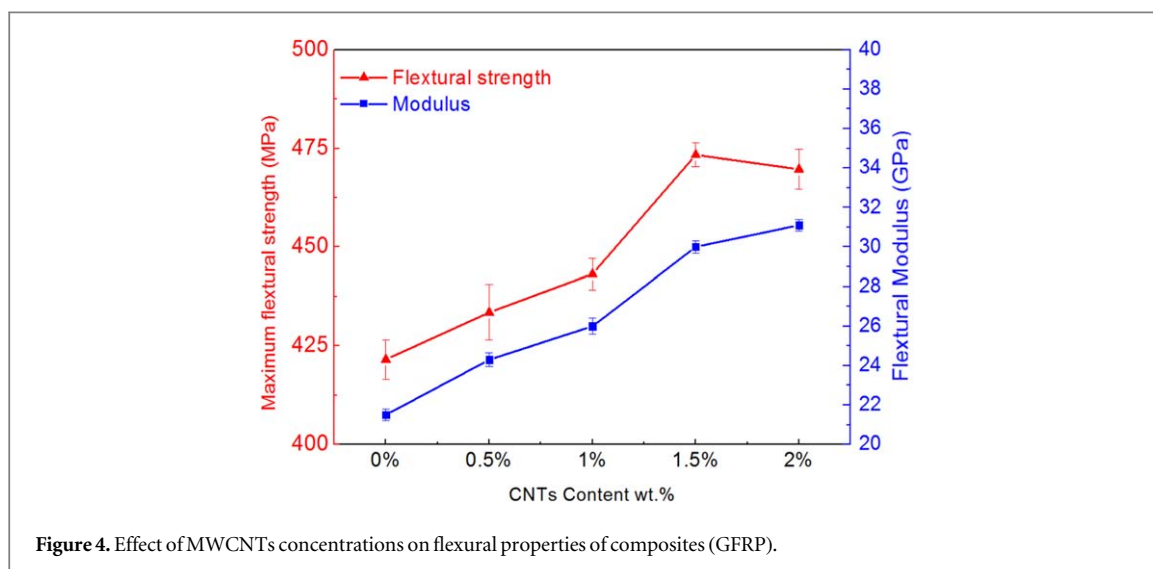**Figure 3.** Electrical conductivity of Glass fibre/MWCNTs composite.

Figure 4. Effect of MWCNTs concentrations on flexural properties of composites (GFRP).

ascribed to increase conductive paths between the CNTs and as a consequence, the gaps between the fillers become narrow. Therefore, the electron charges can pass easily between CNTs.

3.2. Effect of MWCNTs modified Glass fibre composite

3.2.1. Under flexural test

The flexural properties of composites with and without MWCNTs are shown in figure 4. It is clear that the embedding the MWCNTs in the composites improves the flexural strength and the flexural modulus as shown in figure 4. The flexural strength increased from 421.5 MPa (unmodified composite) to a maximum value 473.4 MPa (nearly 12.3%) with the addition of 1.5% of MWCNTs. Moreover, the flexural modulus also improved and increased from 21.5 GPa to 31.1 GPa at 2.0 wt% of MWCNTs content. Ming *et al* [22] studied the flexural behaviour of Glass fibre reinforced polymer with MWCNT and they reveal approximately close results in terms of flexural strength. The increment was nearly 9.2% with 0.75% MWCNTs loading as compared to unmodified composite. Rathore *et al* [23] added different concentrations of MWCNTs to composites (GFRP) and conducted the flexural test. The results showed that the samples at 0.1% MWCNT revealed a higher flexural strength, which is almost 32.8% and modulus 11.5% compared with control samples.

This improvement in strength of composites with embedded CNTs occurs due to good interfacial bonding between the CNTs and epoxy with the fibre surface and the nanocomposite distributed well between the fibre tows as shown in figure 5(a). Therefore, the loads can be transferred efficiently through the composite compound. The good dispersion of CNTs inside the matrix increased the mechanical properties of composites [24, 25]. However, the flexural strength of composites decreased slightly to 469.7 MPa when the MWCNTs content was 2.0%. This could be due to the poor interfacial bonds between the fibre and the matrix. At high
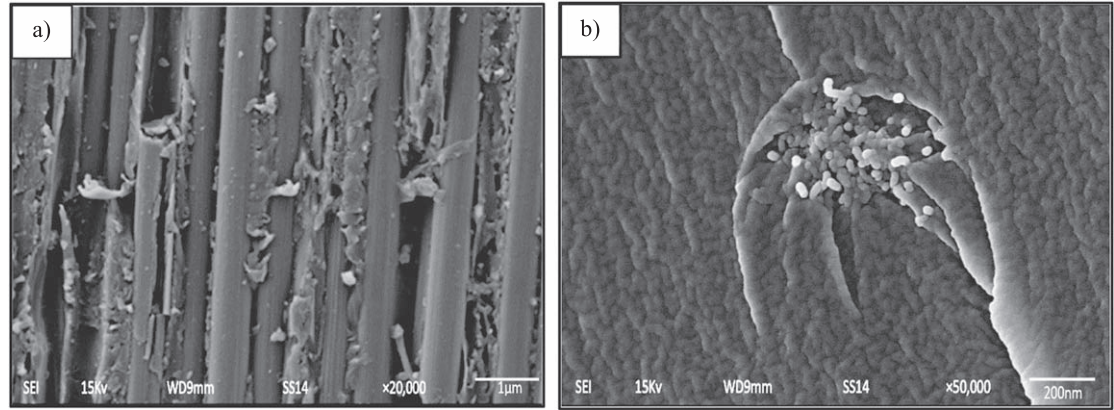
Figure 5. SEM images of (a) epoxy based MWCNTs nanocomposite between the fibre tows, (b) some MWCNTs agglomeration.

concentration, MWCNTs tend to agglomeration due to van der Waals force between them [26]. The agglomeration of CNTs as shown in figure 5(b) reduces the activity of CNTs surface area characteristics and then reduces composite properties. However, in this study, the flexural strength still stronger than that of unmodified composite laminates (GRP). Based on the results, the composite showed good mechanical properties at 1.5 wt% of MWCNTs. Therefore, it was selected for subsequent experiments testing.

3.2.2. Under anticlastic test

The anticlastic bending test is considered a mode of plate deformation in which the plate subjected to a twisting type of deformation and that characterized by two opposite curvatures. The mechanical properties including the maximum stresses and elastic modulus form the anticlastic test with different content of MWCNTs are shown in figure 6(a). It can be observed that the adding of MWCNTs contributed to enhancing both stress and the elastic modulus for the glass fibre/MWCNTs/epoxy composite. The stress increased significantly to 447.2 MPa at 1.5 wt% of MWCNTs doping and then decreased slightly to 431.5 MPa at 2.0 wt% of MWCNTs doping because of CNTs agglomerations as shown in figure 5(b). However, the stress at 2.0 wt% of MWCNTs is also still more than the control samples of the virgin composite. Moreover, with respect to modulus, it can be noted that there are two values of Young's modulus, which are obtained from this test. First one is in the x-direction i.e. (Ex) and the second is in the y-direction i.e. (Ey). By regardless the minus sign of the Ex (which is coming from the negative strain ε_x), both Ex and Ey increased significantly with increasing the MWCNTs content. For example, the maximum modulus values were 39.1 GPa which is in the y-direction and 35.1 GPa which is in the x-direction and was at 2.0 wt% of MWCNTs doping. Ideally, Young's modulus should be same in both directions because the composite is considered isotropic in in-plane but this little different could come from a small orientation of the woven fibre lamina during the hand lay-up processing.

Furthermore, to make a compare with pure flexural test, the load deflection against MWCNTs content for both test; flexural and anticlastic testing shown in figure 6(b). It can be observed that the glass fibre/MWCNTs enduring load for both tests increased with increase the MWCNTs concentration. The control composite demonstrated bearing load 647.7 N and 641.2 N, which are increased to a maximum value 776.9 N and 733.9 N for flexural and anticlastic test respectively. This is due to the incorporation of secondary reinforcement at in nano-scale level such as MWCNTs in composites lead to increase the interfacial bond between the matrix and fibre. However, the decrease of the enduring load with high MWCNTs content i.e. at 2.0% can be contributed to the inhomogeneous dispersion of MWCNTs that at higher loading system [27]. The lower load values in the anticlastic test occur due to the kinematic behaviour of the bent sheet, which has a thickness t, and is subjected to external loads at the opposite corners. There are two equal bending moments were created but with opposite signal i.e. $M_x = -M_y$ along the two pairs of edges as shown in figure 2(a). These two moments occurred around the sheet which are indicated as,

$$M_x = \int_{-t/2}^{t/2} \sigma_x \times z \times dz, \quad M_y = \int_{-t/2}^{t/2} \sigma_y \times z \times dz \tag{5}$$

While in the flexural test, only one moment affects on the sample and the other i.e. $M_x = 0$. Therefore, the loading in the flexural test is statically equal to two forces at the corners of the composite plate which are applied exactly at the two diagonal of the plate corners. In this case, the shear deformation is double and more than at flexural testing and as a result, lower bearing load for the composite under this test. However, in spite of the results from the anticlastic test were lower values than those were from the flexural test in term of stresses but the
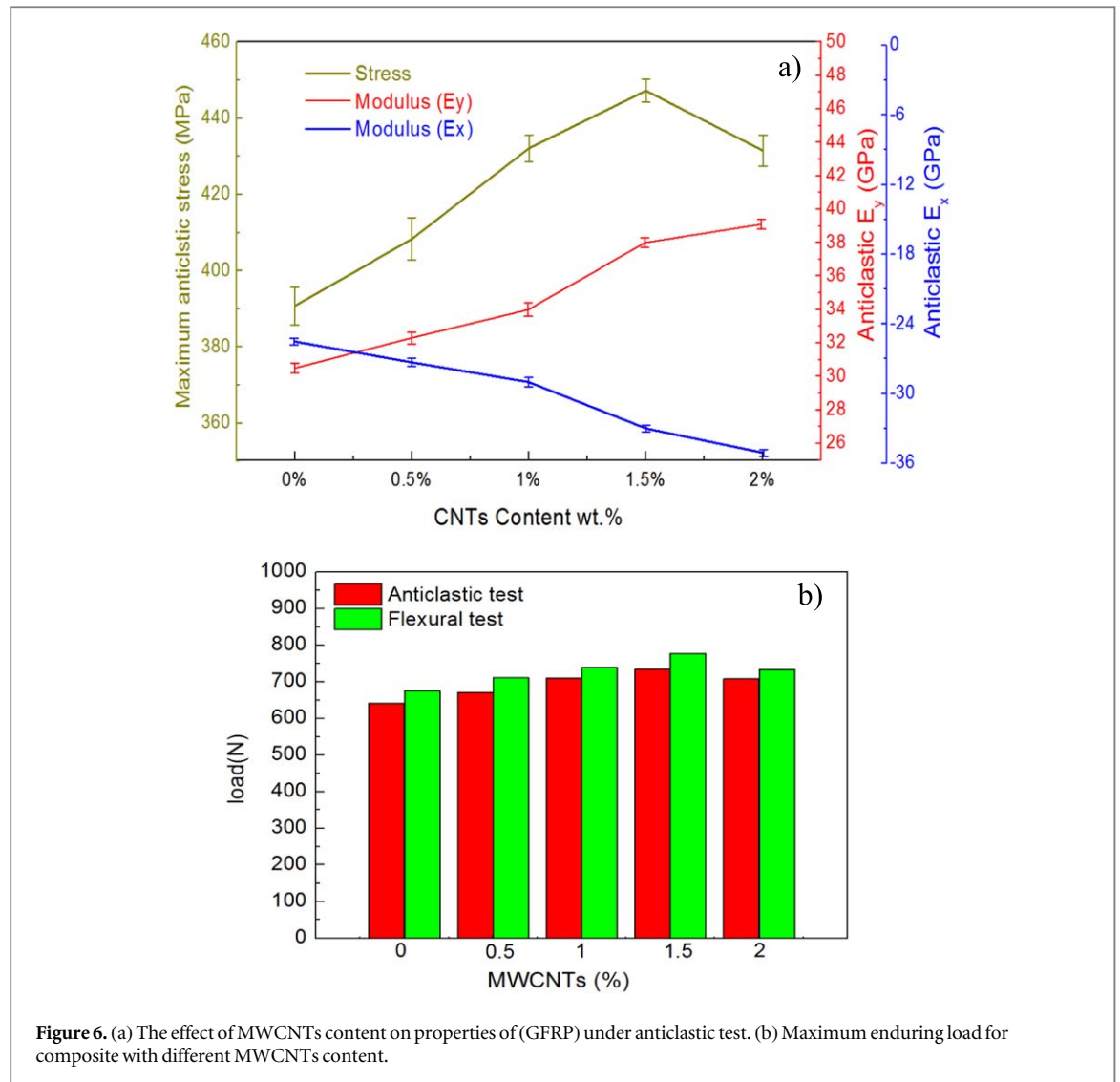
Figure 6. (a) The effect of MWCNTs content on properties of (GFRP) under anticlastic test. (b) Maximum enduring load for composite with different MWCNTs content.

values of Young's modulus were higher. This happens because the values of strains (as shown in more detail in the following section) were obtained from the anticlastic test within the elastic limit of the sample, were lower as a comparing with the flexural test strains values. For this reason, and according to Hooke's law, the values of modulus for anticlastic test become higher.

3.3. Strain deformation under flexural and anticlastic tests

When the stresses applied to the glass fibre/MWCNTs/epoxy composite lead to creating strains. Therefore, to study the difference between the two tests i.e. pure flexural and anticlastic tests in terms of those strains values. A strain gauge was bonded well on the glass fibre/MWCNTs/epoxy composite plates. This type of strain gauge can give two readings at the same time i.e. one is in the x-direction and the other is in y-directions. The strain history readings from this gauge versus displacement are indicated in figure 7. It is observed that there is one strain only was gained from the flexural test which is ε_y and there were two strains were obtained from the anticlastic test i.e. ε_x and ε_y. In general, the strains for both types of tests increased monotonically when the displacement increased as well. In more detail, at 18 mm of displacement, the strain from the flexural test shows a bigger magnitude than both strains were from the anticlastic test in about four times. This is because when the load applied on the composite plate, it was located on the four corners during the anticlastic test and that lead to creating four tension stresses and four compression stresses in the plate as shown in figure 10. These stresses were distributed in an identical mode over the glass fibre/MWCNTs/epoxy composite plate. In consequence, that contributed to minimizing the concentration load on a restricted position like that in the flexural test i.e. (in the centre of the plate only) as shown in figure 9. Moreover, the strains for the anticlastic test are opposite in sign but very close in magnitude. The negative sign for the ε_y within the anticlastic test is ascribed to the surface compression that takes place during the applied load on the corners of the composite plate in order to create the saddle shape as shown in figure 10.
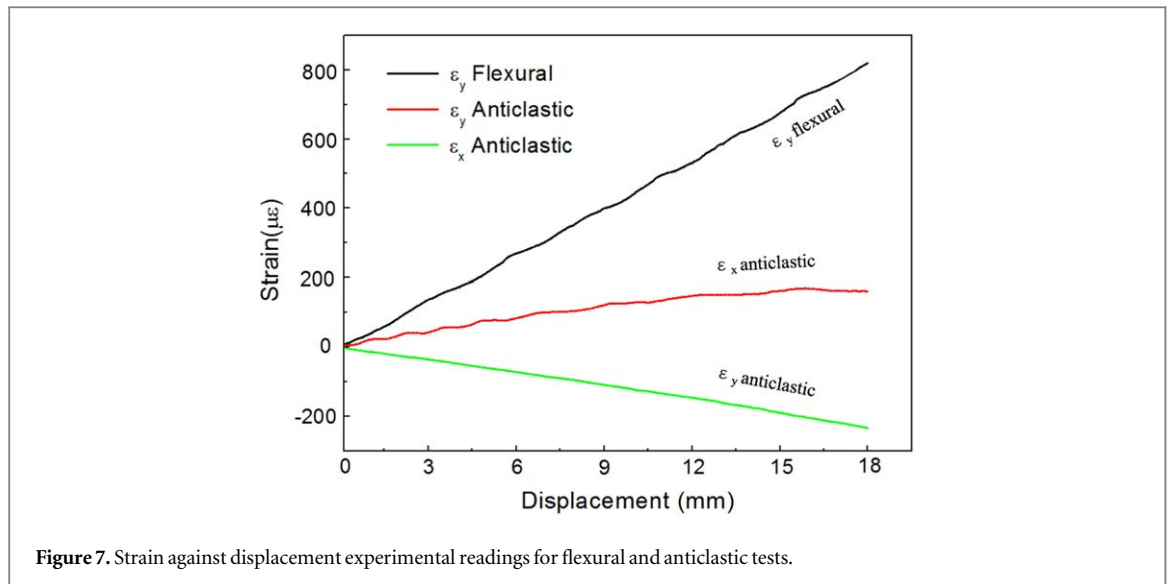
Figure 7. Strain against displacement experimental readings for flexural and anticlastic tests.
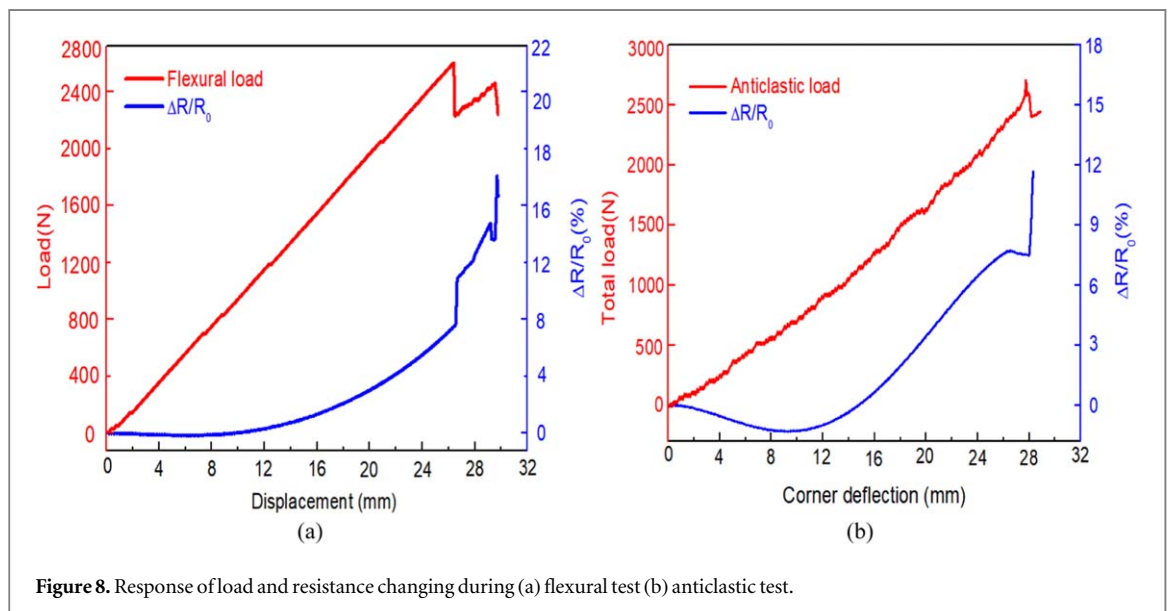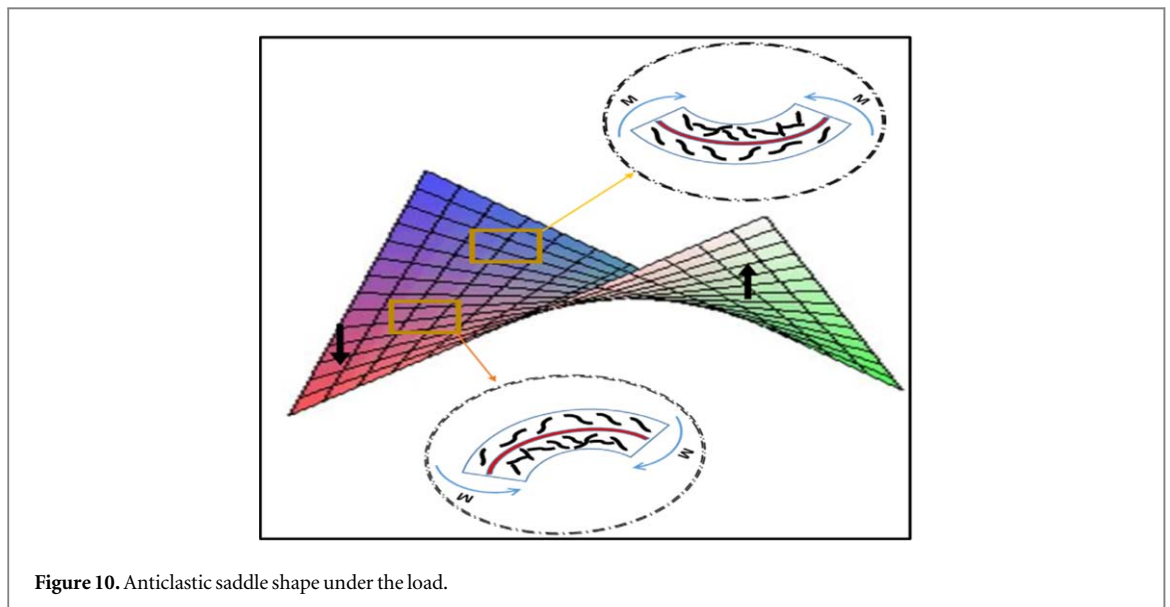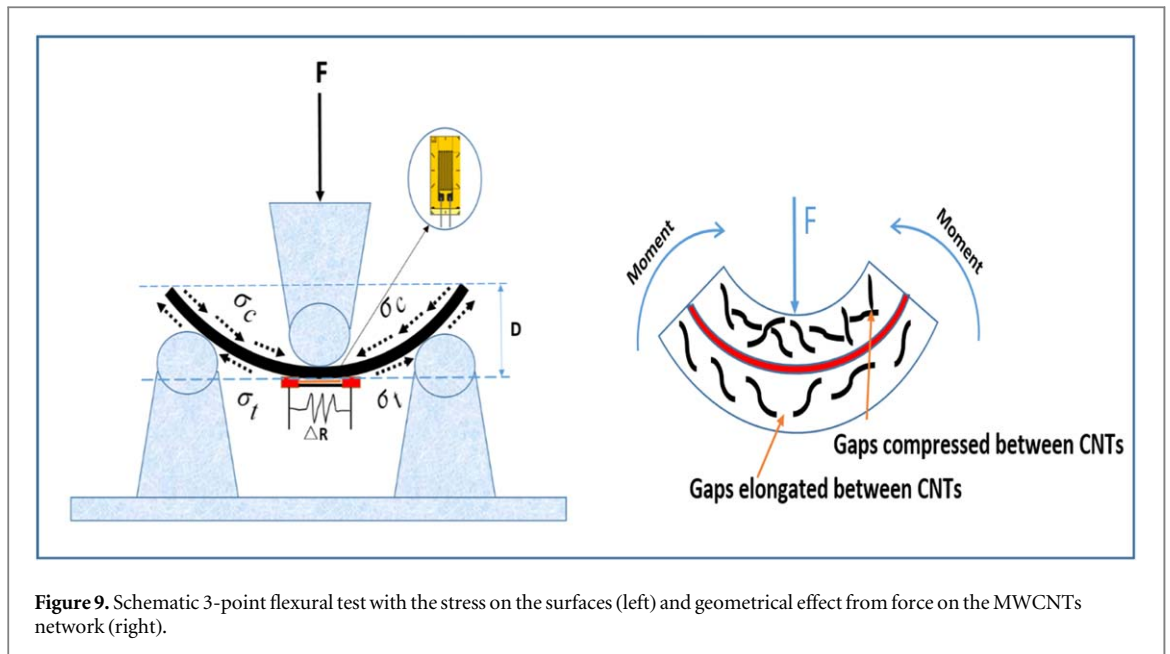

Figure 8. Response of load and resistance changing during (a) flexural test (b) anticlastic test.

3.4. Electromechanical response

The major objective of this study is to investigate the electromechanical properties of the Glass fibre/MWCNTs reinforced polymer under two different mechanical tests, flexural and anticlastic deformations. Same samples dimensions based 1.5 wt% of MWCNTs were tested under flexural and anticlastic tests with a static load. Figures 8(a), (b) shows the changing in the electric resistance and load as a function of displacement for flexural and anticlastic tests under static load. Clearly, from both tests, the changing in the resistance with increasing the load takes approximately similar trend. For the three-point flexural testing figure 8(a), it is clear that when the load increase continuously the normalized changing in resistance $\Delta R/R_0$ readings slightly reduced and demonstrate in negative values. This reduction in the readings continue up to 8 mm of composite plate center displacement and then increase sharply in nonlinear behaviour until the sample start to break. To explain that, the upper surface of the sample under flexural load is compressed and the under surface in tension state see figure 9. Therefore, when the load starts to build up, there is a slight reduction in the gaps between MWCNTs within them network structure and this lead to reduce the resistance. Above 8 mm deflection and due to the increase in the applied load, the layers close to the surface are exposed to large tension. This is the reason for the increasing the resistance due to the internal distance between the MWCNTs expanded. These findings are consistent with other studies such as [2, 28, 29].

In the anticlastic test (figure 8(b)), the changing in the resistance also shows negative readings and gives a maximum value at 9.3 mm corners deflection and this is considered more than at the flexural test in about

Figure 9. Schematic 3-point flexural test with the stress on the surfaces (left) and geometrical effect from force on the MWCNTs network (right).

Figure 10. Anticlastic saddle shape under the load.

69.7%. This due to in the anticlastic test, there are two combined moments occur around the composite plate corners. These lead to creating two compression and tension surfaces at the same time as shown in figure 10.

Therefore, the gaps between the MWCNTs in the out-plane region were reduced in two compression surfaces and increased in two tension surfaces and this cause a huge changing in resistance compared with the flexural test. In general, the signal should be very clear to detect the damage from the early stage of initiated crack. For example, In a previous study [30], which is about the carbon fibre composite, it was not able to detect the crack from an early stage because there is no change in composite resistance during the applied load. Therefore, in the glass fibre based MWCNTs composite have a good ability to detect the damage by changing the resistance from the point that the load start to increase as shown in figure 8.

3.5. Strain sensitivity

The local deformation in both flexural and anticlastic samples was detected using a conventional strain gauge and fixed on the surface of the composite plate. The history-recorded data were used in equation (2) to calculate the strain sensitivity or (GF). Figures 11 and 12 show the normalized changing in resistance as a function of the strain for flexural and anticlastic test respectively. It is clear that the correlation between the $\Delta R/R_0$ and the strain in the flexural test indicated as almost linear behaviour more than that at the anticlastic test. This might because there is only one moment in this test that affected on the curvature of the plate while in the anticlastic
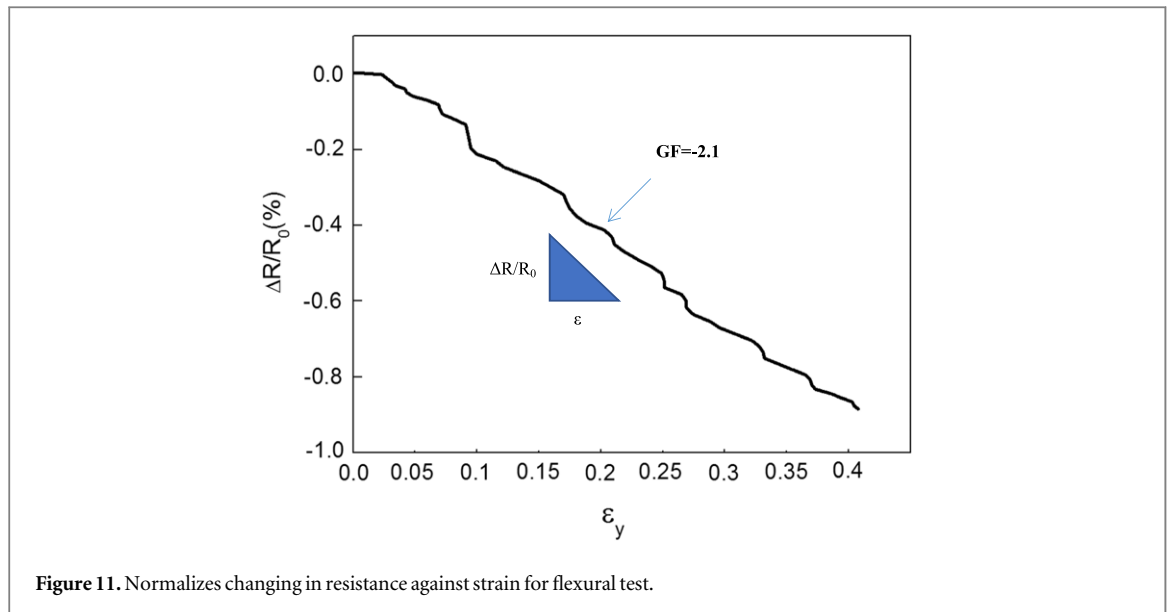
Figure 11. Normalizes changing in resistance against strain for flexural test.
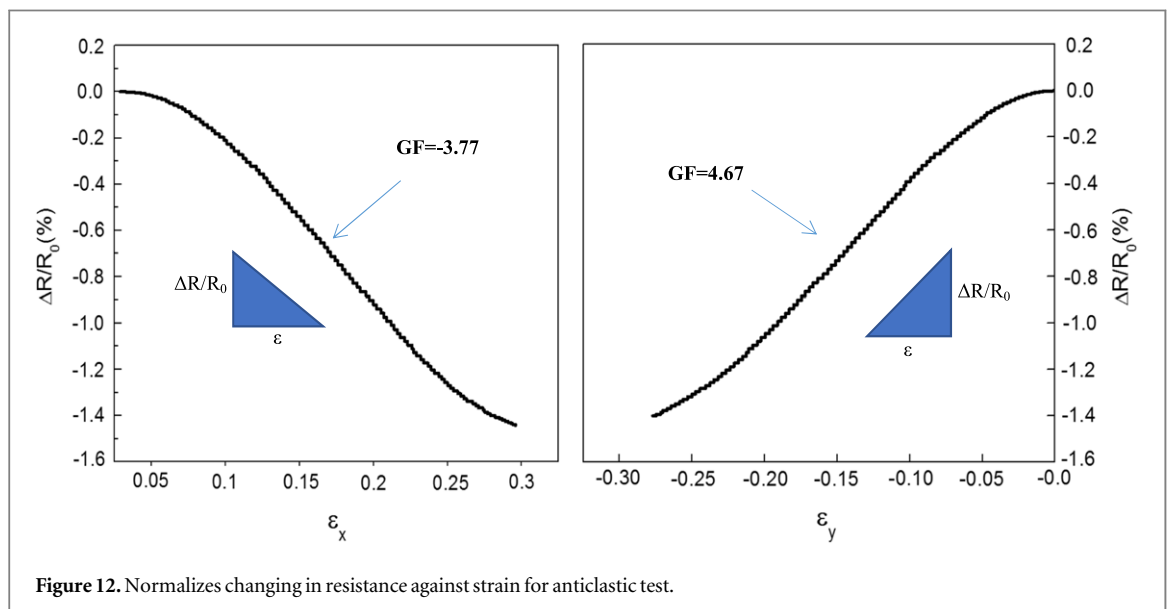

Figure 12. Normalizes changing in resistance against strain for anticlastic test.

test there were two moments available to make the curvatures as figure 10. Moreover, it can be also observed that there are two strain values i.e. ε_x, ε_y in the anticlastic test have obtained while one only i.e. ε_y in the flexural test. This means there are two GFs in anticlastic test and one only is in the flexural test. In addition, the strain sensitivity in the flexural test is equal to -2.1 and lower than with those obtained from the anticlastic tests about 47% as shown in figure 12. This is because of the number of curvatures formed and their features that made from the applied load. The negative sign for the GF in the flexural test and in the anticlastic test in x-direction comes from the compression deformation on the curvature surfaces. In addition to that, during the compression deformation, the internal distance between MWCNTs decreases and consequently the resistance decreases (because the electron can pass easily between the CNTs) as shown in figure 10. Perfectly, the magnitude of the sensitivity strain in the anticlastic should be same in both directions but in this study could be because of the strain gauge position was not exactly at the centre of the diagonal line of the composite plate and its sticking on the plate surface could be not very well.

4. Conclusions

The damage in a structure that made of composites materials is still a critical issue. A self-sensing composite was made by adding MWCNTs inside the epoxy and the mixture considers a matrix for glass/fibre composite. Two different tests have applied on this type of smart composites and the main findings were:

The mechanical properties of the smart composite were considerably enhanced after using the MWCNTs as a second filler.

It is observed that at 1.5 wt% of MWCNTs the strength was maximum and going above it, the agglomeration appeal at 2.0 wt% of MWCNTs and the mechanical performance reduced excepted young modulus.

The normalized changing in resistance ($\Delta R/R_0$) reduced in the flexural test and gives a valley value at 8 mm of displacement and then raise up sharply. While at the anticlastic test, the normalized resistance reduced in about 69.7% more than at flexural test and that was at 9.3 mm of displacement and then increased into positive zone also. This is due to the compound surfaces is deformed to the saddle shape.

The strain sensitivity was one value only in flexural test and equal -2.1. This value was less value than from the both strain gauges that were obtained from the anticlastic deformation in about 47%.

The stresses that obtained from the flexural test were higher than the stresses from the anticlastic test i.e. the maximum value was 473.4 MPa for flexural and was 447.2 MPa for the anticlastic test.

The elastic modulus values were obtained from the anticlastic test were two and close to magnitude but in opposite sign and both were higher than the elastic modulus at the flexural test.

The strains deformation in the flexural test was higher in values than both strains values in the anticlastic test in about four orders.

Therefore, the obtained results from the both type of tests give a deep vision to understand the behaviour of the composites based MWCNTs in terms of loads, stress, elastic modulus, strain deformation and strain sensitivity under complex deformation tests.

Acknowledgments

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