



Damage sensing and mechanical properties of a laminate composite material containing MWCNTs during low-velocity impact

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Abstract:

In this work, we present a new criterion, unlike other attempts, to evaluate and quantify the degree of damage of composite material when it subjected to a sudden impact load. Our criterion exploits the high intrinsic electrical conductivity property of the Multi-walled carbon nanotubes (MWCNTs) after dispersing different concentrations of them (0, 0.5, 1.0, 1.5 and 2.0 %) in the epoxy matrix of a glass fibre composite. Following this goal, the low-velocity impact and flexural after impact (FAI) tests on the MWCNTs-glass epoxy (i.e. MWCNTs-GF) nanocomposite were evaluated. At the same time, the changes in its electrical resistance were measured. The results showed that the properties of the self-sensing composites were significantly affected by impact energy. The damage after impact causes an increase in the electrical resistance of the MWCNTs-GF nanocomposite and increases with increased impact energy. In addition, the samples containing a high concentration of MWCNTs showed lower damage sensitivity under all impact energies levels as compared with the samples contain a lower MWCNTs concentration. Therefore, the results presented in this work have shown that it is possible to associate the change in electrical resistance of the MWCNTs-GF nanocomposite with the degree of damages caused by impact load.

Keywords: MWCNTs; Damage sensing; Low-velocity impact; Mechanical properties.

تحسس التلف والخواص الميكانيكية للمواد المركبة المدعمة بانابيب الكربون النانوية تحت تأثير صدمة واطئة السرعة

الخلاصة:

في هذا البحث، نحن نعرض معيارية جديدة لانتشبه الطرق التقليدية المتبعة بالفحص، لتقييم مقدار الضرر بالمواد المركبة عندما تتعرض الى حمل صدمة مفاجئ. في هذه المعيارية تم استغلال الخواص الكهربائية وقابلية التحسس الذاتي لانابيب الكربون النانوية المضافة بنسب مختلفة (0.5% و 1.0% و 1.5% و 2.0%) الى اساس المواد المركبة (الايوكسي). بعد ذلك تم اجراء اختبار الصدمة بسرعة قليلة وكذلك اختبار الحني قبل و بعد اختبار الصدمة. بالإضافة الى ذلك، تم اختبار مقدار التغيير بالمقاومة الكهربائية للمركب المتحسس اثناء اختبار الصدمة. تبين من خلال النتائج العملية ان الخواص الميكانيكية والكهربائية للمركب المتحسس تتأثر بشدة عندما يحدث حمل الصدمة ويزداد التأثير بزيادة مقدار حمل الصدمة. بالإضافة الى ذلك ان المركب المدعم بانابيب نانوية 2% اظهر نتائج اقل من ناحية التحسس مقارنة مع نسبة الاضافة الاخرى (وخصوصا النسب الواطئة). لذلك بناء على ضوء النتائج المعطاة تبين انه هناك امكانية تنبؤ التغيير بالخواص الميكانيكية للمواد للمركبات بعد الصدمات بشكل مباشر من خلال التغيير بالخواص الكهربائية وبدون الحاجة الى معدات فحص اخرى.

1.1 Introduction:

Composite is defined as the material that made from two or more constituent parts with physically distinct characteristics, which when combined give superior properties than used as individually. Due to their superior properties, over the recent decades has led to composite materials being widely used in ever-increasing applications in different fields such as marine, oil and gas and space applications [1, 2]. However, composite materials are faced with some big issues such as sudden impact during service life. This impact load leads to creating visible and non-visible cracks inside the structure [3].

These cracks will develop during the time and finally, a sudden catastrophic failure for the structure occurs. The difficulty to detect damages in composite materials has created a reliance on regular non-destructive testing (NDT) and careful documentation of any known impacts to a composite. In addition, current NDT of composite materials may mean removing the part from service and this is time-consuming and expensive. Therefore, it is necessary to develop a new high-efficiency non-destructive method to detect damage occurred from the impact.

Currently, there are some techniques of impact detecting in composites materials, for example, either attached on the structure surface such a strain gauge or by embedded them within the composite material such as fibre optic sensors [4] piezoresistive sensor [5]. Those methods of detecting damages were good to detect the damage for small area form the structural and also high number of strain gauge were needed to cover the large structure. In addition, Lots of optical fibres and sensors embedded into the composite leaves spaces around the fibre which can increase the likelihood of resin rich regions which is a detriment to the mechanical properties of the composite. Other conventional NDT methods like X-ray are expensive and require the part to be taken out of use to be inspected because much of the x-ray or CT equipment is lab based [6]. For the more detailed imaging using CT scanners, this can involve the use of a large room and very expensive equipment, for this reason, these kinds of tests cannot be carried out regularly and hence lose their effectiveness.

Carbon nanotubes (CNTs) is considered one of the promising materials that could be used as a nano-sensor additive to the composite materials to make it electrically conductive and then used as a self-sensing composite. CNTs possess superior mechanical, thermal and electrical properties [7, 8]. Therefore, after adding CNTs as a nano-filler conductor sensor, even with lower amount into a resin matrix of the composite, will lead to build huge electrically conductive networks around the fibres tows within the composite material. When this self-sensing nanocomposite is subjected to an external impact energy, the CNTs networks will broke and if the impact energy is high, cracks in the matrix will be develop and propagate quickly. These cracks lead to destroy the networks configuration of the CNTs and as a consequence, the electric resistance of the self-sensing nanocomposite will alter as a piezo-resistive influence [5].

Several studies about the behavior of self-sensing composite when they were subjected to impact loading were performed. For example, Naghashpour *et al* [9] added Carbon Nanotubes (CNTs) to the matrix of the glass fiber laminate composite material. They found that electrical resistance measurements through the glass fibre/CNTs composite thickness are clearly influenced when the composite structure is subjected to impact load. Gao *et al* [10] also added CNTs to the epoxy resin in order to make a self-sensing nanocomposite matrix for the laminate composite.

The specimens were subjected to different levels of impact load and they found that the composite electric resistance increases with increases in the impact loads. Moreover, Monti M *et al* [11] distressed CNTs as a conductive filler in the matrix of the glass fibre composite to make also the composite matrix electrically conductive. Then the composite samples surface was subjected to a low-velocity impact in order to create damage. The results showed that the composite matrix is able to give an electric signal directly when subjected to an impact load. This due to the conductive networks of the CNTs sustaining damage after applying the impact load. Arronche *et al* [12] embedded a low concentration of CNTs to the glass fiber composite matrix to make it electrically conductive and working as self-sensing nanocomposite.

The electric resistance signal was recorded before and after applied an impact load. Their results proved that when the sample is subjected to low impact energies, the electric measurement signal, which caused by the impact, is very small to be recorded by the device recorder. From the aforementioned studies, it can be concluded that after adding CNTs to the matrix of the glass fibre composite and when the sample subjected to impact load, the damage could be detected and sensed. However, the above studies, they have focused only on the behavior (i.e. change) in electric resistances before and after the sample subjected to impact. They did not making any evaluation method or a correlation relationship between the mechanical properties and the changing in electrical resistance of the structure after impact event. Here, in the present study, we develop a simple criterion to evaluate the correlation between the changes in the sample electrical resistances and the energy absorbed by the target after low-velocity impact energy.

The results obtained can show a clear way to fabricate a self-sensing composite in order to sense and detect any damage, which could take place due to impact as an on-line technique. This idea will save lives after preventing or reduce the sudden catastrophic failure.

2. Experimental methods:

2.1 Materials

To fabricate the CNTs/glass fibre self-sensing nanocomposite, the CNTs, its SEM image shown in Fig. (1) **SEM image of as-received**

(), purchased from (US research nanomaterials company, USA). The CNTs prepared by chemical vapour deposition (CVD) according to company data supplier. The average diameter and average length of these CNTs were 10 nm and 50 μm , respectively. Their specific surface area was about 233 ($\text{m}^2 \cdot \text{g}^{-1}$) and also their purity was more than 95%. Regards the glass fibers used were plain woven (290 g/m^3). The matrix of the composite was epoxy resin type EL2 with its hardener type AT30 together have a slow cure time. Both epoxy and hardener material were supplied from (Easycomposites, UK). All the experiment work have carried out at Plymouth University.

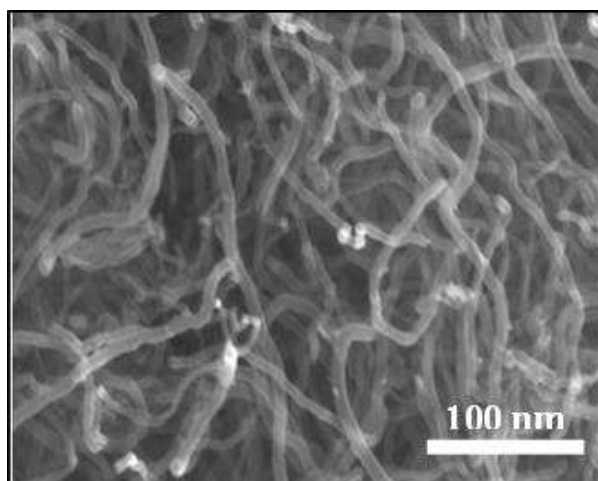


Fig. (1) SEM image of as-received

2.2 Preparation of CNT/GF nanocomposites

Due to the agglomeration of some CNTs in its natural case, therefore, a pre-calculated amount of CNTs was mixed with 60 ml of organic acetone in a glass beaker in order to disperse them. Sonication process was used by ultrasonic device type BR-20MT-10L, 1000W was then performed for 12 minutes. The mixture of (CNTs + acetone) was then mixed with the required weight of the matrix (i.e. epoxy resin) and the obtained solution was sonicated again for 15 minutes. Before the sonication process start, the beaker was placed in an ice container to keep the mixture in normal temperature. After complete the

sonication process, the mixture was put in a furnace for two hours at 70 °C in order to remove the residual acetone. Following this step, the curing agent (i.e. hardener) was then added to the solution at a ratio of (100 epoxy: 30 hardener) by weight. Hence, the solution was degassed for 15 minutes in order to remove all the trapped air bubbles which probability insert during preparation procedures. It is well known that, the mechanical properties of a CNTs-GF nanocomposite definitely depend on the laminates geometry, especially the thickness. Therefore, the thickness of the specimens should be 2 mm based on the British Standard BS EN ISO 14125 (1998-2011). For this purpose, 12 layers of glass fibre mat fabric with 130 g of the nanocomposite were prepared in order to obtain 2 mm thickness of CNTs-GF nanocomposite specimens. The nanocomposite (i.e. matrix) was placed using a rotating roller between the laminates layers according to hand lay-up principle. After finishing the hand lay-up steps with different concentration of CNTs (i.e. 0, 0.5, 1.0, 1.5 and 2.0) %, all of the composite laminates were put inside a vacuum bag at room temperature for 24 hours in order to pull out any trapped air bubbles and unnecessary resin. Post-curing, as a final step, for the panels was at 70°C for 14h.

3. Characterizations:

3.1 Low-velocity impact testing

The main aim of this type of test was to create damage in the CNTs/GF self-sensing nanocomposite to understand the behaviour of the self-sensing nanocomposite after it subjected to impact load. The impact tests on samples were carried out using a drop-weight impact technique, see Figure (2).

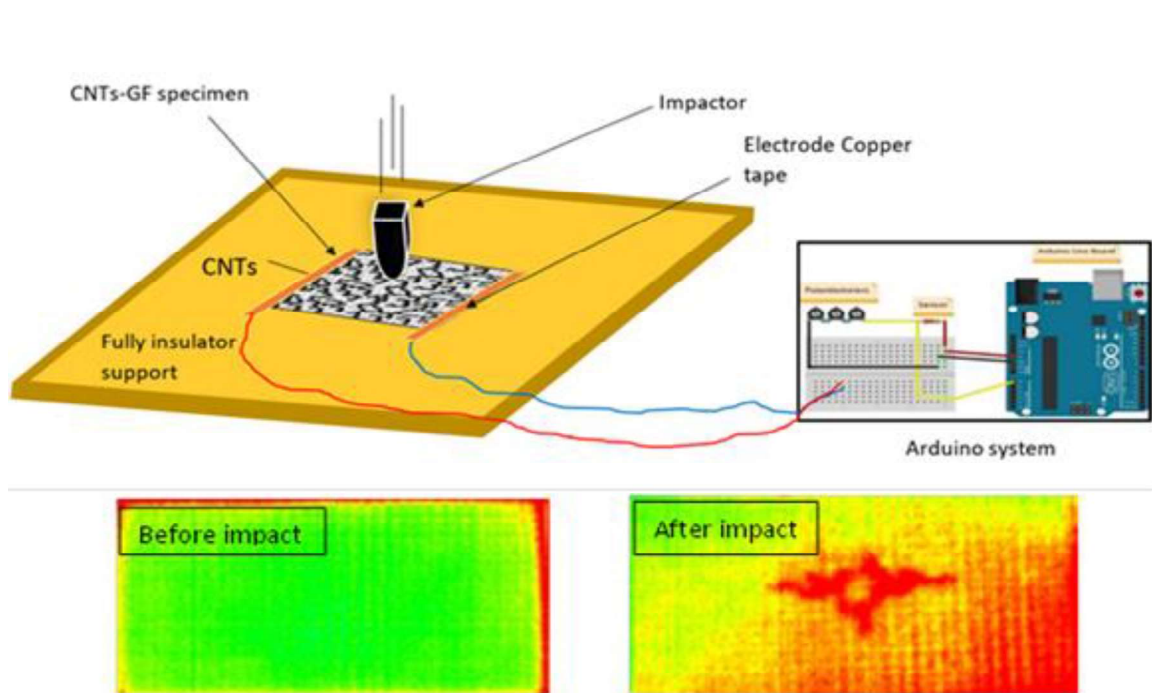


Fig. (2) Layout setup of the impact test of CNTs-GF nanocomposites

The laminate panels samples were cut with the dimensions of (100×50×2 mm). This size were depended based on some pre-experiment tests which were conducted on different samples dimension in order to reach the suit size that prevent the sample from fully broken. Each specimen was clamped to a flat supporting fully isolator frame and then the impactor falls exactly at the specimen centre. The impactor head diameter was 16 mm and was hemispherical in shape. The details of the impact teste parameters conditions are summarised in Table (1). The magnitude of the impact velocity (i.e. initial) was calculated from the first position (i.e. height) from which the impactor was fallen onto the outer specimen surface. Moreover, regards the rebound velocities calculation, the tests event were recorded by a high - speed digital camera type Sony-RX10-II, Japan, which can capture 1000 images per second.

Table (1) Summaries of the low-velocity impact test conditions

Drop mass (g)	Drop height (cm)	Impact velocity (m/s)	Impact energy (J)	No. of samples
2000	50	3.12	9.81	3
2000	100	4.42	19.62	3
2000	150	5.42	29.43	3

3.2 Flexural testing before and after Impact

The flexural after impact test was conducted on the samples to evaluate the damage degree after the impact test. A three-point flexural test method was performed by a 100 KN universal testing machine (type Instron 5582/UK195). The samples were subjected to a flexural load with a constant crosshead speed of 1.5 mm.min^{-1} with 60 mm span distance. The flexural strength can be calculated using the following formula:

$$\sigma_f = \frac{3 \times f \times l}{2 \times w \times t^2} \quad (1)$$

And the flexural strain can be calculated from following formula:

$$\varepsilon_f = \frac{6ht}{l^2} \quad (2)$$

Where, f , l , h , w , t and are the applied flexural force, span length, maximum deformation, sample width, sample thickness, respectively.

3.3 The electrical resistance measurement

Before the impact test, the electrical resistance of all the samples was measured using a two-point probe technique. A multi-meter type Keithley 6517B, USA, was used to read the electrical resistance for each sample. The contact resistance between the probes tip and surface of the sample were minimised by paint the sample edges with high purity silver paint. Then, two copper tape electrodes were attached on the edges of the sample for contact purpose. The samples electrical conductivity was then calculated according to the following formula:

$$\sigma = \frac{L}{RA} \quad (3)$$

Where L , R and A is the length, electrical resistance and the cross-section area of the specimen, respectively. In terms of the electrical resistance recording data during the

impact test, because the test occur very quick (i.e.in milliseconds). Therefore, an Arduino software was employed for this purpose.

3.4 Scanning electron microscopy

CNTs distribution in the matrix of the self-sensing nanocomposite was examined by scanning electronic microscopy (SEM) type JEOL JSM-7001F, Japan. The SEM was operating at an accelerating voltage of 15 kV to obtain an appropriate magnification to very clearly visualise of the MWCNTs inside the sample matrix. All the fractured samples were firstly coated with a micro-thin gold layer by the sputtering device to obtain on very clear SEM image.

4. Results and Discussion:

4.1 The electrical conductivity of the nanocomposite

Figure (3) shows the measured electrical conductivity (σ) of the self-sensing nanocomposite samples as a function of CNTs wt.% concentration. The specimens' electrical conductivity were measured at the case of without any strain applied (i.e. at the zero load).

It can be seen that from the curve that the electrical conductivity for the specimens start to raise after filled with 0.5 wt.% CNTs concentration. However, around this concentration, conductive network pathways were not enough to produce a high electric conductivity.

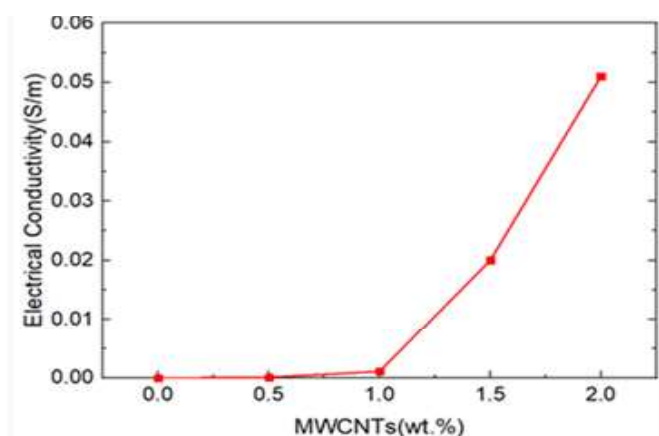


Fig. (3) Variation of electrical conductivity of self-sensing composite with wt.% MWCNT concentration

It is well known that, the point at which the conductive nanocomposite changes from an insulator state to be electrically conductive is called the percolation threshold [13-15]. At this point, the CNTs conductive networks are formed. Previous studies [16, 17] have also been observed that. After the percolation threshold, a gradual increased in the nanocomposite electrical conductivity approaching $5.3 \times 10^{-2} \text{ S.m}^{-1}$ at 2.0 wt.% of MWCNTs concentration. This is attributed to at high CNTs content, the conductive networks numbers increased inside the epoxy resin (i.e. matrix) of the self-sensing nanocomposite samples, and as a consequence, the electrons can pass easily through them. In the present study, the high efficient electrical conductivity of the nanocomposites is considered a main key point to be used for in-situ monitoring process during and after the impact test. High efficient properties of the self-sensing nanocomposite specimens can be obtained from the degree of dispersion of the CNTs inside the matrix [18, 19]. This has been proved by other studied [20].

4.2 Mechanical properties of CNTs-GF nanocomposite before impact

Figure (4) (a, b) shows the measured mechanical for specimens filled with different concentrations of CNTs. It can be seen that the ultimate flexural strength of the CNTs-GF nanocomposite significantly increased together with a (linear) increase in the flexural modulus compared to the unmodified composite. When 1.5 wt.% of CNTs were added to the matrix (see Figure 5a) of the composite, the ultimate flexural strength showed a value of $\approx 504 \text{ MPa}$. While the maximum flexural modulus occurred at 2.0wt.% of CNTs by $\approx 23.9 \text{ GPa}$. The improvement in CNTs/GF nanocomposite mechanical properties can be attributed to the presence of the CNTs. This improves the load transfer between the matrix and the reinforcing glass fibres [21, 22]. In addition, it was proved that the mechanical properties of the nanocomposite can be enhanced when the CNTs are well and uniformly distributed inside the matrix of the nanocomposite, and this also occurred in this study as (Figure 5b).

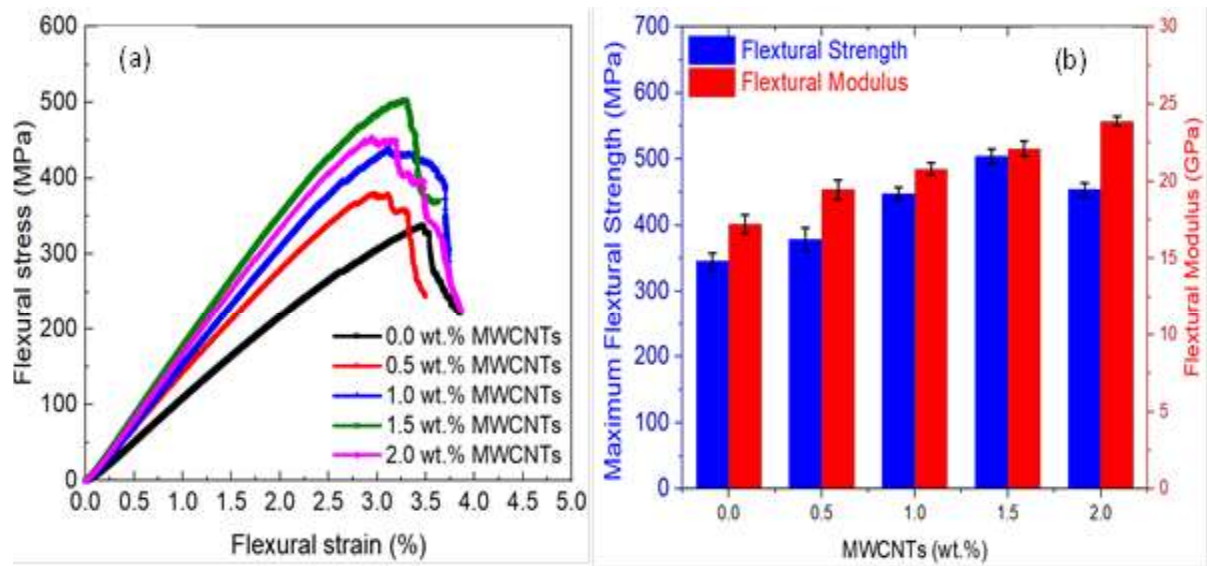


Fig. (4) Mechanical properties of self-sensing nanocomposite as a function of MWCNTs content (a) flexural Stress-strain (b) maximum flexural strength

However, the CNTs/GF nanocomposites flexural strength was reduced to ≈ 454 MPa at CNTs concentration 2.0 wt.%, but still above the unmodified composite flexural strength. This reduction can be ascribed to some of CNTs agglomeration which generated inside the matrix (Figure 5c). This agglomeration caused to lack of the load to transfer well between the matrix and CNTs in addition to glass fibre [21, 23].

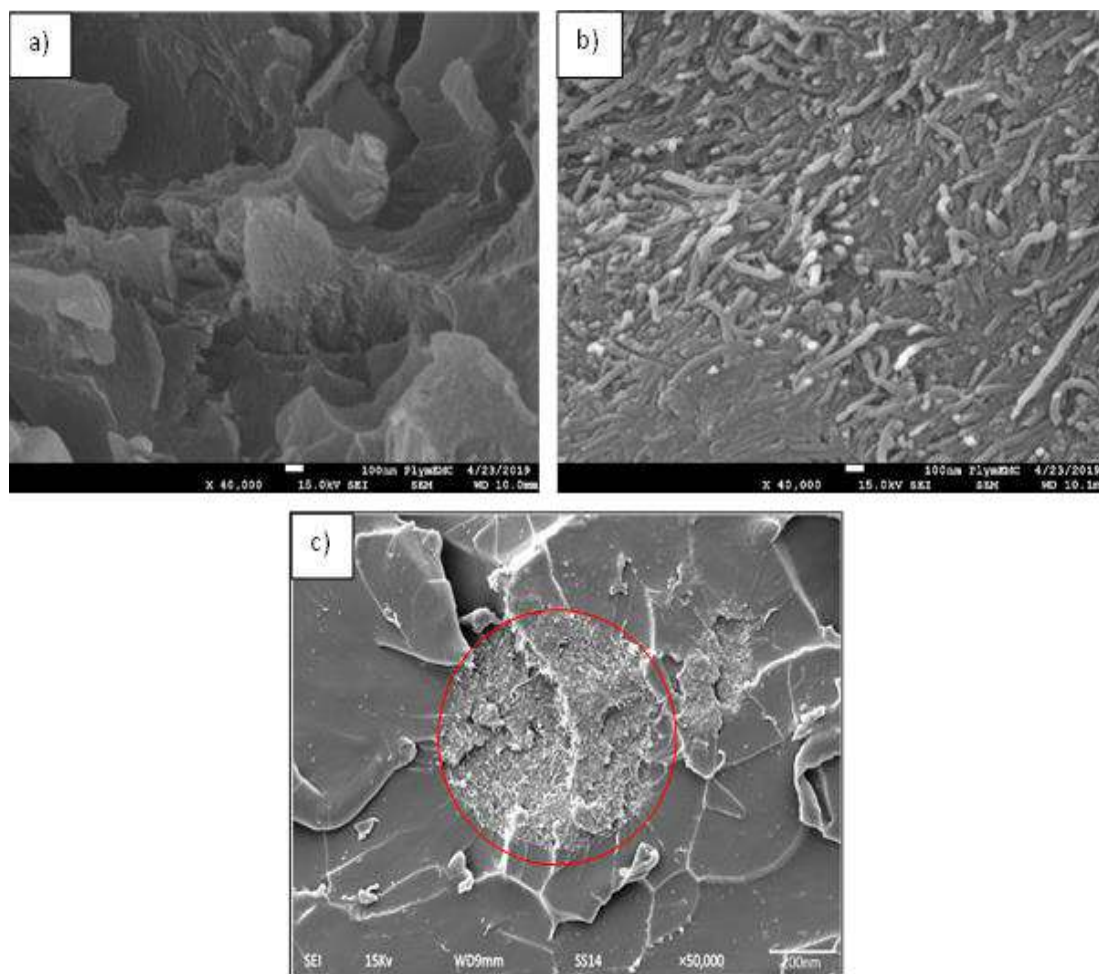


Fig. (5) SEM images of (a) unmodified epoxy resin (b) A good dispersion of 1.5 wt.% MWCNTs nanocomposite within the matrix (c) agglomeration of the MWCNTs inside the matrix at CNTs concentration of 2.0 wt.%.

4.3 Residual mechanical properties (flexural) after impact

Figure (6) shows the normalised residual flexural and modulus properties as a function of different impact energy values. As expected, the impact load test caused a clear reduction in both flexural strength and flexural modulus of the self-sensing nanocomposites. In addition, it can be observed that the nanocomposite's residual flexural strength is decreased when the impact energy increases. Moreover, the residual flexural modulus showed a less degree of reduction in compared to the flexural strength. The maximum reduction is $\approx 15.8\%$ and $\approx 24.9\%$ for the modulus and the residual strength, respectively. From the result, it can be inferred that the residual flexural strength is very sensitive to

cracks initiate when compared to the flexural modulus. These results have also been observed in previous studies [24-26].

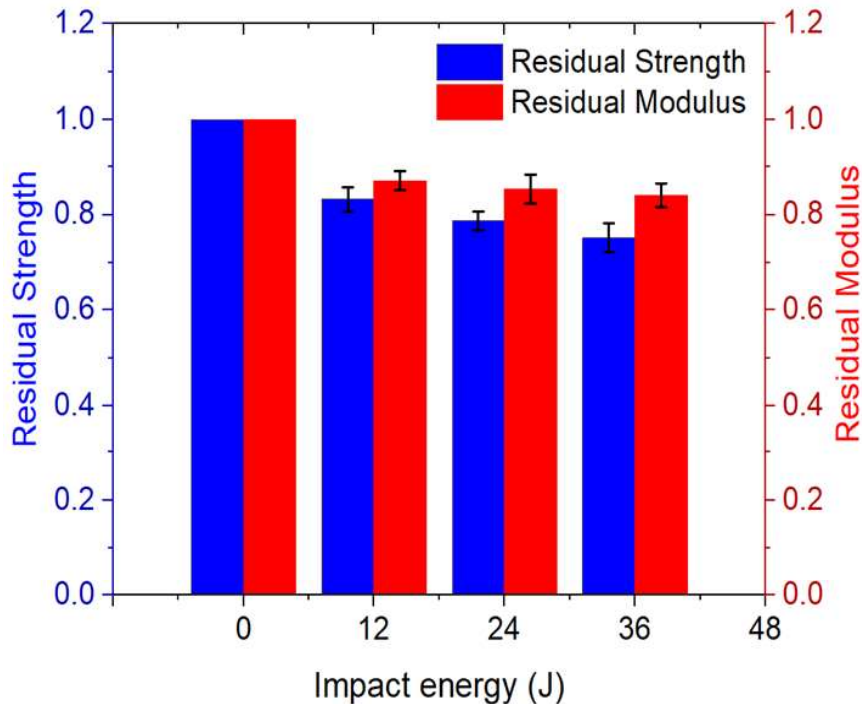


Fig. (2) The normalised flexural properties as a function of impact energy for specimen containing of 1.5 wt.% MWCNTs.

4.4 Electrical properties of the MWCNT-GFRP composite

The decreases in flexural mechanical properties of the self-sensing nanocomposites match with an increase in electrical resistance that occurs during an impact load when MWCNTs are added in the composite laminate. The actual resistance of the laminate was calculated by attaching it to a digital multimeter and using the $\text{Volt} = \text{current} \times \text{resistance}$ equation to calculate the sample resistance. In this test, 0.5 and 2.0 wt.% of MWCNTs were chosen to study the changes in electrical resistance during and after the impact load occur. Figure (7) shows the changes in electrical resistance of the sample after impact energy events. In the initial resistance reading before impact is of roughly 5.1k Ω . After impact, the increase seen is 0.08% increase after a 12J impact, 0.33% increase after the 24J impact and a 0.52% increase after the 36J impact. The increase coincides with greater impact energies,

and this shows that the increase in resistance is due to MWCNT breaking and disrupting the path for current to run through the laminate [9, 27].

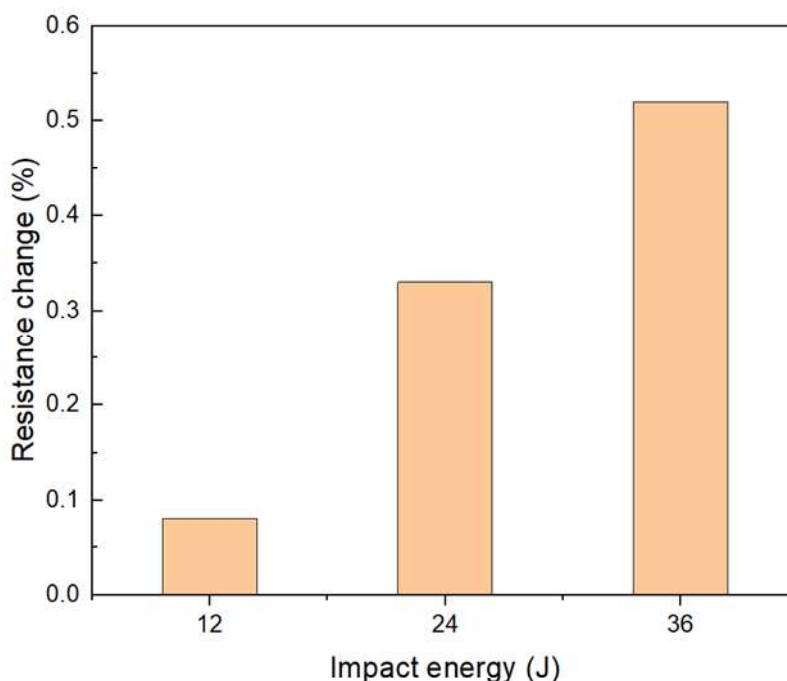


Fig. (3) Variation of electrical of self-sensing composite as a function of energy impact for the samples containing 2.0wt% of MWCNTs.

Moreover, the percentage increase in electrical resistance of the self-sensing nanocomposite based at 0.5 % wt. MWCNTs is slightly greater compared with sample with high MWCNTs content. From Figure (8), it can be noted that the increase in electrical resistance when the sample subjected to 12J, 24J and 36J impacts was 0.25%, 0.53% and 0.74%, respectively. The possible reason for the change resistance is lower in the composites with high MWCNTs content may be due to the enhanced mechanical properties and therefore, the decrease in damage level [28]. In general, when the sample exposed to impact load, the MWCNTs conductive network destroyed and the distance between MWCNT-MWCNTs increase. This increasing in the gaps lead to prevent the electrons from jumping easily between MWCNTs, and as a result, the overall resistance will affect. At low concentrations of MWCNTs, the distance between adjacent MWCNT's is relatively large. As a consequence, the effectiveness of electron transfer (i.e. current flow) between the MWCNT's is very highly dependent on this MWCNTs spacing distance.

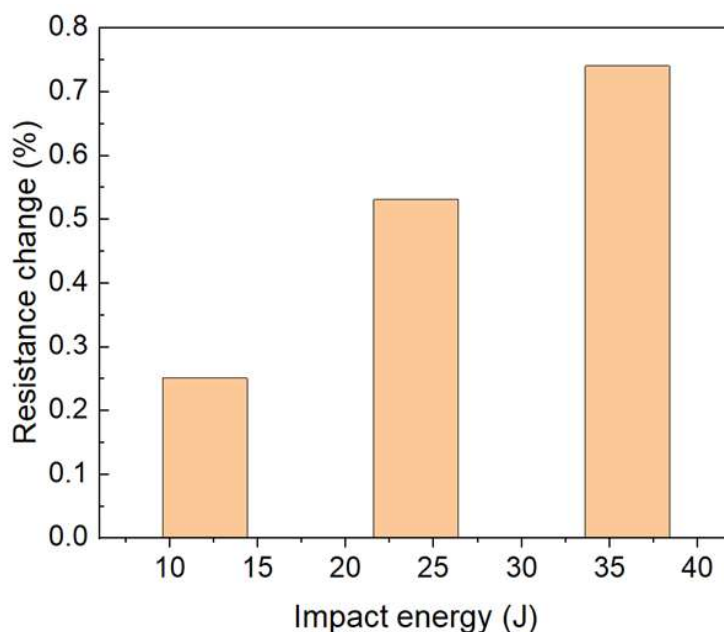


Fig. (4) Variation of electrical of self-sensing composite as a function of energy impact for the samples containing 0.5 wt% of MWCNTs.

5. Conclusion:

The mechanical properties of fabricated MWCNTs-GF nanocomposite are significantly reduced after impact showing up to 40% decrease in flexural strength correlated with higher energy impacts. The relationship of decreased strength is a linear and therefore can be used to calculate whether a composite material is no longer fit for use after it has sustained damage. The residual flexural modulus was also effected and showed a less degree of reduction in compared to the flexural strength. The upper layers of the composite structure received a higher the impact energy from the mid and bottom layers and this leads the modulus was less affected. The implications of this for the industry is important and would be saving money, by enforcement the upper composite layers. Combing this data with the data collected about the resistance changes that occur during an impact in composite materials using MWCNTs will give a clear result about the integratory of the damaged structure. That means the proposed idea of damage detection in composite materials works and is able to determine the severity of the damage that has occurred. It would seem likely that if this was to be used in industry, a lower percentage of carbon nanotubes presented a higher sensitive and would be used as this would make it both cheaper and more easily detected.

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