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High performance epoxy/ MWCNTs nanocomposite sensors for multi-purpose sensing in structural health monitoring

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Keywords

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Abstract

Structural health monitoring (SHM) is necessary for modern technical development. It can be achieved by detecting different quantities such as pressure and strain using highly sensitive and stable sensors. In this work, epoxy/multiwalled carbon nanotubes (MWCNTs) is used as sensing material to detect both strain and pressure. To ensure better performance, dispersion fabrication process was firstly optimized via electrical and microscopic measurements. Then, the piezoresistive performance such as the sensitivity, linearity, stability and drift were investigated under pressure and strain for films prepared with different carbon nanotubes (CNTs) concentration and thickness to define the optimal conditions for each sensing target.

The piezoresistive measurement shows good sensitivity to strain at very low CNTs concentration 0.3 wt. % with a gauge factor (GF = 11.094); which is around 6 times higher than conventional strain sensor, proving the efficiency of the optimized fabrication process parameters that ensure a homogenous distribution. Furthermore, the sensing layer shows also the ability to sustain high pressure load. The sensitivity is found to be highly dependent on the film thickness with higher sensitivity for thicker films in case of pressure sensing in addition to the good sensitivity at very low-pressure range. All these results prove the efficiency of Epoxy/MWCNTs as multi-purpose sensor material.

1 Introduction

Because of the excessive loading conditions and long-term deterioration, damage of structures can be occurred during their operational lifetimes. In order to control structures state, conventional strain sensors such as metallic strain gauges are usually used [1]. However, these sensors are not highly sensitive (GF = 2) and accurate to detect and locate damage effectively.

Therefore, developing sensors with high sensitivity and stability to external load and can be embedded in different complex structures form is remaining challenging [2, 3]. Among several possible smart materials, polymer/carbon nanotube (CNT) nanocomposites are gaining great attention as they can be used as sensing element to detect different quantities such as strain and pressure, providing a good alternative to conventional materials, as they offer higher sensitivity [4, 5].

In fact, the conductance of polymer/CNTs nanocomposites can be dramatically changed under an external load. Owing to this piezo-resistance property, polymer/CNTs nanocomposites have great potential for the realization of stable, sensitive, scalable and low-cost sensors.

However, the realization of this smart material is usually challenging as the CNT tend to agglomerate within the polymer matrix because of the large surface area and van der Walls attractive force between carbon nanotubes [6].

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Therefore, to realize a good performant sensing layer with less CNTs concentration, a homogeneous distribution of CNTs into polymer matrix have to be achieved. In this work, the sensing materials are produced using a simple and cost-effective method, which is the direct mixing. Epoxy/MWCNTs nanocomposites were prepared with different process parameters. Electrical and morphological characterizations were performed to examine the uniformity of the CNTs distribution at different fabrication process parameters. The best fabrication process is used to develop different sensors. Additionally, in this study, different aspects are considered such as the CNT concentration and the film thickness on the performance of pressure and strain sensors.

2 Materials and methods

2.1 Materials

In this work, the required multi-walled carbon nanotubes were purchased from Sigma Aldrich and used as received. These nanotubes have the following characteristics O.D. × L 6-9 nm × 5 µm, and a degree of purity greater than 95 %. This type of multi-walled carbon nanotubes can offer polymers high electrical conductivity and high strength [7]. Epoxy resin L 20 is used, it will act as a polymer that contains CNT particles. It is purchased from R&G Faserverbundwerkstoffe composite technology, GmbH, Germany.

2.2 Preparation of Epoxy/MWCNT nanocomposite

Carbon nanotubes are hydrophobic in nature and have poor solubility with other materials and high aspect ratios and large surface areas, which make them tend to agglomerate into polymer matrix. Therefore, optimization of process parameters is required. Here, a simple and cost-effective mixing process is adopted. In this process, two different techniques were used which are sonication and magnetic stirring as shown in Figure 1. In fact, sonication is a powerful tool; responsible for separation of CNTs individually within the matrix while magnetic stirring is useful to have uniform distribution. However, excessive sonication energy or time can lead to damage CNTs wall [8]. Therefore, optimizing the sonication time is required to avoid CNT damaging.

In this work, the sonication was performed for different amplitudes (15 %, 30 %, 45 %) corresponding respectively to (10.275 kJ, 20.55 kJ, 27.4 kJ) using a horn sonicator (Bandelin GM 3200, Sonication Temperature: 25 °C, Duty cycle: 50 %) for 30 min. Followed by magnetic stirring for 2 hours under a rotational speed of 400 rpm and heating temperature of 80 °C to form a homogeneous dispersion. To cure the material, the hardener was added to the mixture which was stirred for 15 minutes under the same conditions. Before deposition of the film, the solution was kept in the vacuum chamber for 30 minutes in order to extract the excess air in the mixed solution, which was generated from the long stirring process at the high temperature. Using stencil printing, different films were deposit. After deposition, the samples were cured in the oven at a temperature of 160 °C for different hours owning to thickness in order to allow the surface of the CNT film to solidify.

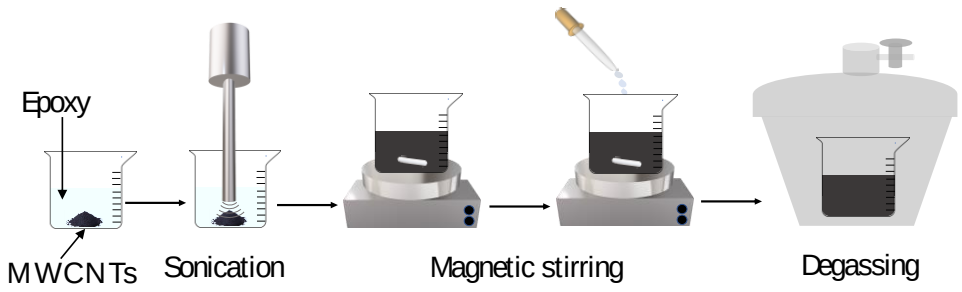

Figure 1: Fabrication process of epoxy/MWCNTs nanocomposite

2.3 Characterization methods

2.3.1 Electrical and microscopic characterizations

Polymer/CNT nanocomposites are highly conductive when CNTs are dispersed uniformly within the polymer matrix [9]. In order to examine the impact of sonication energy on the dispersion quality, DC measurements were carried out using Sourcemeter Keithley 2602A with a Labview software interface. For validation of results, microscopic measurements were performed in the range of 1–2 μm using Leitz Aristomet microscope with a brightness of 3.0 and magnification of 5x.

2.3.2 Piezoresistive characterization under different external load conditions

To investigate the piezoresistive performance of the different prepared films, ten repetitive cycles were performed using universal testing machine TIRA at a crosshead speed 10 mm/min and force from 0 to 40 N. In this measurement, the resistance of Epoxy/MWCNTs films was continuously recorded using a host computer connected to a Keithley 2602A Sourcemeter to investigate the sensitivity to strain deformation in addition to the sensor linear behaviour.

In addition, to characterize the piezoresistive performance under pressure load, a custom build pressure measurement test bench interfaced with sourcemeter is used, which is equipped with high resolution load cell (K307M.200) that can exert a maximum load of 200 N. In this study, five repetitive cycles were performed using pressure test machine at speed of 2 N/s and force ranging from 0 to 180 N. In this test, the thickness of Epoxy/MWCNTs nanocomposite film is addressed. Therefore, five different films were fabricated with different thickness named from A to E and corresponding respectively to (A = 67 μm, B = 119 μm, C = 253 μm, D = 419 μm, E = 534 μm) and characterized under pressure.

The investigation of sensor behaviour at very low load was also addressed, cyclic test was done for force ranging from 0 N to 15 N at a speed of 2 N/s. At the end, different samples were tested under fixed load of 180 N for more than 8 hours.

3 Results

3.1 Electrical and morphological characterizations

According to the results, the film prepared at low sonication energy possesses a very high resistance. By increasing the sonication energy, the resistance is reduced sharply at 30 % energy indicating the efficiency of the sonication process to unbundle aggregated carbon nanotubes. However, excessive sonication leads to increase the resistance as shown in Figure 1, which can be explained by the destruction of CNTs walls. Microscopic characterization of nanocomposites illustrates uniform CNTs distribution and the existence of more conductive network when the sample is prepared with 30 % amplitude.

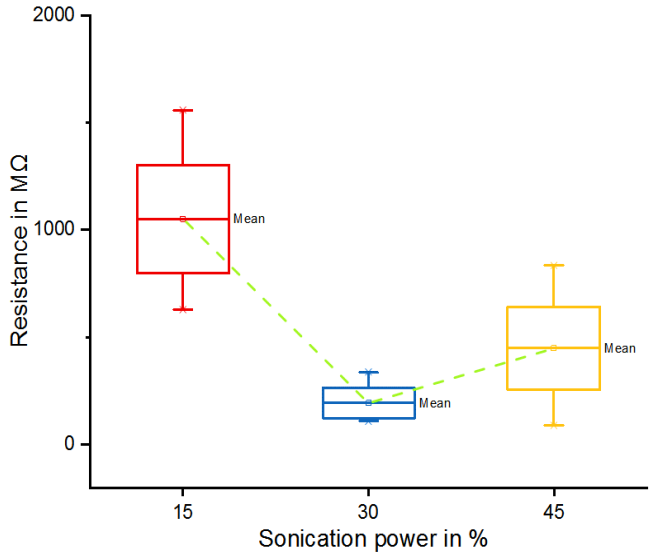

Figure 2: Electrical resistance depending on the sonication energy

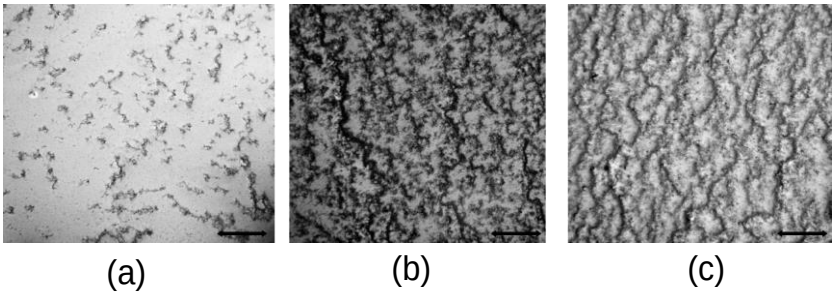

Figure 3: Microscopic images of the different composites depending on sonication amplitude, (a) 15 %, (b) 30 % and (c) 45 %

3.2 Epoxy/MWCNTs nanocomposites response under strain

After the optimization of the fabrication process, the piezoresistive behaviour of different films prepared with 0.3 wt. %, 0.5 wt. % and 1.0 wt. % concentration was tested under 10 repetitive cycles. In fact, the piezoresistivity is defined as the change of resistance under applied load. To evaluate the sensitivity of the sensor to external load, the gauge factor "GF" can be calculated using the following equation:

$$GF = (\Delta R \,/\, R_0) \,/\, \varepsilon \tag{1}$$

Gauge factor is the ratio of relative change in electrical resistance R divided by the applied strain.

Where ΔR is change in strain gauge resistance and R_0 is unstrained resistance of strain gauge.

Usually, the strain is obtained by calculating the static length of the sensor and the change of the length under the action of strain.

$$\varepsilon = strain = \Delta L \,/\, L_0 \tag{2}$$

Where ΔL is the absolute change in length and L_0 is the original length.

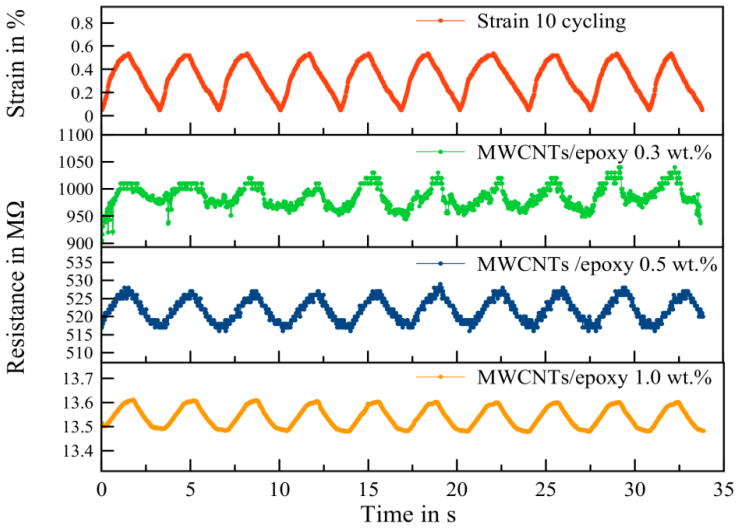

Figure 4: Ten cycling strain measurement

As it can be seen from Figure 4 and 5, the resistance is following the strain. For all the composites, the resistance is increased as strain increases and decreased when the force is released. The film prepared with high amount of CNTs show better stability and linearity than at low CNTs concentration a linear regression coefficient of 0.976, due to the weakness of tunneling effects [10]. This latter leads to reduced sensitivity. The increased sensitivity at very low CNT concentration from 1.203 at 1 wt. % to 11.094 at 0.3 wt. % can be also explained by the reduced mechanical properties of the material leading to larger the distance between the adjacent CNTs.

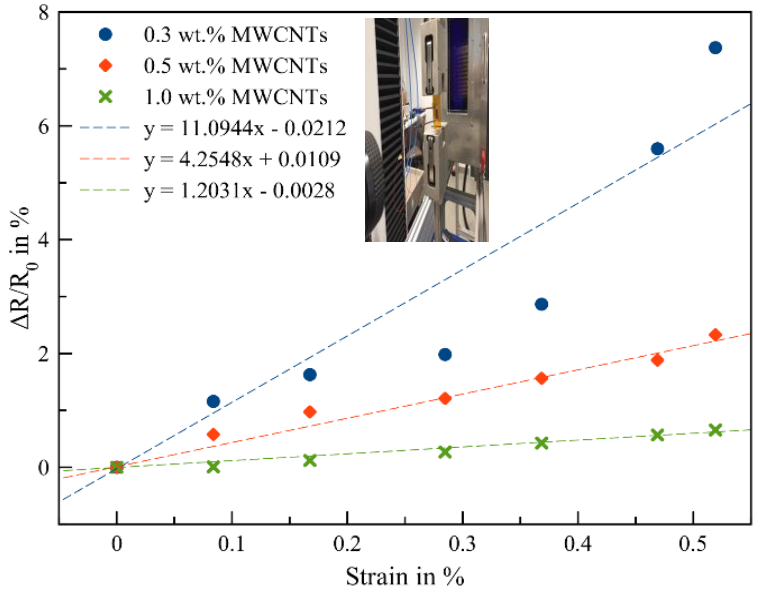

Figure 5: Resistance change vs strain at different CNT concentration

3.3 Epoxy/MWCNTs nanocomposites response under pressure

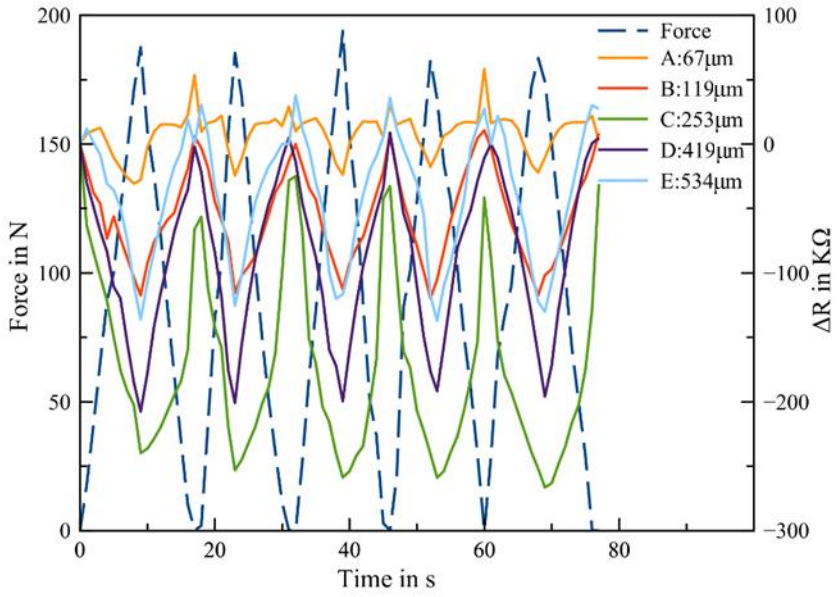

Figure 6: Piezoresistive behaviour of different sensors under high pressure force

The resistance change due to the external pressure at different film thickness is presented in Figure 6. According to this figure, film resistance is reduced when the pressure force is increased due to minimization of the gaps between neighbouring nanotubes.

As the thickness of the nanocomposite film increases, the film resistance variation with the pressure force is increasing because of the increased possibility to form more conductive paths within the nanocomposite [11]. However, the resistance change of the film from C to E were reduced, which can be explained by the sedimentation of all the CNTs on the bottom of the film during the long curing process and leading to inhomogeneous CNTs distribution. The obtained results show that the film with thickness 253 µm has the best performance in terms of sensitivity and stability. The sensor sensitivity is found to be at high pressure range approximately 1.4 kΩ/N. This sensor shows also better sensitivity to low pressure range around 6.7 kΩ/N as illustrated in the cyclic measurement presented in Figure 7.

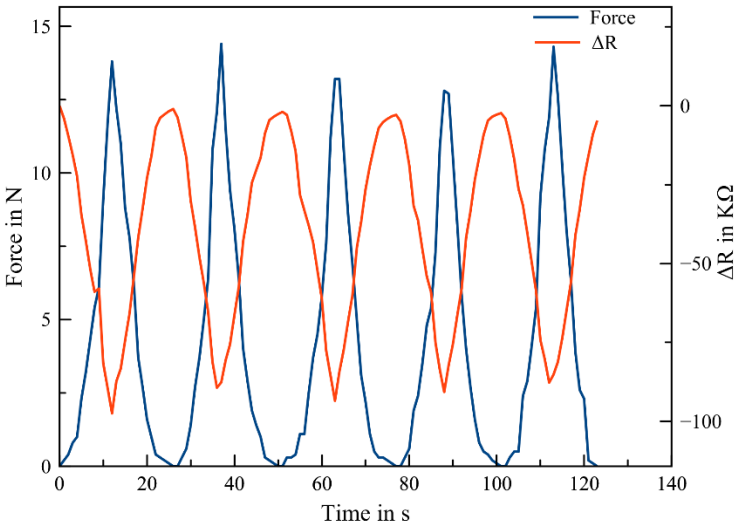

Figure 7: Cycling test for the nanocomposite "C" at low pressure

To investigate the stability over time of the Epoxy/MWCNTs nanocomposites, drift test is performed as shown in Figure 8. The results show also that the film with 253 µm thickness has better stability over time under high pressure range, which indicates the efficiency of the chosen fabrication process to ensure good homogeneity of the nanocomposite sensor. A particular result was observed for film prepared with the lowest thickness; the resistance is reduced sharply over time because the material cannot afford high forces due to thickness issue.

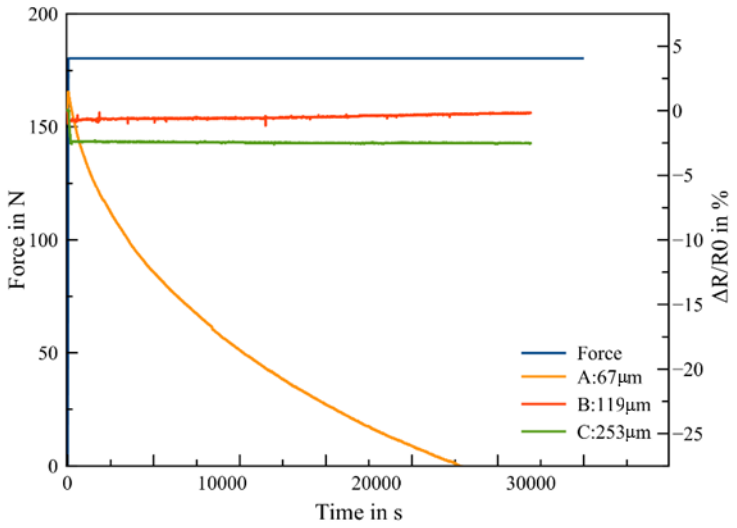

Figure 8: Drift test for different sensors thickness

4 Conclusion

Epoxy/MWCNT nanocomposites exhibit piezoresistive characteristics which make them have great potential for fabrication of multi-functional sensors to detect different targets in SHM. The developed nanocomposite sensors have multiple features. According to the piezoresistive measurement under strain, the nanocomposite shows good sensitivity to strain with a gauge factor (GF = 11.094); which is around 6 times higher than conventional metallic strain gauge sensor at very low CNTs concentration 0.3 wt. %, proving the efficiency of the selected fabrication process to ensure a homogenous distribution. Different CNT concentration levels directly affect the conductivity and strain sensitivity of the sensor. By performing cyclic test, the sensor with 0.5 wt. % CNT concentration shows stable performance over cycling.

Furthermore, Epoxy/MWCNT nanocomposites shows the potential to be as pressure sensor working up to 180 N. The sensitivity is found to be highly dependent on the film thickness with higher sensitivity for thicker films. In addition, Epoxy/MWCNTs film was highly sensitive to low-pressure range.

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