Strain sensing with CNT Nanocomposites: static, cyclic and dynamic electromechanical material characterization

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ABSTRACT

Carbon Nanotube (CNT) nanocomposites are one of the most important candidates to realize innovative strain sensors for Structural Health Monitoring (SHM) applications.
In this work, the effect of static and dynamic strain on the electromechanical properties of carbon nanotubes (CNTs) nanocomposites, is investigated. In particular the nanocomposite is formed by multi-walled CNTs (MWNTs) embedded in a PolymethylMethacrylate (PMMA) matrix. The MWNTs randomly distributed within the PMMA matrix form conductive paths. These paths modify they morphology when the material is strained. Consequently the overall material conductivity changes. Continuous monitoring is possible by correlating these electrical changes to the deformation level of the material. Different specimens are made by varying the MWNTs content (3%, 5%, 7%, weight fractions) and are tested under varying static, cyclic and dynamic loading conditions. It is found that the Gauge Factor (GF) and nanocomposite sensitivity to strain, are directly related to the MWNTs content. Nanocomposites with higher MWNTs percentages (7%) show the best behaviour with a smaller dispersion of the experimental data. This data reproducibility is comparable to that of conventional strain gauges.
The proposed functional material has the beauty of being ultralight and flexible. Moreover this material design has the potential of being scalable in size allowing continuous monitoring of larger structural areas than commercial sensors. The results shown in this paper highlight that this nanocomposite is a great candidate for the realization of advanced sensing devices.

Keywords: carbon nanotubes, nanocomposite, strain sensor.

1 INTRODUCTION

Strain sensors are widely used for Structural Health Monitoring (SHM) applications. Strain gauges are traditional sensors which are commonly used thanks to their low cost and ease of use. However strain gauges can only measure strain at specific locations and directions. To overcome these limitations, there is the need to develop sensors that can allow to monitor strain in larger structural areas.

The superior electromechanical properties of carbon nanotubes (CNTs) and the well established techniques for embedding them into a hosting matrix, make them an ideal candidate for this scope. In fact, when CNTs are randomly dispersed in a polymer matrix, they form paths through which electrical current flows if a voltage gradient is applied. A mechanical deformation of the nanocomposite induces variations of the CNTs network morphology. These variations are directly correlated to changes of the overall material conductivity [6]. In other words, these type of nanocomposites perform as a piezoresistive material which, in particular, increases its resistance in response to mechanical strain [7]. In general, the piezoresistive sensitivity, or Gauge Factor (GF), correlates the electrical and mechanical response of a sensor.
Recently, a significant amount of research activities have been devoted to this topic. Kang et al. compared the response of a buckypaper (a special type of single-walled carbon nanotubes-SWNTs- thin film) and of SWNTs-PMMA nanocomposites for strain sensing [5]. Li et al. characterized MWNTs films by a uniaxial load/unload tensile test to investigate the material capability to sense strain [3]. Bu et al. found that a long sonication time improved the sensor sensitivity because, in this way, it is possible to reach a better dispersion of carbon nanotubes into the polymer matrix [1]. Loh et al. realized a sensing skin with SWNTs-Polyelectrolyte composite. Samples were mounted onto cementitious composites and a conductivity reduction was recorded as a respomse to the applied mechanical strain [4]. Vemuru et al. highlighted a linear relationship between voltage and strain changes [9]. Park et al. obseved an increase in electrical resistance of epoxy/MWNTs films under tensile stresses.
The aim of this experimental work is to investigate the electromechanical response of PMMA nanocomposites when loaded under varying conditions (static, cyclic and dynamic loading) as well as to correlate this response to different MWNTs contents.

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2 NANOCOMPOSITE FABRICATION

Multi-walled Carbon Nanotubes (MWNTs) from US Research Nanomaterials, Inc. are used (purity higher than 95%, outer diameter between 50-80 nm, inner diameter between 5-15 µm, net weight of 100 g, length of the tube between 10-20 µm). PolymethylMethacrilate (PMMA) produced by Sigma Aldrich with a molecular weight of 35000 by GPC and density of 1.17 g/ml at 25°C is used to disperse MWNTs. Chloroform by Sigma Aldrich is used (molecular weight of 119.39 g/mol, density of 1.17 g/ml at 25°C).

A polymer/solvent volume ratio of approximately 1:40 is used to fabricate the film. PMMA is dissolved in Chloroform using a mechanical stirrer for 20-30 minutes. The weighted amounts of MWNTs are added to the solution and sonicated for 2 hours [8]. The sonicated solution is poured in a glass mould and is placed in a vacuum oven for 30 minutes to realize MWNTs-PMMA nanocomposites with different MWNTs weight fractions (3%, 5%, 7%) (Fig. 1).

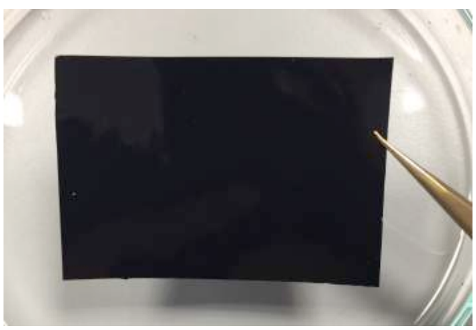

Figure 1: MWNTs-PMMA Nanocomposite.

3 RESULTS AND DISCUSSION

3.1 Electrical Characterization of the nanocomposite

In order to characterize the piezoresistive material behaviour, electromechanical tests are performed. A Semiconductor Parameter Analyzer (Agilent) is used to carry out electrical measurements while, simultaneously, samples are mechanically tested with a Dynamic Mechanical Analyzer Q800 (TA Instruments). The material piezoresistive behaviour is quantified through the Gauge Factor (GF) definition , which correlates the electrical and the mechanical response of the material:

$$GF = \frac{\Delta R/R}{\varepsilon} \qquad (1)$$

where R is the initial electrical resistiance (without strain), ΔR is the change of the electrical resistance when the material in strained, ε is the mechanical deformation.

Electrical contacts are realized with the help of a silver paste.

Initial I-V tests are performed when the nanocomposite is in an unloaded state in order to evaluate the intrinsic electrical properties of the material. An increasing voltage, from 0 to 10 Volts, is applied to the samples while measuring the current that was consequently flowing between the two electrodes. The resulting electrical resistance is calculated:

R = V/I, where V is the applied voltage and I the intensity of the measured current. It is worth noting that the applied voltage range is larger than the voltage at which strain sensors typically work. This is done in purpose to deepen the nanocomposite response.

The material resistivity (ρ) is calculated from the electrical resistance, the distance between electrodes (l) and the section area of the sample (A) by means of the well known formula:

$$R = \rho \frac{l}{A} \qquad (2)$$

The material resistivity is found to have a pseudo-linear trend. Slight variations from this trend are observed and addressed to a non-uniform distribution of CNTs into the polymer matrix. The non-uniform distribution leads to a larger CNTs content in some specific areas. As a consequence, the nanocomposite resistivity, that is directly related to the points of contact between the neighboring fillers, is non-uniformly distributed all over the sample.

3.2 Electromechanical characterization of the nanocomposite

Initially, tensile tests in displacement control are carried out with a Dynamic Mechanical Analyzer. The nanocomposite is deformed in specific independent steps within which an increasing deformation level is imposed. At the end of each deformation step, the electrical response of the nanocomposite is investigated with the same procedure as that described above. In this case, however, the applied voltage range is between 0 and 2 Volts. It is found that the electrical resistance of the nanocomposite increases with increasing strain as shown in Figure 2 in which the average trend of the normalized electrical resistance variation is reported as a function of the applied mechanical strain. It is worth noting that the slope of the curve represents the material (sensor) Gauge Factor. These data clearly highlight that the relationship between the film resistance and the applied strain is nearly linear up to approximately 0.1% [1]. These tests also highlight that the GF depends on the CNTs weight fractions (see the three different curves in Figure 2). In particular the sensitivity of the film decreases when the CNTs weight fraction increases [8].

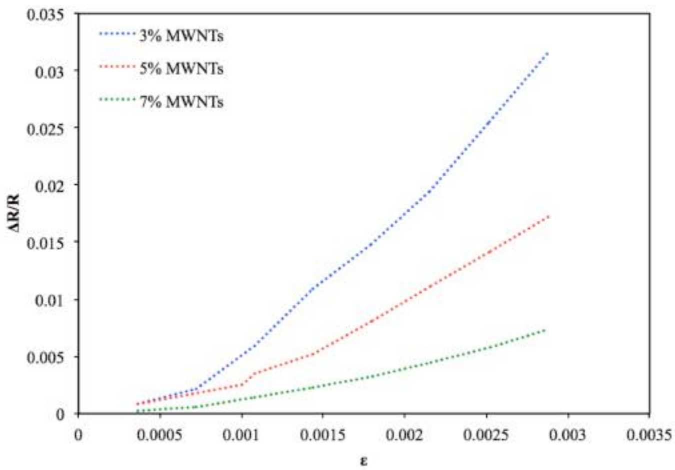

Figure 2: Gauge Factor of the samples with different amount of MWNTs.

It is also observed that the Gauge Factor increases with the increasing applied strain. It is believed that this piezoresistive material behavior is mainly due to the characteristics of the conductive network formed by the MWNTs dispersed into the PMMA matrix. A schematic of this concept is shown in Figure 3 that represents a close-up of the random CNTs distribution. The CNTs in the network interact electrically, either by a direct physical contact, or by a close proximity. These two types of interactions are represented as "points of contacts" highlighted in the figure. When the material is strained the distance between the CNTs increases locally and this might cause a decrease in number of contact points as represented in Figure 4. Since the electrical conductivity of the material is closely related to the points of contact between neighboring carbon nanotubes, variations of material conductivity are consequently recorded. In particular, an increase of the overall material resistivity is obviously observed.

The nanocomposite is also investigated in terms of its response speed and capability to recover its initial electrical properties. This is done by testing the material under a large number of cyclic loading-unloading conditions (60 cycles). During these mechanical tests a constant voltage of 1 Volt is applied, and the flowing current is simultaneously monitored. The electrical resistance is calculated from the detected experimental data. A direct correlation of electrical resistance changes with mechanical strain is recorded and reported in Figure 5. A great agreement is found between the calculated resistance over time, with the displacement imposed by the instrument.

However, it is observed that the average calculated resistance experiences slight variations but these variations do not considerably affect the overall GF value. This result is in agreement with Ref [2,10]. In fact, even if a change of the minimum and maximum values of the electrical resistance is observed, the GF remains the same because the slope of the cycles does not vary. This behaviour is again related to the previously described configuration of the conductive paths but it is also related to the viscoelastic response of the embedding matrix. In fact, when the nanocomposite is strained the configuration of these paths changes (thus the resistance changes); but, as the mechanical strain is removed, the polymer chains surrounding CNTs possibly slow-down the recovery of the initial network configuration. This leads to a slight change of the unloaded electrical resistance.

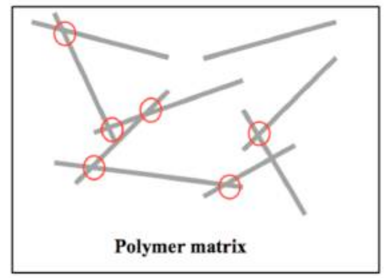

Figure 3: Conductive network of random nanotube embedded in the polymer matrix.

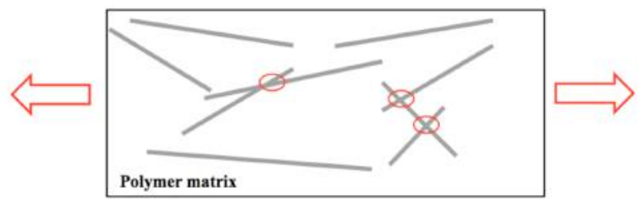

Figure 4: Configuration of the conductive network when the material is strained.

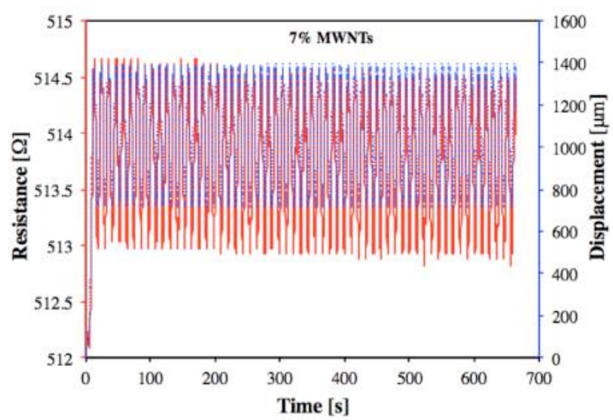

Figura 5: Piezoresistive measurements for 60 cycles.

Finally, the dynamic response of the nanocomposite is also investigated. A piece of nanocomposite is installed on a fiberglass cantilever. An impulsive force is applied on the free end of the cantilever to evaluate the oscillating frequencies of the supporting structure and of the surface-mounted nanocomposite.

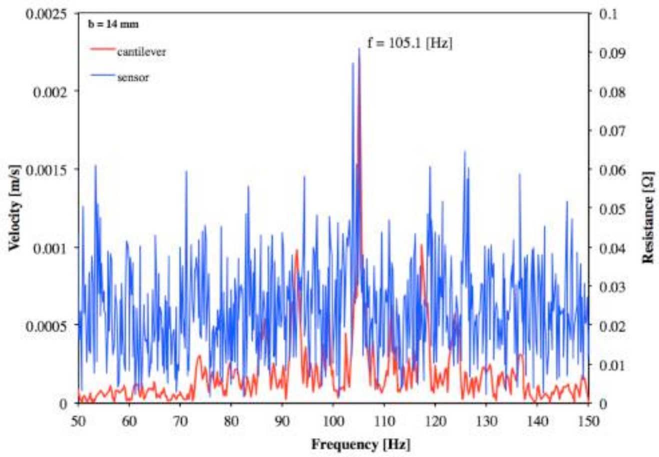

Figure 6: Frequencies of the sensor and the cantilever.

Figure 6 shows the resulting oscillating frequencies of the supporting structure (red line) and of the nanocomposite (blue line). The nanocomposite signal is affected by a considerable noise, that can be addressed to a non optimized electrical contact with the probes as well as to a non fully uniform dispersion of the CNTs within the matrix. CNTs aggregates cacn infact lead to an increas of internal resistance. Despite the observed noise, it is found that the dynamic electromechanical response of the material allows to clearly identify the structural frequency with great accuracy. This implies that the nanocomposite have the potential to be suitably adopted to realize sensing devices for structural health monitoring systems.

CONCLUSIONS

The superior properties of carbon nanotubes make them one of the most important candidates to realize a new promising kind of strain sensor.
In this study PMMA- MWNTs (3%, 5%, 7% weight fractions) nanocomposites are proposed for this purpose
The piezoresistive behaviour of the material was investigated: under varying static, cyclic and dynamic loading conditions. The electromechanical results show that the Gauge Factor is directly related to the amount of MWNTs embedded into the PMMA matrix, as to the applied strain, as well as to the mechanical response of the hosting matrix.
The dynamic test results show that even though the signal is affected by noise it allows to clearly indentify the structural frequency.
This study shows the great potential of this new kind of ultralight material for strain sensing applications.

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REFERENCES

[1] Bu L, Steiz J and Kanoun O Influence of processing parameters on properties of strain sensors based on carbon nanotube films 7th International Multi-Conference on Systems, Signals and Devices - Sensors, Circuits & Instrumentation Systems, June 27-30, 2010, Amman, Jordanien.

[2] Gonçalves B. F., Oliveira J., Costa P., Correia V., Martins P., Botelho G., Lanceros-Mendez S., Development of water-based printable piezoresistive sensors for large strain applications, Elsevier Composites Part B 112, 2017, p. 344-352.

[3] Li X., Wang W., Liao K., Hu C., Huang Z., Feng Q., Piezoresistive effect in carbon nanotube films, Chinese Science Bulletin, 2003, vol. 48, no. 2, pp. 125-127.

[4] Loh K. J., Hou T. C., Lynch J. P., Kotov N. A., Nanotube-based Sensing Skins for Cracks Detection and Impact Monitoring of structures, Proceedings of the 6th International Workshop on Structural Health Monitoring, 2007.

[5] Kang I., Schulz M. J., Kim J. H., Shanov V., Shi D., A carbon nanotube strain sensor for structural health monitoring, Smart Materials and Structures, 2006, 15, p. 737-748; doi:10.1088/0964-1726/15/3/009.

[6] Kanoun O., Müller C., Benchirouf A., Sanli A., Dinh T. N., Al-Hamry A., Bui L., Gerlach C., Bouhamed A., Flexible Carbon Nanotube Films for High Performance Strain Sensors, 2014, 14, p. 10042-10071; doi:10.3390/s140610042.

[7] Park JM, Kim DS, Lee JR, Kim TW. Nondestructive damage sensitivity and reinforcing effect of carbon nanotube/epoxy composites using electro-micromechanical technique. Mater Sci Eng C 2003;23:971–5.

[8] Pham G. T., Park Y. B., Liang Z., Zhang C., Wang B., Processing and modeling of conductive thermoplastic/carbon nanotube films for strain sensing, 2007, Elsevier Composites: Part B 39 pp. 209-216.

[9] Vemuru S. M., Wahi R., Nagarajaiah S., Ajayan P.M., Strain sensing using a multiwalled carbon nanotube film, Journal of Strain Analysis for Engineering Design, vol. 44, no. 7, pp. 555-562, 2009.

[10] Zhao Y., Schagerl M., Viechtbauer C., Loh K.J., Characterizing the Conductivity and Enhancing the Piezoresistivity of Carbon Nanotube-Polymeric Thin Films, Materials, 2017, 10, 724.