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## **CARBON NANOTUBES BASED SENSORS FOR DAMAGE DETECTION**

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### **ABSTRACT**

In this paper Multi-walled Carbon Nanotubes (MWNTs) - based nanocomposite films were investigated as dynamic strain sensors for damage detection. After a thorough study to optimize the electromechanical properties of the material as a function of the MWNTs content, the proof-of-concept was carried out by installing the film onto the surface of a fiberglass cantilever. The cantilever was made to oscillate through an impulsive force applied at its free end. The free oscillations of the structure are monitored and the frequencies are identified before and after reducing locally the cross section of the cantilever (to simulate damage propagation). The experimental data show the capability of the manufactured nanocomposite film to clearly identify structural frequencies in dynamic regimes. The obtained experimental data also show that this material has the potential to monitor incipient cracks (nano/micro-scaled cracks) due its sensitivity to nanoscale morphology changes.

**Keywords:** carbon nanotube, nanocomposite, strain sensor, damage detection.

### **INTRODUCTION**

Structural health monitoring systems are essential to guarantee safety and reliability of structures. These systems are typically made of an array of sensors coupled with data acquisition and processing devices. Several types of sensors are used for this purpose. For instance, strain sensors and their properties, differ from each other depending on the physical principle that is exploited to sense strain. Piezoresistive strain sensors, in particular, are based on the concept that the electrical resistance changes when the sensor is strained. This effect is related to the molecular or crystal structure of the material that forms the "sensitive" part of the device. Strain gages are largely selected to monitor deformation because of their low cost, ease of usage and sensitivity. However they can only measure strain on the surface of the structure and in a small number of locations/directions. Consequently, there is the need to develop distributed monitoring systems, which can measure the occurring strain and incipient cracks in a larger number of sensing locations and directions.

The unique electro-mechanical properties of Carbon Nanotubes (CNTs) (Lanzara, 2010; Lanzara, 2015), make them very useful for this scope and in particular to realize ultra-light and flexible piezoresistive strain sensors for Structural health monitoring specialized for damage detection (Zhang, 2009).

CNTs can in fact be embedded in an insulating polymer matrix to form functional nanocomposites characterized by a great capability to sense strain. If the hosting polymer

matrix is mechanically flexible and in the form of a thin film, then these nanocomposites have the potential to represent a good alternative for thin film strain sensors.

The electrical conductivity or resistivity of CNT nanocomposites, is closely related to the conductive network formed by the conductive carbon nanotubes randomly dispersed in the insulating polymer matrix. Thus, the piezoresistive behaviour of the sensors is strongly influenced by the actual configuration of the CNTs network that can vary when mechanical stress is applied (Kanoun, 2014).

A few studies were focused on the capability of CNTs nanocomposites to sense strain. Kang *et al.* compared the strain sensing response of Single-walled Carbon Nanotubes (SWNTs) buckypapers with that of SWNTs-PMMA nanocomposites. The buckypaper was found not to be appropriate for measuring strain in the elastic range, although it showed an overall higher sensitivity than the nanocomposite. The latter were in fact characterized by a linear response (strain) under tension and compression (Kang, 2006). Li *et al.* characterized samples of thin MWNTs film with uniaxial load/unload tensile tests. The results showed that electrical resistance changes were again closely related to the applied strain (Li, 2008). Bu *et al.* investigated the effect of sonication time on the sensitivity of CNTs strain sensors. They found that the sensitivity of the sensor was improved thanks to a longer sonication process (Bu, 2010). A sensing skin (SWNTs-Polyelectrolyte composite) was proposed by Loh *et al.* to monitor strain on cementitious composites (Loh, 2007). Finally, Vemuru *et al.* highlighted a linear relationship between voltage and strain changes (Vemuru, 2009).

Despite the large amount of studies which successfully reported the possibility of using CNT nanocomposites for strain sensing, to the best of the authors knowledge none of these studies investigated the use of these nanocomposites as "dynamic strain sensors" for damage detection. With this in mind, an experimental work is here conducted and the dynamic electromechanical response of CNT nanocomposites is assessed while monitoring an increasing damage.

## MATERIALS AND NANOCOMPOSITES FABRICATION PROCESS

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

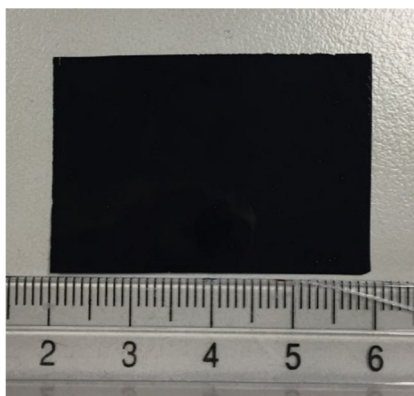


Fig. 1 - Nanocomposite MWNTs-PMMA film.

For the film fabrication, it was considered a volume ratio of approximately 1:40 between the polymer and the solvent. PMMA was dissolved in Chloroform using a mechanical stirrer for 20-30 minutes. The weighted amounts of MWNTs were added to the solution and sonicated for 2 hours. (Pham, 2007). The sonicated solution was poured in a glass mould and was placed in the vacuum oven for 30 minutes to realize MWNTs-PMMA nanocomposites with different amount of nanotubes (3%, 5%, 7%, weight fractions), Figure 1.

The thickness of the samples was measured by using a microscope and the observed average values are reported in Table 1.

Table 1 - Thickness of the sample.

3% MWNTs	82.3	μm
5% MWNTs	65.5	μm
7% MWNTs	103.0	μm

## ELECTROMECHANICAL CHARACTERIZATION

Several electromechanical tests are carried out in order to correlate the electrical and the mechanical nanocomposite response.

In this work the electrical measurements are performed using a Semiconductor Parameter Analyzer (Agilent), whereas the mechanical tests are carried out with a Dynamic Mechanical Analyzer Q800 (TA Instruments). A thin copper foil and silver paint are used to make the electrodes, on the two opposite ends of the rectangular sample.

First, the resistivity ranges are evaluated by performing electrical measurements in a stress-free configuration. A voltage from 0 to 10 V is applied and the intensity of the corresponding current, which flows between the two electrodes, is measured. The actual resistance of the samples is consequently identified. Because of the extreme accuracy of the adopted acquisition system adopted, it was not necessary to provide a Wheatstone bridge-based circuit.

As it is known, the material resistivity is directly related to the carbon nanotubes content (Table 2) and to the conductive network formed by the neighbouring carbon nanotubes that are physically in contact or separated by a very small gap. The electrical resistance in particular decreases with increasing content of well dispersed carbon nanotube. This is because a larger number of points of contact that is formed with increasing filler content. The results highlight a linear material behaviour.

Table 2 - Resistivity of the nanocomposite films.

	3% MWNTs	5% MWNTs	7% MWNTs
$\rho$ [ $\Omega$ m]	0.70	0.12	0.10
$\sigma$	0.184	0.059	0.043

Table 2 shows the average resistivity measured for different CNTs concentrations and the relative standard deviation. As the CNTs content in the film increases, there is a less dispersion of the experimental data and the resistivity considerably decreases.

Tensile tests in displacement control mode are then performed. During the entire mechanical test duration, I/V tests are carried out to evaluate the electrical resistance while the material is strained.

The Gauge Factor (GF) of the sensor is defined to correlate the electrical and mechanical response:

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

Where  $\Delta R$  is the electrical resistance change,  $R$  is the initial electrical resistance (unloaded sample),  $\varepsilon$  is the deformation.

An increasing electrical resistance is recorded with increasing applied strain, in agreement with previous studies (Heyd, 1997; Maiti, 2002; Yang, 1999).

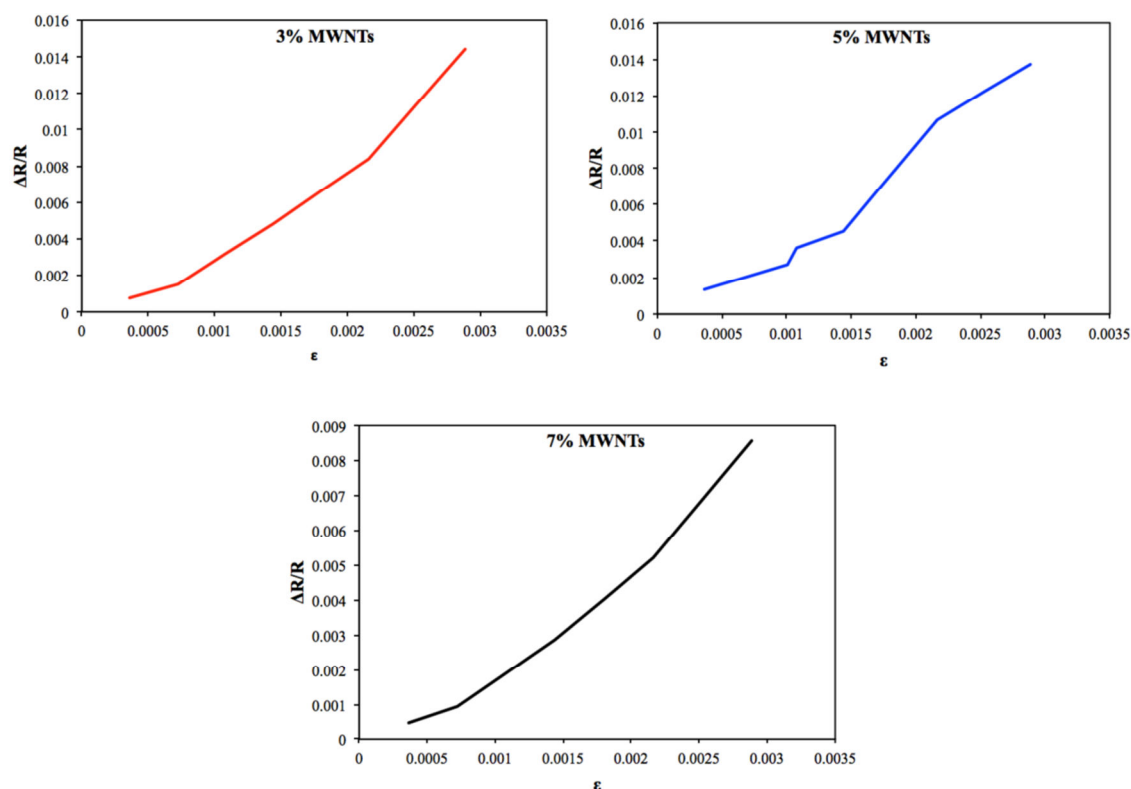


Fig. 3 - Gauge Factor of the film with different carbon nanotubes concentration (3%, 5%, 7%, weight fractions).

This is because when the material is stretched, its nanoscale morphology changes and the distance between neighbouring CNTs increases. Loss of contact or an increase of distance between CNTs has the direct consequence of inducing a higher electrical resistance to the overall material, in agreement with the experimental data.

The obtained experimental data highlight a nearly linear relationship between the electrical resistance and the applied strain, up to 0.1%, as it is shown in Figure 3. The GF, that is the slope of the curves represented in Figure 3, increases gradually for higher strain levels (over 0.1%) and decreases with increasing carbon nanotube weight fraction. It is worth noting that

commercial strain gages, generally used in strain sensing applications, are characterized by a GF of maximum 3 while the proposed nanocomposite has the potential to reach higher sensitivity by controlling the CNTs concentration.

## DAMAGE DETECTION

The capability of the nanocomposite to detect damage is investigated by testing the material under dynamic loading conditions. The nanocomposite film is bonded on a fiberglass cantilever. An impulsive force is applied on the free end of the cantilever and the oscillating frequencies are observed (see test-setup in Figure 2). The time history of the cantilever is acquired by using a Laser Vibrometer (Polytec), whereas the sensor response is acquired by using a Semiconductor Parameter Analyzer (Agilent). The I/V data over time are recorded to calculate the electrical resistance.

First the undamaged cantilever is tested to verify the capability of the sensor to identify the dynamic response of the structure. The test is then repeated by cutting the cantilever with symmetric and progressively larger cuts (2 mm, 4 mm and 5 mm) shown in Figure 3.

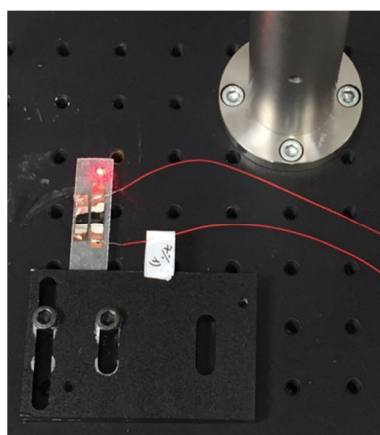


Fig. 2 - Experimental set-up.

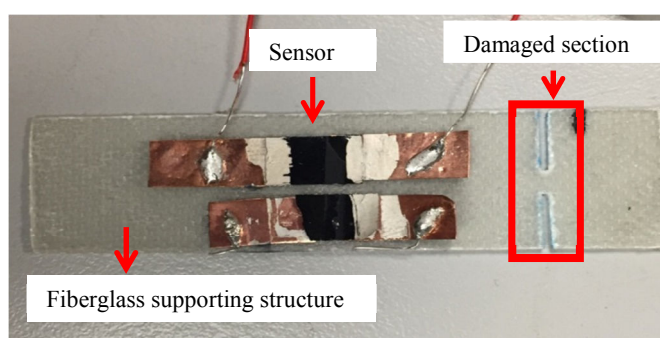


Fig. 3 - Damaged structure with the nanocomposite sensor.

The acquired oscillating frequencies of the sensor and the cantilever are compared.

Figure 4 shows the acquired oscillating frequencies of a symmetrically damaged cantilever (2mm for each side). In particular the graph reports the velocity recorded with the laser and the resistance of the surface-mounted nanocomposite, both vs time.

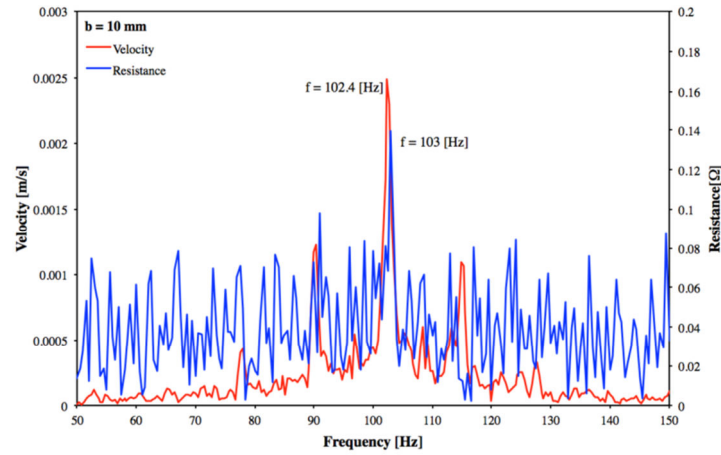


Fig. 4 - Damaged cantilever (red line) and sensor (blue line) frequencies.

Table 3 shows the oscillating frequencies of the cantilever and of the sensor.

Table 3 - Identified frequencies for the damaged beam.

<b>b [m]</b>	<b><math>\Delta J</math>[%]</b>	<b><math>f_{\text{cantilever}}</math>[Hz]</b>	<b><math>f_{\text{sensor}}</math>[Hz]</b>	<b><math>\Delta f</math>[%]</b>
0.014	-	105.1	105.1	0.00
0.010	-30	102.4	103.0	0.59
0.006	-60	98.3	97.1	-1.22
0.004	-70	84.0	83.0	-1.19

The results illustrated in Figure 4 clearly show that the nanocomposite signal is affected by a significant noise. This can be related to the partially optimized manufacturing process, i.e. the thickness of the obtained film is not perfectly constant and the CNTs dispersion is not uniform as desired. Moreover, the adopted electrodes do not provide optimal connectivity.

Despite the recorded noise, the nanocomposite allows to identify the structural frequencies with sufficient accuracy. In particular, the frequency of the undamaged structure is perfectly identified. As damage increases, the frequency detected by the nanocomposite is highly accurate when compared with that derived from the laser signal. The results are summarized in Tab. 3, which shows that the two measuring systems (laser and nanocomposite) have a difference in their response that does not exceed 1.2%.

Hence, the sensor can properly be adopted for structural monitoring applications but it can still be improved in terms of electrical connections and CNTs dispersion.

## CONCLUSION

In this study, MWNTs-PMMA nanocomposites are realized with different carbon nanotubes amounts (3%, 5%, 7%, weight fractions).

In order to understand and characterize their functional response electromechanical tests are performed. These tests highlight the piezoresistive properties of the material: when the material is strained, the electrical resistance increases. This phenomenon is due to morphology variations of the carbon nanotubes dispersion in the polymer matrix. The experimental data show that the relationship between the deformation and the electrical resistance is nearly linear for low deformation values.

The Gauge Factor of the sensor is consequently defined: higher values are found for lower CNTs concentrations.

The capability of the nanocomposite to detect damage is investigated by bonding it on a fiberglass cantilever which was dynamically excited and monitored with the nanocomposite and with a laser. The cantilever is then gradually damaged and the test is repeated. The oscillating frequencies are compared.

The frequencies of the cantilever and of the laser are in great agreement and both show that the natural frequency decreases with increasing damage

Even though the nanocomposite signal is affected by considerable noise, that is due to a non-optimized manufacturing process, it enables to identify the structural frequency with excellent accuracy. The results reported in this paper clearly show that the nanocomposite has the potential to be adopted as sensor for Structural Health Monitoring applications.

## **ACKNOWLEDGMENTS**

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