



## Material Behaviour

## Requirements of amount of carbon nanotubes for damage detection in large polymer composite structures

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## ARTICLE INFO

## Article history:

Received 7 July 2017

Accepted 13 August 2017

Available online 18 August 2017

## Keywords:

Nano-structures

Polymer-matrix composites (PMCs)

Electrical properties

Mechanical testing

## ABSTRACT

Due to their very high electrical conductivity, the addition of carbon nanotubes (CNTs) into polymers such as epoxies makes these materials conductive. This conductivity has been utilized to provide damage sensing in composite structures. Usually, the amount of CNTs needs to be more than the percolation threshold to assure electrical conductivity. The percolation threshold is usually determined using small samples. For large samples, the amount of CNTs needs to be higher to take into account some non-uniformity of the dispersion. More CNTs would provide better conductivity. One normally expects that more CNTs would also provide better damage detection. However, it was found that this is not the case. Certainly, the amount of CNTs needs to be more than a certain lower limit to assure conductivity throughout the large structures. Once this condition is met, adding more CNTs would reduce the sensitivity for damage detection. The sensitivity of damage detection can be measured by the change in electrical resistance (due to the occurrence of damage) between grid points that are attached on the surface of the composite structure. Higher sensitivity in damage detection would enable coarse grids (larger distance between grid points). Coarse grid points would mean lower number of grid points, less space, less wiring and less weight. This paper describes this phenomenon in detail. It provides models that simulate the conductivity configurations. It also introduces a new term call "Aggregately Conductive Materials" to distinguish the particular conductive characteristics of materials that are made conductive by the addition of nano-particles.

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## 1. Introduction

The increased use of Polymer Matrix Composites (PMC) in space and aerospace applications demonstrates the essential need for continued health monitoring of polymer composite structures due to their susceptibility to different types of damage [1]. Many SHM techniques have been used for this purpose [2]. However, their usefulness is limited. For instance, metallic strain gages mounted on the surface of structures is a common and commercially feasible SHM technique for measuring strains. However, a strain gage is not capable of monitoring matrix cracks in fiber/polymer composites where matrix cracks cannot be translated into significant increase

in strain because the load may be carried by fibers. Electrical resistivity based nanocomposite sensing has found considerable attention where CNTs are introduced in polymer composites [3]. This is because of their small size, high aspect ratios (length to diameter ratio), exceptional electrical conductivity [4], thermal conductivity [5] and mechanical properties [6]. The addition of CNTs in PMC (above the electrical percolation threshold) provides electrical conductivity for the polymer allowing the resistance of the modified polymer composite to be measured. This presents tremendous potential for SHM of the composites using electrical resistance measurement (ERM). Significant work has been performed in producing both embedded and attached sensors made using CNTs for strain and damage sensing in PMC [7]. Hu et al. [8] investigated the effect of processing conditions and material properties on the sensitivity of an epoxy/CNT composite as an attached sensor. They reported that the sensitivity of the sensor is affected by many parameters including types of CNT, electrical conductivity of CNT, curing temperature and stirring rate. They

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found that the sensitivity of the sensor increases with decreasing CNT concentration, curing temperature and increasing stirring rate. Gao et al. [9,10] found that three-roll-milling (TRM) technique provides highly uniform dispersion of CNTs in epoxy composite for damage monitoring, while non-uniform dispersion of CNTs is obtained using nanotubes-containing fiber sizing agent. They also observed good correspondence between the repeated impact loading and the change in electrical resistance for thick laminated glass/epoxy/CNT composites. Rosca and Hoa [11] obtained more uniform dispersion of CNTs in epoxy by TRM compared to ultrasonication. Naghashpour and Hoa [12,13] dispersed CNT in epoxy matrix to make glass/epoxy/CNT composite laminates using TRM. They indicated that the change in through-thickness electrical resistance correlates well with the change in through-thickness strain for the laminate subjected to a thickness-wise load. Thostenson and Chou [14] showed that cracking in glass/epoxy/CNT composite samples can be detected based on the increase in electrical resistance. The aforementioned efforts show promise for damage and strain monitoring of small composite samples using CNT networks. Naghashpour and Hoa [15–18] demonstrated a novel SHM technique to detect, locate and quantify damage in large polymer composite structures using CNT networks. They proposed two criteria for producing self-sensing in large PMC structures that consist of uniformity of CNT networks distribution and sensitivity of CNT networks for damage detectability. They detected, located and quantified damage created by drilled holes and by impact in large composite structures made of glass fiber/epoxy/CNTs [15,18], Kevlar fiber/epoxy/CNTs [16] and carbon fiber/epoxy/CNTs [17]. Normally, the amount of CNTs needs to be more than the percolation threshold to assure electrical conductivity. This value is usually determined using small size samples. For larger structures, in order to account for the lack of uniformity in dispersion, the amount of CNTs used should be higher than the percolation threshold. As such, one would expect that more CNTs used would provide better sensitivity for damage detection. However this may not be the case. For the ease of discussion, let us denote the amount of CNTs to assure electrical conductivity anywhere inside a large composite structure be the **lower limit**. More CNTs than the lower limit would make the materials more conductive. However, the higher conductivity may reduce the sensitivity to the occurrence of damage. Table 1 (taken from reference 15) shows this phenomenon. In the work shown in Ref. [15], plates made of glass/epoxy/CNTs were made. Grid points were attached to the plates and electrical resistances between the grid points were measured. The amounts of CNTs were varied and the distribution of electrical resistance is shown in Fig. 1. Even though it was found in Ref. [11] that the percolation threshold of CNTs in epoxy was 0.18 wt%, Fig. 1a shows that there is large non-uniformity in electrical resistance from point to point (for a plate of dimensions (559 × 330 mm<sup>2</sup>)) when the amount of CNTs is 0.2 wt%. The non-uniformity still remains even at 0.25 wt% CNTs. Uniformity in resistances appears for 0.3 wt% CNTs (see Fig. 1b) and 1 wt%. The sensitivity for damage detection was obtained by measuring the changes in resistance between the grid points when some damage, such as a hole, was created inside the plates. With the introduction of a hole of 6.4 mm diameter, plates containing 1 wt% CNTs show a change in resistances of 0.38% while

plates containing 0.4 wt% CNTs show a change in electrical resistance of 1.9% (see Table 1). The largest change in resistances is seen in plates containing 0.3 wt% CNTs with 4.8% change. The optimal amount of CNTs for damage detection in this case is 0.3 wt% CNTs (see Table 1). As long as the amount of CNTs is more than a certain lower limit to assure uniformity in electrical resistances, the sensitivity for damage detection will be for the plates containing the smallest amount of CNTs. An explanation for this phenomenon requires further investigation. This paper presents the results from an investigation on the effect of conductivity on the sensitivity for damage detection.

## 2. Experimental methods

### 2.1. Materials

Two different types of materials are considered. One has excellent conductivity (copper purchased from McMaster-Carr) and the other is a composite material made of glass fiber/epoxy/0.30 wt% CNTs. The multiwalled carbon nanotubes (MWCNTs) in the composite having 95% purity, diameters of 2–20 nm and lengths of 1 μm to more than 10 μm were purchased from Bayer Material Science. Three layers of glass fabric (15 oz/yd<sup>2</sup>) purchased from HL were used as reinforcement. Plasto, Epon 862 and EPIKURE W purchased from Miller-Stephenson chemical company were utilized as epoxy and curing agent, respectively.

### 2.2. Methods

#### 2.2.1. Fabrication

The epoxy resin was first mixed with curing agent (26.4 wt%). Then, 0.30 wt% MWCNTs [15] was dispersed in the epoxy matrix using a three roll mill (EXAKT 80E, EXAKT Technologies Inc.). The plates were fabricated from modified epoxy matrix and three layers of glass fabrics using hand lay-up method. The plates were cured using an autoclave.

#### 2.2.2. Sample specification, arrangement of electrical connections

Many grid points were attached to these plates with dimensions (305 × 305 mm<sup>2</sup>) as shown in Fig. 2. The grid points were arranged concentrically and along four radial directions (0°, 45°, 90° and 135°), in order to detect damage that is created by drilling a hole at the center point of the plate. Holes of diameters of 1.6 mm, 3.2 mm, 4.8 mm, 6.4 mm, 8 mm and 9.6 mm were successively drilled at the center of the plates. The electrical resistances before and after the occurrence of these holes were measured. Note in Fig. 2b that there are 8 locations surrounding the center point for a particular radius distance. At each location, there are two grid points. This utilizes a technique called the four-electrode method to measure electrical resistance in materials that are relatively conductive. In Fig. 2b, one is interested in measuring the electrical resistance at two regions across the center point of the plate. For example, the resistance between region 1 and region 5 is shown in the encircled area in Fig. 2b. Region 1 has one grid point for current injection (1I) and one grid point for voltage measurement (1V). The same thing goes for region 5. The current is passed through grid points 1I and 5I (red

**Table 1**  
Comparing electrical resistance, uniformity of MWCNTs distribution and sensitivity of resistance change for plates containing different MWCNTs concentrations [15].

CNTs concentration (wt%)	0.20	0.25	0.30	0.40	1.00
Average Electrical Resistance (AER) (Ohms)	$0.95 \times 10^{12}$	$24.4 \times 10^6$	$276.6 \times 10^3$	$64.8 \times 10^3$	$2.5 \times 10^3$
Standard Deviation (Ohms)	$2.35 \times 10^{12}$	$32 \times 10^9$	$79.67 \times 10^3$	$18.1 \times 10^3$	$0.44 \times 10^3$
Coefficient of variation (%) = (SD/AER) × 100	247.4	131.1	28.8	27.9	17.4
Change in resistance (%) due to hole (6.4 mm) drilled			4.8	1.9	0.38

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