



**HAL**  
open science

## Strain Sensors Based on Carbon Nanotube - Polymer Coatings

Krzysztof Grabowski, Paulina Zbyrad, Alan Wilmański, Tadeusz Uhl

► **To cite this version:**

Krzysztof Grabowski, Paulina Zbyrad, Alan Wilmański, Tadeusz Uhl. Strain Sensors Based on Carbon Nanotube - Polymer Coatings. EWSHM - 7th European Workshop on Structural Health Monitoring, IFFSTTAR, Inria, Université de Nantes, Jul 2014, Nantes, France. hal-01022024

**HAL Id: hal-01022024**

**<https://inria.hal.science/hal-01022024>**

Submitted on 10 Jul 2014

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

## Strain sensors based on carbon nanotube – polymer coatings

**K. Grabowski<sup>1</sup>, P. Zbyrad<sup>2</sup>, A. Wilmański<sup>2</sup>, T. Uhl<sup>1</sup>**

<sup>1</sup>AGH University of Science and Technology, Department of Robotics and Mechatronics  
al. Mickiewicza 30, Kraków Poland

<sup>2</sup>AGH University of Science and Technology, Department of Ceramics and Refractories  
al. Mickiewicza 30, Kraków Poland

kgrabow@agh.edu.pl

### ABSTRACT

In this work there have been investigated the potential usage of the CNT's as strain sensors for the structural health monitoring based on the spray coatings. Experimental work was performed on the metal and glass-reinforced composites. Multiwalled Carbon Nanotubes (MWCNTs) were mixed with different matrix materials (acrylic and epoxy) and then applied to the test material with the use of two techniques (screen printing and spray coating). Furthermore, sensors were investigated using SEM. Response of the sensors was measured due to the tensile test of the specimens..

**KEYWORDS :** *Carbon Nanotubes, strain sensors, CNT/epoxy, CNT, screen printing, spray coating*

### 1. INTRODUCTION

Carbon Nanotubes have been intensively investigated after Ijima's publication in 1991[1]. CNT's structure consist of concentric cylinders with a few nanometers diameter. The length of carbon nanotube is up to hundreds of micrometers. The structure of CNT base on hexagonal rings, stress and force can be distributed due to their geometry. Carbon nanotubes are hundreds of times stronger than steel and they are also extremely elastic[2]. This is partly due to their hexagonal geometry, which can distribute forces and stresses over a wide area, and partly due to the strength of the carbon-carbon links. They have unusual electronic properties derived from the 'free' electrons left at the surface of the tubes after the sp<sup>2</sup> hybridization of the carbon orbitals. Simple electronic devices including diodes, switches and transistors have recently been made using nanotubes. These devices are much smaller than their silicon equivalents that are currently used in computer chips. Several fields now take advantage of the exceptional properties of carbon nanotubes. Their significant properties make them great candidate for multifunctional sensors.

#### 1.1 APPLY TECHNOLOGIES OF CNT/POLYMER COMPOSITES

Applying CNT sensors on desired surfaces is usually complex and multistage process. Also the surfaces have to be flat, in many cases, in order to have a good adherence of the sensor piece. Here we review a few examples of them.

Thin-film sensors can be fabricated by depositing dichloro-ethane-dispersed SWNTs onto SiO<sub>2</sub> substrates prepatterned with gold electrodes[3]. Sumanasekera et al. prepared sensors by lightly pressing the fibrous powder into thin pellets or mats[4]. Knite et. al. pressed and vulcanized polyisopren-CNT composite in a form of sheets to prepare multifunctional sensor[5]. PMMA-SWNT composite has been prepared also in a form of a sheet in a Teflon mould, which involved long mixing in elevated temperature and prolonged curing in an vacuum oven[6]. Another preparation method is filtrating a suspension of CNTs with a surfactant on a cellulose sheet

membrane, than washing out the surfactant and applying on a substrate, with additional heating for faster water evaporation and baking stage in 600°C[7].

Layer-by-layer assembly method can be employed for different curvatures of substrate, but the element on which the sensor is deposited has to fit in the solutions containers. This process is time consuming, first layer is a positively charged solution like PVA than the negatively charged polyanionic CNT dispersion in 1.0 wt% aqueous solution of poly(sodium 4-styrene-sulfonate)[8].

Nanotube-epoxy based sensor is a mixture of epoxy resin and MWCNT. Preparation is as follow, three-roll milling and a vacuum assisted resin transfer molding was used to fabricate the epoxy-fiber composites with embedded nanotubes[9]. In previous article authors presented MWCNT/epoxy based sensors' preparation[10].

Buckypaper can be utilized to create a strain sensor. Addition of a latex rubber cement dissolved in decane sprayed on the surface increases flexibility of the sensor[11].

An UV curable urethane acrylate was used for creating a composite with SWCNTs. Nanotubes were dispersed in liquid urethane acrylate by ultrasound and then the hardener was added and mixed mechanically. The mixture was spread on a glass slide and a doctor blade was used to shear the liquid and promote nanotube orientation. The 150 mm thick film was immediately cured by exposure to an UV source so that relaxation of the orientation is minimal. The manual application with a scalpel is not reproducible and can create significant differences between sensors[12].

Next method is attaching bucky paper made of SWCNT to a brass plate by a layer of PVC as an adhesive and an electrical insulator under vacuum is quite simple, still requiring a flat surface[13].

Ramaratnam used Poly(vinylidene fluoride-trifluoroethylene) (P(VDF-TrFE)), a copolymer of PVDF for blending it with nanotubes. Polymer pellets were dissolved in an organic solvent, N,N-dimethylacetamide and the same was done for the CNTs. Both the solution and the suspension were mixed and sonicated and the highly viscous solution was poured on an aluminum plate and a wet film applicator was used to draw a thin film of required thickness on the aluminum base. Heat treatment evaporated the solvent so that the thin film could be peeled of. Difficulties of this method are possibilities of damage of thin film during preparation[14].

Spray coating is one of the easiest method for thin CNT-film preparing. Therefore we have focused in this work to prepare sensors which would be suitable for SHM applications. Moreover in previous article we have presented screen-printing method for applying CNT-sensor on based material. We compared those two kind of sensors.

## 2. Experiment

To prepare sensors MWCNTs have been used. The multiwalled carbon nanotubes were ordered from the Sigma-Aldrich (O.D.  $\times$  L: 6-9 nm  $\times$  5  $\mu$ m, diameter: 5,5 nm-mode, 6,6 nm-median). Spray ingredients were obtained by Mipa, for preparation Universal-Prefilled-Spray.



Figure 1: Sprayed sensor

First step was to prepare 50 ml of solution by dissolving 30 ml acrylic acid in 20 ml diluent. Then weighed amounts of CNTs were added. Thus prepared mixture was stirred manually for 20 minutes. Next step was to improve dispersion of Carbon Nanotube in the mixture. In that case

ultrasound probe was used for 3 minutes. After that mixture was placed in an injector and 50 ml clearcoat was bubbled from in order to clean the cartridge of injector. Operation was performed to supplement mixture to normalized quantities of 100ml. The working pressure of spray was about 2 bars. Finally opacity test was performed, which confirmed the proper preparation of a spray. CNT-film was sprayed on based material and tested.

### 3. Results

In order to check the performance of the sensors we have first, inspected the structure of the material using Scanning Electron Microscope. Moreover we have measured the electrical response of the sensor which were treated as a typical strain gauges applied on the tested material. Also for investigation of sensors SEM pictures were taken.

#### 3.1 SEM investigation

Prepared sensors were investigated by Scanning Electron Microscope and SEM pictures of sensors were taken. SEM studies on sprayed sensors shows concentration and structures of CNT-films.

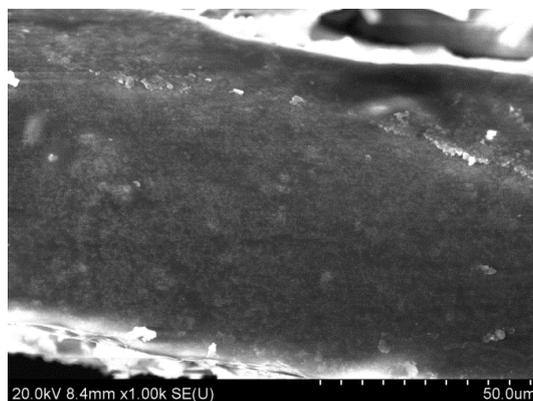


Figure 2. SEM image of screen printed sensor

Figure 2 shows Carbon Nanotubes in epoxy matrix which was done in our previous work. Picture shows that nanotubes are dispersed in polymer really good but they tend to agglomerate in bigger bundles.

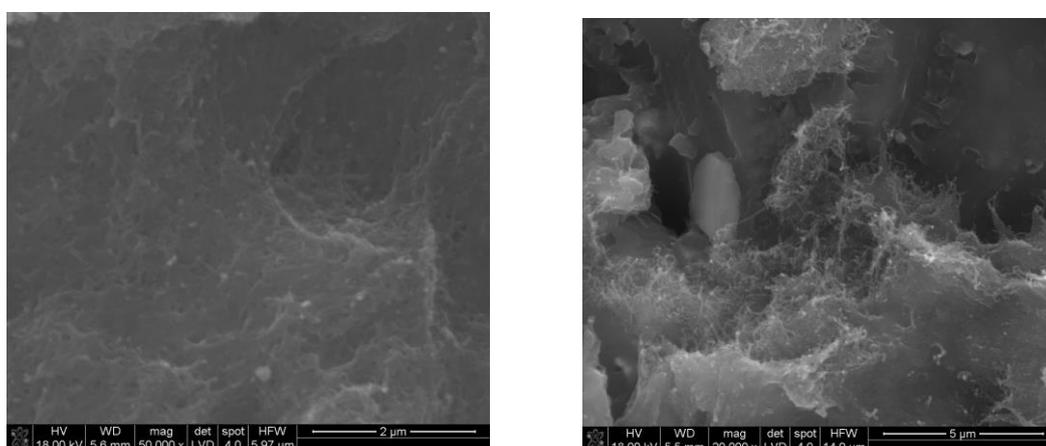


Figure 3. SEM images of sprayed sensors

Figure 3 shows that Carbon Nanotubes are dispersed in acrylic matrices. However when we compare it to the structure of the sensor which was screen printed we may see that the sprayed sensors contain more air bubbles.

### 3.3 Voltage Response

To test electrical response of the sensors due to the elongation there was prepared the setup consisting of the Wheatstone Bridge connected to SCADAs Mobiles and LMS Test.Lab (Figure 4). Wheatstone Bridge was designed as it was for measurement of the strain gauges. Samples were then tested using Instron 8872 servo hydraulic machine. Samples were subjected to the tensile test which caused specimens to elongate for about 1 % of their original length.



Figure 4. Test sample mounted on test machine

Initial resistance was recorded before the tests. Resistances were approximately for 1.5 % and 0.75% wt. 8000  $\Omega$  and 12000  $\Omega$  respectively (averaged out of 5 samples). It shows strong dependency of wt. of CNTs and initial resistance. Considering screen printing technique resistances of the sensors was higher – approximately 15000  $\Omega$  and 25000  $\Omega$  for 0.75% and 1.5% CNT wt. respectively.

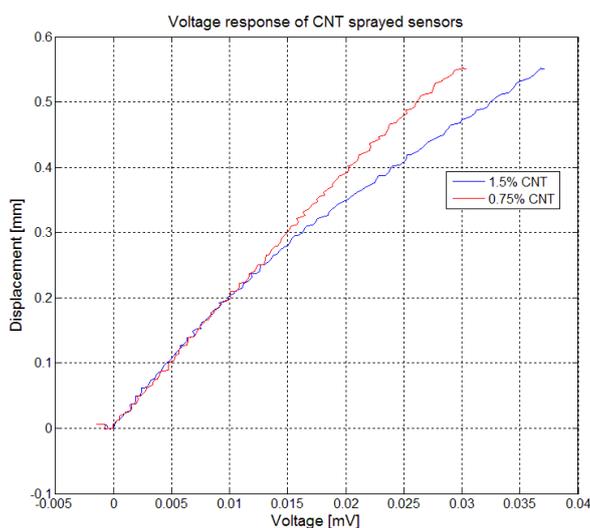


Figure 5: Voltage response of the sensors

Furthermore there was observed linear change of the resistance due to the elongation of the specimens. However, higher wt. of CNTs (1.5%) in material has shown nonlinear behavior. This is due to the deformation of the large bundles of CNTs in the matrix material.

#### 4. Conclusions

There were discussed different techniques of application of CNT as sensors for the SHM. In this work specifically, spray coating was presented as potential technique for relatively easy and lightweight application directly on the tested material. There was shown linear behavior of the voltage change to due to deformation of the sensors.

Development of the sensors presented in this work could bring a lot of efforts and problems. One of the most influencing factors is dispersion of carbon nanotubes in a matrix solution. Nanotubes aggregate formation is a direct result of high intrinsic interactions between CNTs. CNTs easily aggregate and are really difficult to separate. During a sensor work this phenomenon could give some noises in results. Ultrasonic treatment is a convenient way to mix and separate Carbon Nanotubes in polymer matrix. Disadvantage of sonication is breakage and reducing the nanotubes during ultrasonic bath. Second way to separate CNTs in a polymer matrix is a modification of carbon nanotube surface e.g. chemical surface functionalization or surface adsorption.

Another issue is applying sensor on a base material. In the presented case, the screen printing method and spray coating method were investigated. Screen printing method is widely used technique for obtaining thin layer of a nano-composite. The spray coating approach is particularly attractive deposition method. It has been proven fast and easy for various applications. However, a mixture of low viscosity is a crucial issue. Among the disadvantages of the spray coating method are difficulties in controlling layer thickness and what follows distribution of Carbon Nanotubes in polymer matrices. Nevertheless, spray technique seems suitable for application in SHM.

Further research should be focused on the performance of the sensors in different environments and considering different shapes of the sensors. Studies on CNTs' dispersion in polymer matrix should be also taken into consideration.

- 
- [1] S. Iijima, "Helical microtubules of graphitic carbon," *Nature*, vol. 354, pp. 56–58, 1991.
  - [2] J. Bernholc, D. Brenner, M. Buongiorno Nardelli, V. Meunier, and C. Roland, "Mechanical and electrical properties of nanotubes," *Annu. Rev. Mater. Res.*, vol. 32, pp. 347–375, 2002.
  - [3] Philip G. Collins, Keith Bradley, Masa Ishigami, A. Zettl "Extreme Oxygen Sensitivity of Electronic Properties of Carbon Nanotubes" *Science* 287, 1801 (2000)
  - [4] G. U. Sumanasekera,1 C. K.W. Adu,1 S. Fang,2 and P. C. Eklund "Effects of Gas Adsorption and Collisions on Electrical Transport in Single-Walled Carbon Nanotubes" *PHYSICAL REVIEW LETTERS* VOLUME 85, NUMBER 5 31 JULY 2000
  - [5] Maris Knite, Igors Klemenoka, Gita Shakale, Valdis Teteris, Janis Zicans "Polyisoprene-carbon nanocomposites for application in multifunctional sensors" *Journal of Alloys and Compounds* 434–435 (2007) 850–853
  - [6] Inpil Kang, Mark J Schulz, Jay H Kim, Vesselin Shanov and Donglu Shi "A carbon nanotube strain sensor for structural health monitoring" *Smart Mater. Struct.* 15 (2006) 737–748
  - [7] Jennifer Sippel-Oakley, Hung-TaWang, Byoung S Kang, ZhuangchunWu, Fan Ren, Andrew G Rinzler, and Stephen J Pearton „Carbon nanotube films for room temperature hydrogen sensing" *Nanotechnology* 16 (2005) 2218–2221
  - [8] Kenneth J Loh, Junhee Kim, Jerome P Lynch, Nadine Wong Shi Kam and Nicholas A Kotov "Multifunctional layer-by-layer carbon nanotube-polyelectrolyte thin films for strain and corrosion sensing" *Smart Mater. Struct.* 16 (2007) 429–438
  - [9] Erik T. Thostenson and Tsu-Wei Chou" Carbon Nanotube Networks: Sensing of Distributed Strain and Damage for Life Prediction and Self Healing" *Adv. Mater.* 2006, 18, 2837–2841
  - [10] K. Grabowski, P. Zbyrad, T. Uhl "Development of the strain sensors based on CNT/epoxy using screen printing " *Key Engineering Materials* ; ISSN 1013-9826. — 2014 vol. 588, s. 84–90.

- 
- [11] X Li, C Levy and L Elaadil „Multiwalled carbon nanotube film for strain sensing” *Nanotechnology* 19 (2008) 045501 (7pp)
- [12] Qing Zhao, Mark D. Frogley, H. Daniel Wagner „Direction-sensitive strain-mapping with carbon nanotube sensors” *Composites Science and Technology* 62 (2002) 147–150
- [13] Prasad Dharap, Zhiling Li, Satish Nagarajaiah and E V Barrera „Nanotube film based on single-wall carbon nanotubes for strain sensing” *Nanotechnology* 15 (2004) 379–382
- [14] Arun Ramaratnam and Nader Jalili "Reinforcement of Piezoelectric Polymers with Carbon Nanotubes: Pathway to Next-generation Sensors" *Journal of Intelligent Material Systems and Structures* 2006 17: 199