Title: **On the Dynamic Acquisition of Electrical Signals for Structural Health Monitoring of Carbon Nanotube Doped Composites**

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ABSTRACT

In the last decades, the interest on fiber reinforced polymers (FRPs) has increased due to their mechanical properties and weight saving potential. This has led to the development of novel inspection techniques, being the failure modes of composite structures complex to identify. In this regard, carbon nanotubes (CNTs) have been widely used for Structural Health Monitoring (SHM) purposes, thanks to their excellent electrical properties and piezoresistive behavior, which ensure for SHM higher sensitivities than other conventional techniques. The correlation between electrical properties and mechanical behavior on CNT doped nanocomposites is well known, and several analytical and numerical models have been proposed. However, most of these studies considered static and quasi-static load configuration, whereas the dynamic electromechanical behavior still unknown. This work aims to develop a novel technique for dynamic acquisition of electrical signals for SHM using CNTs. For this purpose, impact and vibration analyses on multiscale CNT-doped glass FRP have been performed. Besides, fatigue tests on CFRP lap joints bonded by a CNT-doped adhesive have been carried out. SHM is carried out by means of voltage measurement during the tests. A potentiometer is used to keep the current constant so that voltage variations correspond to resistance changes across the specimen. Dynamic acquisition is carried out using a high frequency acquisition system. The results of impact, vibration and fatigue tests prove the validity and applicability of the acquisition system. The electrical signals have been correlated with the mechanical behavior of the material, providing information on the system dynamic response and, simultaneously, on damage propagation mode.

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1. INTRODUCTION

The use of fiber reinforced polymers (FRPs) in the aircraft industry has increased during the last years. Their excellent specific properties and good corrosion resistance makes them very competitive with respect to traditional alloys. However, their inspection is not easy, as they present complex failure modes. Thus, the development of novel techniques for a proper Structural Health Monitoring (SHM) is necessary. To date, there are many studies on fiber Bragg gratings (FBGs), guided Lamb Waves, and other techniques, such as thermographic analysis for SHM purposes on composite structures [1-6]. Nevertheless, some techniques only give local information about the structural damage [1,3,7,8], while others required complex numerical models to properly correlate the acquired signal to the mechanical performance of the composite structure [4].

In this context, the use of carbon nanoparticles and carbon nanotubes (CNTs) for SHM applications has widely increased over the past years [9-11]. This is due to their piezoresistive behavior and to the tunneling effect between adjacent particles, making the material itself a sensor [12-14], and leading to high values of sensitivity [15,16]. Thus, they not only give local information, but a general overview about the structural integrity. Nevertheless, there is still some lack of knowledge about how the electrical response and the mechanical material behavior are correlated. Although there exist some literature studies correlating changes of electrical resistance to typical failure mechanisms on composites [17-19], it is necessary to get a deeper understanding of failure vs. electrical response correlation. More specifically, the electromechanical behavior owing to dynamic loading is not well understood. There also are many research studies on SHM monitoring by CNTs on adhesive joints [20,21], proving the great potential of CNTs to detect debonding and crack propagation. More specifically, fatigue testing has also been monitored by using CNT-doped adhesives [22,23]. However, they do not give detailed information about electromechanical behavior in a single cycle, but rather show a general overview about electrical response trend as a function of number of cycles. Another recent application of CNTs as embedded sensors regards the assessment of impact damage. In this context, there are many studies on pre- and post-impact diagnosis using Electrical Impedance Tomography (EIT) techniques [24-26] and by comparing the electrical response of the pristine and the damaged specimens [10,27]. However, they do not give detailed information about the dynamics, but only about the quasi-static behavior.

This work aims to develop a novel technique for dynamic acquisition of electrical signals using CNT percolating networks. Specifically, the real-time monitoring of three mechanical systems subject to dynamic excitation and characterized by increasing complexities has been attempted. First, the dynamic behavior of the CNT-based measurement system has been characterized within low frequency range and compared with a strain gauge output for a cantilever beam CFRP specimen. The same measurement system has been used to measure the fatigue load on a single lap joint (SLJ) with CNT-doped adhesive. Finally, the low velocity impact monitoring has been performed on CNT-doped GFRP specimens. A summary of the research activities has been provided in the following, proving the huge potential of both CNT-doped multiscale composites and the developed measurement technique for SHM purposes.

2. MATERIALS

Materials design

Multiscale plates for preliminary vibration analysis and impact tests have been prepared by using multi-walled CNTs (MWCNTs) embedded in an epoxy resin and glass fiber (GF) as reinforcement. MWCNTs (NC7000 by Nanocyl) have a length up to 1.5 μm and an average diameter of 9.5 nm. The resin is a bi-component epoxy with low viscosity (Araldite LY556, by Huntsman), and is mixed in 100:23 proportion with a hardener called (XB 3473, by Huntsman). GF (EBX600) are supplied by Selcom. The laminates have the following stacking sequence: $[0/90/+45/-45]_s$.

The same MWCNTs have been selected to dope an epoxy-based adhesive film (FM 300KN supplied by Cytec), used for the SLJ fatigue tests.

Manufacturing

MWCNT/epoxy mixtures are prepared by three roll milling, using an EXAKT 80E mini calender. The CNT content is set to 0.1% wt., as it provides a good electrical sensitivity [28]. After the mechanical dispersion, a degasification step is carried out at 80 ºC for 15 minutes, to remove the air entrapped in the mixture. Then, the hardener is added to the mixture prior to its infiltration into a GF preform. Multiscale GFRP plates are manufactured by Resin Transfer Molding (RTM). MWCNT/epoxy mixture is injected at a constant pressure of 2 bar and temperature of 80 ºC, to facilitate the infiltration process, then cured for 8 h at 140 ºC.

Adhesive joints are manufactured by secondary bonding of CFRP substrates with the CNT-doped adhesive film. CNTs (0.1% wt.) are first dispersed in distilled water; then a surfactant (sodium dodecyl sulfate, SDS) is added to improve CNT dispersion, reaching 0.25% wt. [15]. The aqueous dispersion is sonicated for 20 min (0.5 cycles at 50 Hz) using a horn sonicator (UP400S supplied by Hielscher). Then, the CNT dispersion is sprayed over the adhesive surface using an airbrush, placed at 40 cm distance, and a pressure of 1 bar for 0.5 s to achieve a homogeneous distribution of CNTs over the sample. The CFRP bonded joints are then subjected to a two-stage low-pressure high-temperature curing.

3. METHODS

Vibration analysis

Vibration analyses have been performed on multiscale CNT-doped GFRP, by means dynamic testing. To measure the deformation on the applied specimen, two strain gauges are mounted on the specimen (half-bridge configuration) and a conditioning unit (HBM Scout 55) is used to amplify the measured signals. The specimen (rectangular: 133 x 33 x 3 mm) is mounted as a cantilever beam, with the load applied at the free edge by means of an electrodynamic shaker. Dynamic testing is carried out controlling the shaker head in open loop condition with a signal waveform generator. A plastic rod, connected to the shaker head, is used to apply displacement to the cantilever beam and an accelerometer is used to monitor the acceleration at the beam tip. Sine wave with constant amplitude (40 mV) and varying frequency (between 5 and 40 Hz) provides the driving voltage signal. The excitation time is kept to 10 s at each frequency and sampling frequency is set to 90 Hz. RMS spectral amplitude at the forcing frequency has been computed for each time history, and compared with the strain gauge measurements.

Fatigue testing

Four fatigue tests have been carried out on CFRP SLJs, bonded with a CNT doped adhesive film. Tests have been performed in a servohydraulic testing machine (*MTS810*), setting a peak load of 7 kN, a load ratio R = 0.1 and a frequency of 10 Hz. These parameters have been chosen to obtain a finite fatigue life of approximately 10^5 cycles, according the S-N curve for adhesives [29]. The experimental setup is depicted in Figure 1a-b.

Impact testing

Low-velocity impact testing have been carried out on multiscale CNT doped GFRP, by following the standard (ASTM D7136). The specimen size is 100mm width, 3mm thickness and 150mm length. The experimental setup includes a fixture of two 20mm thick steel plates to hold the specimen and an impactor, a mass of 0.75 kg with a tip diameter of 16mm dropped from a specified height. The impactor is instrumented with a Kistler Quartz Force Link Type 9331B (±20 kN range), allowing the load history to be recorded. The impactor fall was guided by a droptower (i.e. 1.5m long polycarbonate tube, with a diameter of 75mm and a thickness of 4mm). Holes along the tube are drilled to avoid air compression during the fall; friction between the impactor and the guiding tube is considered negligible, while a clearance of 1mm is guaranteed along the length of the tube. The impactor is held in position by an electromagnet at the desired height: when turned off, the electromagnet drops the impactor on the underlying specimen. As permitted in the referred standard, rebound hits are averted by simply sliding an aluminum plate over the fixture after the first impact has occurred. The impact velocity, hence the energy, is measured by means of a “speed trap” configuration: two lasers (Mikroelektronik M5L/20) are placed at a 41mm distance, recording the impactor crossing time. The acquisition of the signals of load cell and lasers is synchronous (sampling frequency of 51.2 kHz) using a NI9234 module by National Instruments. The experimental setup is shown in Figure 1c,d,e.

Structural Health Monitoring

SHM during testing is carried out by continuously measuring the voltage between two points of the samples. During loading, the specimen is fed by a stabilized current generator (i.e. 0.5 mA). Copper wire electrodes are attached with silver ink to the sample surface, sealed with an adhesive to protect them from accidental damaging, and used to acquire the voltage in different channels associated to different electrode positions. This setup allows measurement of the voltage variations, influenced by the resistance changes across the specimen. Noise reduction has been achieved by grounding the entire system. For impact testing, a digital multimeter (Agilent 34401A) is used offline, before and after each drop test, to verify the stability of the current during the tests. Dynamic acquisition is carried out using a high frequency acquisition system.

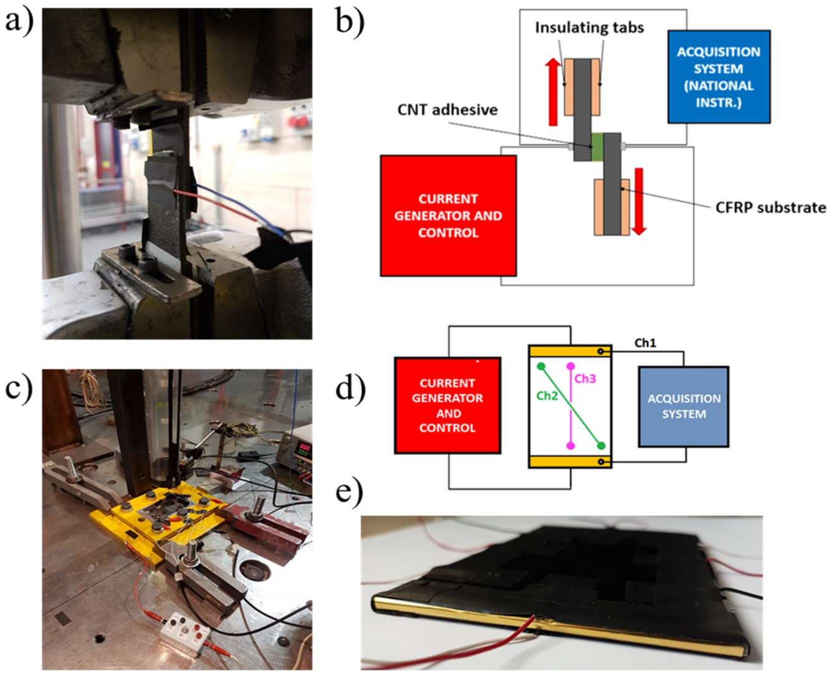

Figure 1. a) Testing of a SLJ with CNT-doped adhesive. b) Schematic of a SLJ shear test and SHM system. c) Impact test setup. d) Schematic of sample with the SHM system (i.e. current generator and acquisition system); highlighted the electrical channels. e) GFRP sample for impact tests with metallic foils providing homogeneous current and cables for voltage acquisition.

4. RESULTS AND DISCUSSION

Vibration analysis shows that the CNT-doped sample is able to detect the input vibration, as testified by measured time domain signals (Figure 2a). The measured spectral amplitudes decrease with frequency (Figure 2b), showing an overall agreement with the strain gauges' measurements. This result was expected since the beam tip displacement was reduced increasing the testing frequency. The CNT structures proved a high potential for sensing an input vibration, opening the way to develop proper control strategies in different application fields, like space and aerospace structures.

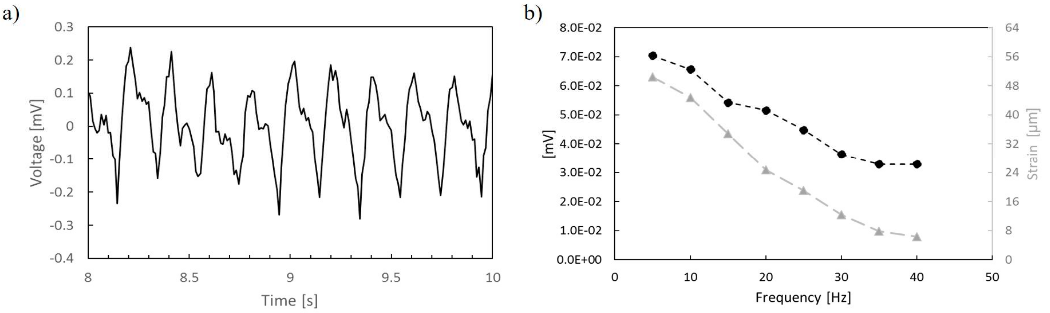

Figure 2. a) Measured time domain voltage signals (5 Hz excitation frequency), showing the ability of CNT-doped samples to detect vibrations. b) Measured spectral amplitude vs. frequency for a CNT-doped composite sample: a decreasing trend showing an agreement with signals from strain gauges.

The fatigue testing carried out on SLJs showed that CNT-doped adhesive films are able to monitor the crack propagation, by measuring the variation of the electrical resistance through the thickness. Not only the crack growth is correlated to the increase in the electrical resistance as a function of the number of cycles but also to a modification of the shape of the measured signal as a function of the load cycle, as visible in Figure 3a. A larger increase corresponds to a faster crack propagation, occurring when the joint is close to failure. Therefore, it is possible to predict the final failure by observing the changes of the electrical resistance. Also, no decrement of the fatigue life of CNT-doped adhesive joints compared to the neat ones has been observed, thus proving the huge potential for structural application of CNT-doped adhesives.

Figure 3b shows the outcome of an impact test carried out with an impact energy of 3.5 J. By observing the electrical CNT outputs in comparison with the load cell signal, it is possible to distinguish three phases: 1) an initial instability of the electrical signals, owing to the impact-induced compression in the hit region, with the immediate formation of the first damage [30,31], such as debonding and matrix cracking (at this stage, the impactor load favorites the formation of alternative links thus preventing the rise of the electrical resistance), 2) an increase in the electrical resistance during specimen unloading, probably associated with the opening of electrical links in correspondence of damage, and 3) subsequent rises and drops corresponding to the oscillation of the sample after the impactor leaves the laminate surface. The baseline signal modifications due to impact can be also clearly identified in the same figure.

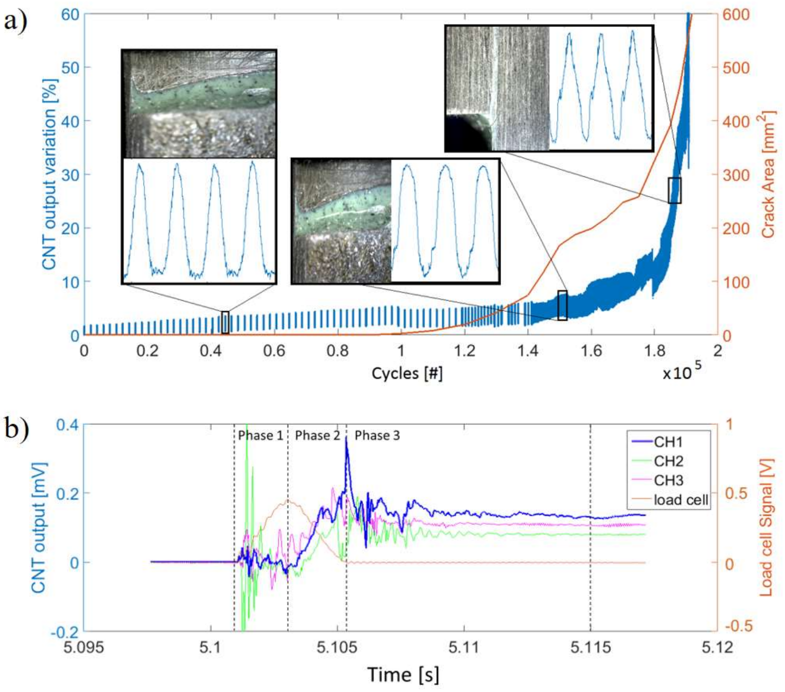

Figure 3. a) Electromechanical behavior during fatigue test of SLJ. Red and blue lines correspond to crack size and the electrical, respectively. The magnifications show the variation in resistance when the sample is subject to cyclic loading and the progressive failure. b) Electromechanical outcome of impact test carried out with an impact energy of 3.5 J.

5. CONCLUSIONS

This preliminary study proves the possibility of using CNTs as embedded structural sensors for dynamic acquisition of different types of signals, allowing for SHM of composite materials. The results of vibration, fatigue and impact tests demonstrate the validity and applicability of the proposed dynamic acquisition system: the electrical signals have been correlated with the mechanical behaviour of the material, providing information on the system dynamic response and, simultaneously, on the way damage is initiating and propagating. The dynamic acquisition, carried out using a high frequency acquisition system, ensures proper noise reduction, through a complete isolation of all the electrically conductive elements and a correct grounding of the interconnected systems. These preliminary results provide the basis for the development of a proper SHM procedure based on the self-sensing capabilities of CNTs.

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