# Development and testing of nano-filled polymer sensors for aerospace structural health monitoring

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#### **ABSTRACT**

Structural Health Monitoring deals mainly with structures instrumented by lightweight and low-costs sensors to acquire and analyze static and dynamic strain fields during operations and correlate them with the health status in order to reduce maintenance costs and weight of aerospace composite structures. Depending on the operative frequency bandwidth, SHM methodologies can be based on low-medium frequency approaches, where static strains and/or modal parameters are monitored within the operative conditions searching for deviations respect to the "healthy" status of the structure, or, alternatively, they can rely on the analysis of ultrasound strain waves propagation signals acquired on the structure. Piezoresistive sensors based on a low percentage of carbon nanotubes or graphene like particles dispersed into a polymer or any other insulating matrix have been developed and characterized during latest years since they present the advantages of easy application and low costs and weight addiction to the primary structure. Their sensitivity and gauge factors can vary a lot depending from the content and dispersion of nano-filling material, from the matrix type and viscosity, from the dimensions of the sensor and from its shape. This work presents some results related to new typology of sensors obtained by melt compounding of polystyrene with different amounts of carbon nanotubes and graphene nanoplatelets and characterized by varying extensions and shapes applied on beams and plates like structures. These materials have been produced and characterized by the authors in terms of its static and dynamic sensing capabilities as well as employed in different geometric configuration for health monitoring purposes. Interesting sensing capabilities resulted for both static and dynamic strain fields monitoring, and signal analysis dedicated approaches have been investigated to enhance the sensitivity characteristics.

**Keywords:** Nano-filled piezoresistive sensors, Structural Health Monitoring, sensor mechanical characterization.

### 1. INTRODUCTION

Real-time analysis methods have been studied for some time in the field of structural monitoring (SHM: Structural Health Monitoring) in many structural engineering research fields and in detail within the area of aerospace structures where conditioned monitoring is becoming a very relevant topic. This is necessary because knowing the

structural integrity of an element is essential to predict its residual operational life, organize maintenance operations, inspections or replacements in the case of components that cannot be accessed easily or for elements made of composite materials, where internal damage, fatal to their integrity, can be hardly detected only thanks to sophisticated non-destructive technologies (i.e. ultrasound C-scan).

However, installing permanent sensors is a challenge, especially in aerospace applications, where cost and weight containment is critical.

For this reason, attention to the creation of sensors made of nano filled materials has grown greatly during last years. These are obtained by combining electrically conductive nanocomponents, such as graphene nanoplates (GNPs: Graphene NanoPlatelets) and/or carbon nanotubes (CNT: Carbon NanoTube), to a primer insulating material, like, for example, glass fiber or (thermoplastic) polymer surfaces. The conductive nanocomponents are typically present in minimal quantities compared to the overall weight of the structure to preserve the structural characteristic behaviour of the primer insulating material. The monitoring takes place by applying electrodes to the sensor and evaluating, under the application of a static or dynamic load, the change in resistance of the material to a certain deformation: for this reason, the Gauge Factor is generally used as a reference, represented by the ratio between the relative variation of the resistance and the deformation obtained:

$$GF = \frac{\Delta R/R}{\Delta L/L} \tag{1}$$

The mechanism that governs electrical conduction in a nanofilled material is known as 'percolation': it consists in the creation of a continuous network of conductive nanoparticles through the insulating polymer matrix. Increasing the volume of filling material φ (nanofillers) increases the possibility of obtaining groupings, connected to each other via a 'tunnel effect', so that the electrical conductivity of the material also gradually increases. The critical volume value of  $\varphi$  at which there is an increase of several orders of magnitude in the conductivity of the material is called the 'percolation threshold', beyond which the properties of the material tend to decrease until they stabilize. The distribution of the filler is fundamental for determining the critical φ. In fact, among the main difficulties encountered in the creation of nanofilled materials is the need to uniformly distribute the nanoparticles in the material, also because it influences the possibility of indiscriminately evaluating the deformations by applying the electrodes in different points of the sensor. This work presents some results related to new typology of sensors obtained by melt mixing of polystyrene with different amounts of carbon nanotubes and graphene nanoplatelets and characterized by varying extensions and shapes applied on beams and plates like structures. Different materials obtained changing the primer insulating material as well as the percentages of CNT and GNPs have been tested both under static loads/deformation and dynamic ones obtaining some preliminary but interesting results.

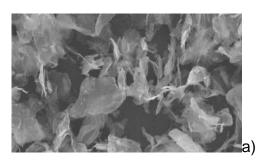
### 2. THERMOPLASTIC TRANSDUCER DESIGN AND MANUFACTURING

In line with recent research levers such as the ecological transition aimed to the sustainability of new products and industrial processes as well as the need for circularity, i.e. reusability/recyclability of materials at the end of their useful life, this research

considers the use of flexible thermoplastic composite components intended for electronic applications.

At present, a lot of experimental evidence are already available in the literature about the adequacy of flexible multifunctional devices with specific sensory peculiarities [1-5], adaptable to geometrically complex or rapidly moving components and obtainable with easily scalable process technologies. In this frame, an out-standing interest has been gained by items based on thermoplastic substrates, given their intrinsic recyclability with respect to thermosetting one, filled with fillers of different shape, nature and size, offering distinctive thermal, electric, optical functionalities. Among matrices, the use of thermoplastic techno-polymers such as polyimides, polyester, polyurethane and polyether ether ketones is largely established for com-ponents subjected to heavy mechanical stress but also of commodities as polycarbonate, polyolefin and polystyrene for less demanding uses.

As far as fillers are concerned, a broad spectrum of active phased validated such as non-transition-metal oxides, carbonaceous particles as nanotubes and graphene-like ones, boron nitride and so on was also investigated. In addition, hybrid formulations obtained through the inclusion of two or more particles of different nature and/or dimensions can offer synergisms usually attributed to the formation of interconnected networks between the included particles [6-9]. In this work, a hybrid composite system based on a polystyrene R850 furnished by EMI Versalis (density: 1.04 g/cc – MFR@200 °C-5kg: 4 g/10 min – HDT: 85 °C) was investigated. The matrix was modified including 3 wt% of single walled carbon nanotubes (SWCNT) with average aspect ratio L/d: 3000 produced by OCSiAI from TUBALL and 1 wt% of graphene nanoplatelets (GNP – G2Nan) by Nanesa s.r.l..



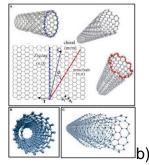


Fig. 1 Graphene Nanoplatelets [a)] and Carbon Nanotubes [b)]

In details, base constituents were melt mixed with the aid of a Brabender-like apparatus (Rheocord EC – Haake Inc.) (see Fig. 2) at 200 °C by setting the screw speed at 40 rpm for 5 min. The compound produced was granulated and transformed into a film with a thickness of approximately 300 microns using a lab-scale hot-press Collin model P400. This last operation was performed at 220 °C using the follow-ing pressure time profile: 2 min at 0 bar, 1 min at 5 bar, 1 min at 10 bar, 1 min at 20 bar; followed by uncontrolled cooling of the material to room temperature, maintaining the maximum compaction pressure (20 bar). Finally, the procedure ends with the pressure relief, the opening of the press and the unloading of the sheet of composite material from which the sensor elements used in this research were easily cut.

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Brabender batch mixer

Lab press

Fig. 2 CNR-IPCB Production facilities used for sensors production

#### 3 THERMOPLASTIC TRANSDUCER TESTS AND RESULTS

From the sheet of nano-filled composite material obtained following the procedure described in the previous paragraph, many pieces have been cut and instrumented with different electrodes configurations and tested both as stand-alone structures as well as bonded on a cantilever aluminum beam as strain sensor. Following a summary of the configurations tested is reported, intending as configuration the combination of composite material shape and electrodes locations and number. In the third configuration, the small nano-filled material sheet has been bonded close to the constraint of a cantilever beam in order to use it as "strain sensor" in that location.

### 3.1 First configuration – Electric and static tests: set-up and results

The first experimental set-up consisted in a squared plate (5cm x 5cm) made of nano-filled material (polystyrene + 3 wt% of single walled carbon nanotubes and 1 wt% of graphene nanoplatelets) with a four bonded strips of copper called A, B, C, D and four cables (numbered from 1 to 4) soldered onto each of them, except for C, on which only three cables were soldered for space reasons (See Fig. 3). In this configuration the variation of resistance between fixed points of the plate was investigated (See Fig. 4). Nano-filled materials for their own nature tends naturally to reduce the resistance when the voltage increases due to the creation of more "electric" tunnels inside the material itself.

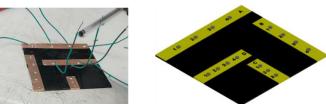
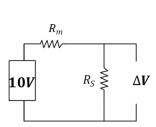


Fig. 3 First configuration of nano-filled plate – electrodes application

This behavior, anyway, is often far from being linear depending from many parameter, and, among those, from the homogeneity of the nano-materials distribution inside the material. This parameter, due to the production process, was not under control for the materials under testing. It was possible to measure the resistance between many

electrodes thanks to an external known resistance and a multimeter measuring the voltage at its electrodes by the following scheme:



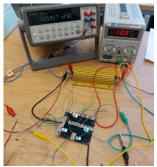


Fig. 4 (left) schematic diagram of the circuit created (10V as an example driving voltage) (right) circuit created: yellow and red cables associated with the multimeter.

In Fig. 5 are presented the results in terms of resistance variations with changing driving voltage measured by the circuit presented in Fig. 4. For the all three cases of electrodes combination the behaviour of the resistance increasing the voltage was as expected.

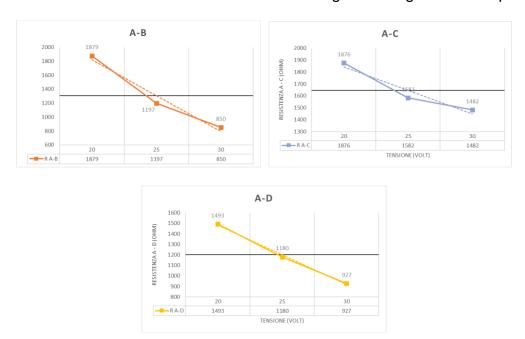


Fig. 5 Resistance (Ohm) Vs driving voltage (Volt) for different location of electrodes

Finally, for this configuration, the behaviour of resistance with a static load applied in the center of the plate was analysed in order to start characterising the electro-mechanical coupling of the nano-filled plate. For static loads tests a fixture was produced by additive manufacturing in order to constrain the plate on the four angle (by simply supporting it) as presented in Fig. 6.

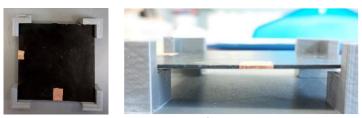


Fig. 6 3D printed support for static load tests

To measure variations of voltage at two electrodes (1A and 1D) of the plate when it was mechanically loaded and excited by a an applied voltage of 20V: a more accurate method was used by constructing a divider to evaluate it between two specific points. That was possible by knowing the values of the resistances between the different points, obtained from the preceding analysis.



Fig. 7 Voltage variations (Volt) between electrodes 1A and 1D for varying loads (gram)

3.2 Second configuration – Electric, static and preliminary dynamic tests: set-up and results

In the second configuration it was tested a plate characterized by the same shape and size of the first one presented in paragraph 3.1 by applying only four electrodes at the midpoint of each side (Fig. 8) without wrapping both surfaces.

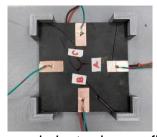


Fig. 8 Second electrodes configuration

This configuration was employed for qualitative analysis of the plate's behaviour under static and dynamic loads considering slow time-averaged acquisitions as well as higher sampling rate and frequency analysis acquisitions. Following are reported the obtained results. The first graph shown in Fig. 9 presents the time-averaged (0.1sec RMS value) value of the resistance measured between electrodes B and C for varying dynamic (impacts) and static (small loads positioned in the middle of the plate in sequential events.

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Fig. 9 Time-averaged history of resistance between B and C electrodes under dynamic (impacts) and static loads.

Furthermore, with the same set-up it was acquired the fast response (sampling rate fixed at 5000Hz) from two electrodes of the plate during the free-decaying response after an impact. The time history and the corresponding Fast Fourier Trasformed spectrum are reported in Fig. 10. The electrodes were subjected at a driving voltage of 5 V.

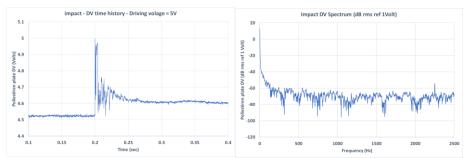


Fig. 10 Fast response of plate (DV) after an impact in time (s.f.=5000Hz) and FFT

From this first attempt of analysing fast dynamic electro-mechanical behaviour of the nano-filled plate it is clear that the response after an impact is fast enough to excite a wide band also in terms of voltage variations (as can be seen also from the spectrum where relevant response values are evident until at least 500Hz). Furthermore, from the time-history of the voltage drop it becomes evident that the voltage itself after a sudden excitation like an impact is composed by a fast behaviour (rapid oscillations in time) and a slower behaviour still conditioning the DV values after the fast oscillation tend to disappear. This characteristics will be better investigated in the third set-up configuration.

### 3.3 Third configuration – sensor bonding on a cantilever beam

The third and last set-up configuration presented in this work represent a first application of the nano-filled material as structural sensor. In order to test the structural sensing capabilities of the produced material a small rectangle (1.5cm x 3cm) of the material produced as presented in paragraph 2 has been bonded by a structural adhesive on a thin alluminum plate (size 3cm width and 30cm length) constrained as a cantilever beam (See Fig. 11). The sensor has been instrumented with four electrodes on the sides of the rectangle.

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Fig. 10 Cantilever beam sensorised with nano-filled sensor at the root and instrumented with four electrodes on the sides

The experimental set-up was made by the sensorised beam, a multichannel National Instrument acquisition board type NI USB-6466, a stabilised voltage source, a known valued resistance and a small circuit connecting all the components as from the following scheme:



Fig. 11 experimental set-up circuit scheme and sensor resistance equation

Voltages V1 and V2 were acquired by two acquisition channels of the NI board with available sampling rate up to 2 MHz. The voltage source permitted to change the excitation voltage of the sensor between 5 and 40 Volts.

A preliminary study of the electro-mechanical static behaviour was conducted measuring the resistance of the sensor at varying loads (applied at beam's tip) and excitation voltage. The results are presented in Fig. 12.

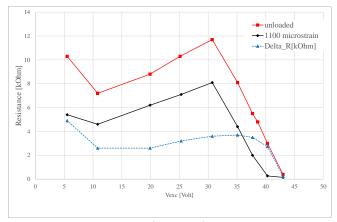


Fig. 12 Electro-mechanical behaviour of nano-filled sensor at varying static loads (at beam tip) and excitation voltage.

Finally, applying a dynamic load at the tip (impulse), the dynamic resistance from the sensor has been acquired with a sampling rate of 2KHz and, from this signal an RMS value each tenth of second has been evaluated in order to analyse both the fast and slow

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behaviour of the resistance under free response of the beam after an impulsive excitation. In Fig. 13 the two measures are presented.

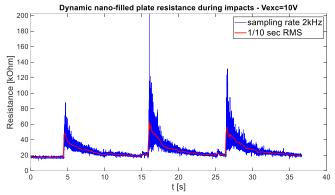


Fig. 13 Dynamic (fast and slow) resistance of the nano-filled sensor after impacts (free-response)

From Fig. 13 is possible to distinguish a fast resistance behaviour following the beam vibrations time-history pattern (blue line) combined with a slower behaviour do to a sort of electrical inertia (capacitive effect) that has always been noted during dynamic acquisitions (red line). A final interesting result has been obtained subtracting the slow behaviour from the full response: the final signal obtained has the classical vibration pattern of a damped dynamic free-response of a beam under impact load as expected. This result open to possible use of nanofilled plates as cheap sensors for dynamic response acquisition for, as an example, modal analysis and/or impact detection.

#### 4 CONCLUSIONS AND FUTURE WORK

This work present the latest activities carried out by the authors in the field of nanofilled piezo-resistive composites. These materials, in the form of thin plates, can be envisaged both as a possible thermoplastic layer in a composite lay-up providing a full sensitive layer as well as can be shaped as small sensors of arbitrary geometry and secondary bonded on a metallic or composite structure. The presented combination of insulating matrix and conductive nano-fillers presented interesting performances in terms of static as well as dynamic electromechanical behaviour under varying configurations of electrodes, excitation voltages and experimental set-ups. Next step will be oriented to a quantitative characterization of some specific shapes of these materials in order to measure the gauge factor of a set of sensors obtained from a nano-filled plate both in static anc dynamic load conditions. In terms of application this composites will be employed for Structural Health Monitoring applications in combination with machine learning algorithms capable to extrapolate essential information about loads, strains and stresses levels in the structures even considering the evident non linear nature of the electro-mechanical behaviour presents.

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