

Self-Sensing Concrete Nanocomposites for Smart Structures

A. D'Alessandro, F. Ubertini, A. L. Materazzi

Abstract—In the field of civil engineering, Structural Health Monitoring is a topic of growing interest. Effective monitoring instruments permit the control of the working conditions of structures and infrastructures, through the identification of behavioral anomalies due to incipient damages, especially in areas of high environmental hazards as earthquakes. While traditional sensors can be applied only in a limited number of points, providing a partial information for a structural diagnosis, novel transducers may allow a diffuse sensing. Thanks to the new tools and materials provided by nanotechnology, new types of multifunctional sensors are developing in the scientific panorama. In particular, cement-matrix composite materials capable of diagnosing their own state of strain and tension, could be originated by the addition of specific conductive nanofillers. Because of the nature of the material they are made of, these new cementitious nano-modified transducers can be inserted within the concrete elements, transforming the same structures in sets of widespread sensors. This paper is aimed at presenting the results of a research about a new self-sensing nanocomposite and about the implementation of smart sensors for Structural Health Monitoring. The developed nanocomposite has been obtained by inserting multi walled carbon nanotubes within a cementitious matrix. The insertion of such conductive carbon nanofillers provides the base material with piezoresistive characteristics and peculiar sensitivity to mechanical modifications. The self-sensing ability is achieved by correlating the variation of the external stress or strain with the variation of some electrical properties, such as the electrical resistance or conductivity. Through the measurement of such electrical characteristics, the performance and the working conditions of an element or a structure can be monitored. Among conductive carbon nanofillers, carbon nanotubes seem to be particularly promising for the realization of self-sensing cement-matrix materials. Some issues related to the nanofiller dispersion or to the influence of the nano-inclusions amount in the cement matrix need to be carefully investigated: the strain sensitivity of the resulting sensors is influenced by such factors. This work analyzes the dispersion of the carbon nanofillers, the physical properties of the fresh dough, the electrical properties of the hardened composites and the sensing properties of the realized sensors. The experimental campaign focuses specifically on their dynamic characterization and their applicability to the monitoring of full-scale elements. The results of the electromechanical tests with both slow varying and dynamic loads show that the developed nanocomposite sensors can be effectively used for the health monitoring of structures.

Keywords—Carbon nanotubes, self-sensing nanocomposites, smart cement-matrix sensors, structural health monitoring.

I. INTRODUCTION

MULTIFUNCTIONAL materials are an emerging topic in the field of civil engineering. New technologies permit the development of composites with enhanced properties. Thanks to the nanotechnology, new structural tools are now available for engineering applications. Appropriate nano-sized additives can be inserted into cementitious materials as fillers, providing enhanced chemical, physical, mechanical and electrical properties. In particular, carbon nano-inclusions, as carbon nanotubes, appear suitable for electromechanical utilizations. With respect to traditional electromechanical transducers, nano-modified cementitious sensors can be easily embedded into structures, giving rise to the transformation of the surface into infinite sets of potential sensors with enhanced durability and easiness of use. This ability appears similar to the self-sensing property of biological systems. Moreover, conventional monitoring systems are affected by well-known drawbacks that frequently reduce their performance for structural health assessment.

The self-sensing capability of new nano-modified cementitious sensors is obtained through the correlation between the variation of stresses or strains and the variation of particular electrical properties of the composites, such as electrical current or resistance. Due to the strong innovation of that technology, depth studies must necessarily be conducted to optimize the implementation of sensors and fully understand their electrical and mechanical behavior.

The authors started an experimental campaign to investigate issues related to the dispersion of the nanofiller into the cementitious matrix, the composition of the nano-modified materials, the realization of the sensors, the electrical characterization, the analysis of the sensing abilities.

The paper is organized as follows. After a brief background on the most significant researches about the cement materials with nanofillers and their applications, materials, sample preparation and experimental setups are described. Section IV concerns the illustration of the electrical experimentation and the different types of electromechanical tests carried out to investigate self-sensing properties of the sensors. The results of the experimental campaign are discussed and interpreted in Section V. The conclusion examines the future development and the possible applications of the presented innovative sensors.

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II. BACKGROUND

The cementitious materials with conductive particles have been studied from the 90s [1], [2]. Many properties of cementitious materials, such as the electrical ones, are affected by their micro- and nano-behavior. In particular, the addition of micro- and nano-fillers into cement-based matrices allows to significantly modify such properties [3]-[5]. The development of new nanoengineered carbon particles, such as Carbon Nanotubes (CNTs), has determined an important research interest in nano-modified cementitious materials [6]-[9]. The main motivation for such an interest is that the remarkable mechanical and electrical properties of carbon fillers makes them suitable for casting smart cementitious composites with strain-sensitive features [10]-[12]. The materials exhibit piezoresistive behavior resulting in measurable changes of their electrical properties under applied stresses or strains [13]-[16]. A uniform dispersion of the carbon fillers into the matrix results a delicate task investigated in the literature in different ways [17], [18]: Through physical, chemical and mechanical methods. The authors are carrying out a research study about the implementation of new cement-based sensors with carbon nanofillers for structural monitoring [19]-[22] and control [23] applications. Permanent structural health monitoring (SHM) systems are indeed becoming fundamental to improve safety and minimize maintenance costs of civil structures and infrastructures.

III. MATERIALS AND EQUIPMENT

The samples realized for the experimental campaign are cubes with the side of 51 mm (2 inches), made of cement paste, mortar and concrete and doped with different percentages of carbon nanotubes, from 0% to 1.0% with incremental steps of 0.25%, and 1.5% with respect to the weight of the cement. The nanotubes used as nanofillers for the cementitious matrices are Arkema Graphistrength C100 Multi Wall Carbon Nanotubes - MWCNTs (Table I). A second-generation plasticizer based on polycarboxylate ether polymers was added to the mixes in particular amounts in order to obtain a comparable workability.

TABLE I
CHEMICAL, PHYSICAL AND MECHANICAL PROPERTIES OF MWCNTS

Property	Value	Property	Value
Mean agglomerate size	200–500 μm	Carbon content	>90% In weight
Mean number of walls	5–15	Surface area	100-250 m^2/g
Outer mean diameter	10–15 nm	Apparent density	50–150 kg/m^3
Length	0.1–10 μm	Weight loss at 105 $^\circ\text{C}$	<1%
Young Modulus	> 1 TPa	Thermal Conductivity	> 3000 W/(mK)
Tensile strength	About 150 GPa	Electrical Conductivity	up to $10^7 (\Omega\text{m})^{-1}$

The water /cement ratio chosen for all the mixes was 0.45. The cement was type 42.5 pozzolanic. Table II shows the mix designs for cement paste, mortar and concrete with 0.75 % of carbon nanotubes compared with the normal materials. Sand and coarse aggregates were added to obtain mortar and concrete.

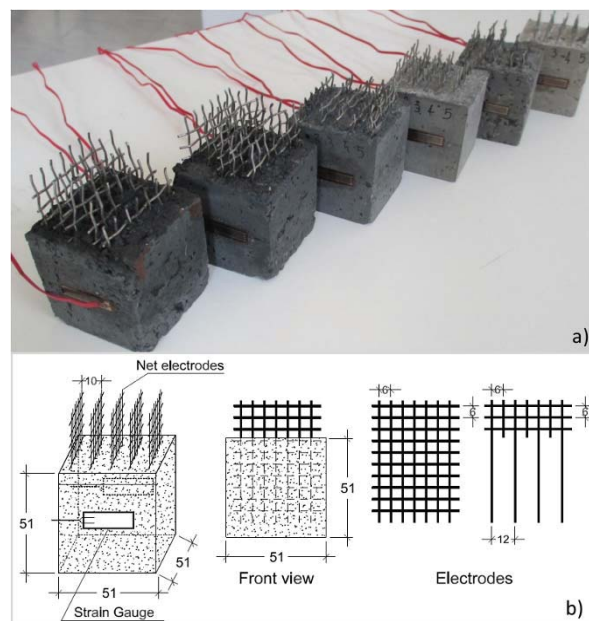


Fig. 1 (a) Picture of concrete samples with 1.5, 1.0, 0.75, 0.5, 0.25 and 0% of MWCNTs; (b) geometry of specimen and electrodes

TABLE II
MIX DESIGN ON PASTE, MORTAR AND CONCRETE WITH 0.75% OF MWCNTs

Mix design	Paste (kg/m^3)		Mortar (kg/m^3)		Concrete (kg/m^3)	
	normal	with CNTs	normal	with CNTs	normal	with CNTs
cement	1277	1241	654	644	524	518
water	574	558	294	290	234	231
CNTs	-	9.3	-	4.8	-	3.9
surfact.	-	9.3	-	4.8	-	3.9
sand	-	-	1308	1289	951	940
gravel	-	-	-	-	638	631
plastic.	-	-	-	6.4	2.62	7.8
w/c ratio	0.45	0.45	0.45	0.45	0.45	0.45

Fig. 2 represents the preparation process of the samples made of paste, mortar and concrete.

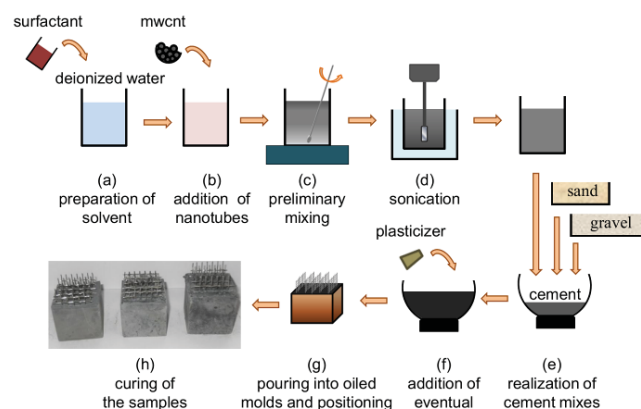


Fig. 2 Preparation process for paste, mortar and concrete samples doped with MWCNTs

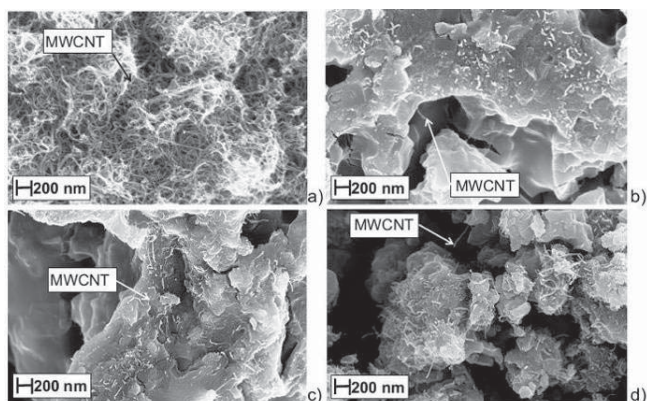


Fig. 3 SEM images of (a) aqueous suspension, (b) paste, (c) mortar and (d) concrete with MWCNTs

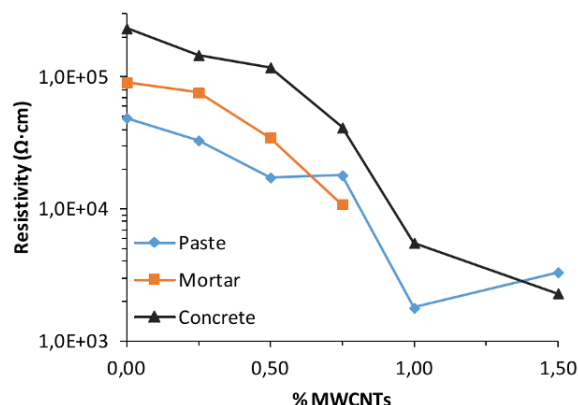


Fig. 5 Electrical resistivity obtained from electrical tests on composites with different amounts of MWCNTs

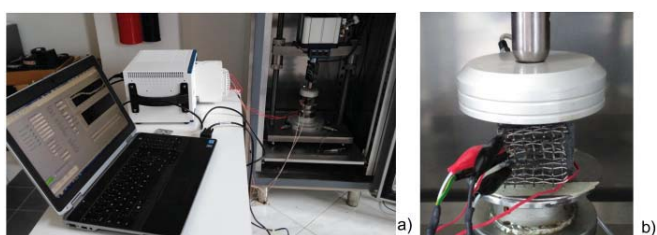


Fig. 4 (a) Experimental setup for cyclic and dynamic tests, (b) detail of the instrumented sample in the testing machine, with strain gauges and four electrical connections

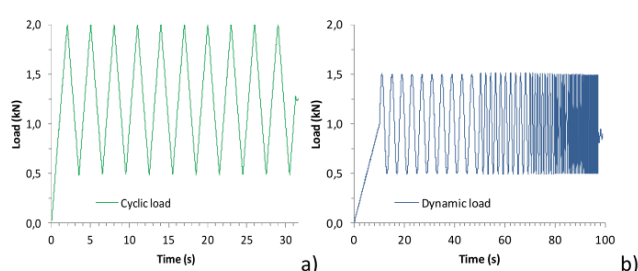


Fig. 6 Electrical resistivity obtained from electrical tests on composites with different amounts of MWCNTs

Fig. 2 represents the preparation process of the samples made of paste, mortar and concrete.

The composites were obtained through two sub sequential steps: first the MWCNTs were dispersed in the aqueous solution (Figs. 2 (a)-(d)), then the nanomodified suspension was added to cement and eventually aggregates to realize the cementitious materials (Figs. 2 (e)-(f)). The dispersion of the MWCNTs was obtained through the use of a physical SLS (Lignosulfonic acid sodium salt) dispersant, mechanical mixing and sonication.

The fresh composites were mixed and poured into oiled molds and electrodes were embedded (Fig. 2 (g)). They were five stainless steel nets for each sample, composed by 0.5 mm diameter wires with a distance of 6 mm, placed at a mutual distance of 10 mm and embedded approximately 85% of the thickness (Fig. 1 (b)).

In the case of concrete specimens, the nets have been reduced in the embedded part to 4 wires having a distance of 12 mm. The two types of electrodes are depicted in Fig. 1. After a couple of days, the specimens were unmolded for curing (Fig. 2 (h)).

A good dispersion of the MWCNTs in the matrix is essential to obtain a homogeneous cement-based material. The effectiveness of the dispersion methods adopted for the realization of the composites was observed through Scanning Electron Microscope (SEM) images of the nanomodified aqueous suspensions and of the hardened pastes, mortars and concretes.

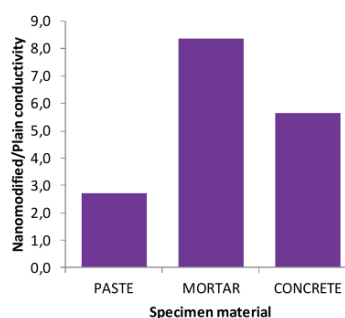


Fig. 7 Ratio between electrical conductivity of cementitious materials with 0.75% MWCNTs and plain ones

Regarding aqueous-suspensions, a little drop was placed on a silicon wafer and the water was evaporated, while for hardened composites, a piece of materials was directly located into the microscope. The micrographs indicate the integrity of the nanotubes after the mixing and their good dispersion (Fig. 3). Both electrical and electromechanical were carried out. Electrical tests were realized by measuring the electrical current $I(t)$ between the two external electrodes placed at 40 mm under a constant voltage difference of 1.5 V. The polarization effect due to the dielectric behavior of the cementitious materials resulted in a variation of the current intensity that stabilized with increasing time. A high precision digital multimeter, model Keithley 6517B was used for the tests.

Electrical resistivity was achieved by (1), with a final time t_f equal to 180 seconds.

$$\rho = \frac{V}{I(t)} \frac{d}{A} \quad (1)$$

Electromechanical tests were carried out with axial loads, both slow varying and dynamic, after 20 minutes of polarization. Their purpose was to investigate the strain-sensing ability of the nano-modified sensors.

The equipment was a model IPC Global UTM14P servo-controlled pneumatic universal testing machine with a maximum load capacity of 14 kN (Fig. 4 (a)).

Two types of load histories were tested: loading-unloading cycles at constant low speed and constant amplitude of 2 kN, and sinusoidal loads at constant amplitude of 0.5 kN and increasing frequency, from 0.25 to 4 Hz.

The signal acquisitions were recorded at a sampling rate of 1 kHz for all tested samples.

The samples were first compressed with a force of 0.5 kN and 1.0 kN for cyclic and dynamic tests, respectively, and then they were subjected to the load sets. Average compressive strain in the sensors was measured with two electric strain gauges placed on opposite faces of each sample, with a nominal resistance of 120 Ω and a gauge factor of 2. Strain sensitivity was carried out through a high precision data acquisition system, NI PXIe1073. Four probe method has been adopted for electromechanical measurements. A source measure unit, model NI PXI4130, provided a stabilized current to two electrodes at a distance of 30 mm, while a NI PXI4071 digital multimeter measured the potential difference V between two internal electrodes at a distance of 10 mm. In this way the contact resistance at the electrodes doesn't affect the electrical tests.

Fig. 6 shows the time histories of the load for cyclic (Fig. 6 (a)) and dynamic tests (Fig. 6 (b)).

IV. EXPERIMENTAL CAMPAIGN

The electrical resistance of the materials with carbon nanotubes decreases with the increase of the filler content, up to the percolation threshold, when the inclusion forms a continuous electrical network. Above that threshold, an increase in fiber content does not significantly influence the conductivity of the materials.

For studying that behavior of the nano-modified cementitious materials, electrical tests were carried out on paste, mortar and concrete samples.

Fig. 5 reports the variations of resistivity of the composites with increasing percentages of MWCNTs content. They indicate a clear percolation threshold between 0.75 and 1% of carbon filler content for all the cementitious materials. Unsatisfactory results for contents from 1.0% of MWCNTs were achieved in mortar samples, probably due to the presence of a significant amount of fine aggregates, with a higher absorbing capacity, which reduces the effectiveness of the physical dispersant. However, the percolation threshold appears evident around this percentage.

The samples to be submitted to the electromechanical tests were chosen after analyzing the results of electrical tests.

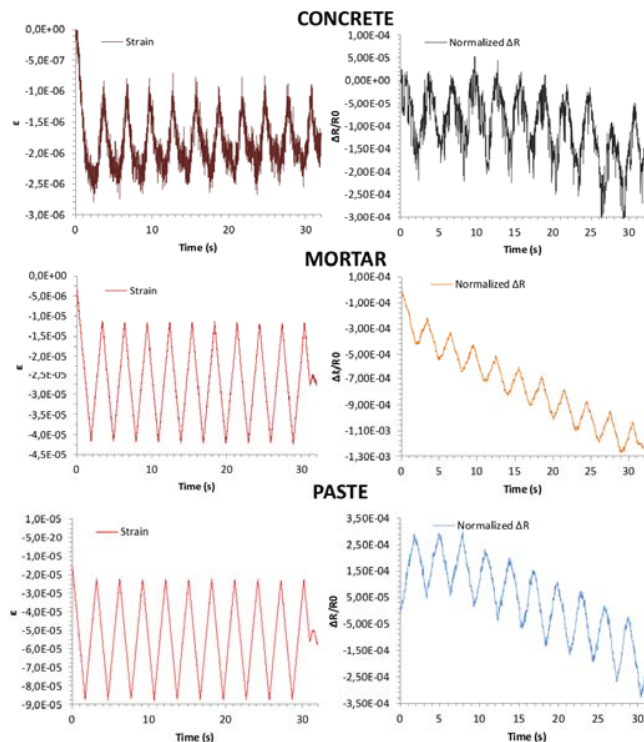


Fig. 8 Results of a strain sensing test for cement paste, mortar and concrete with 0.75% nano-inclusions of MWCNTs, subjected to cyclic loads: strain outputs and normalized variation of electrical resistance

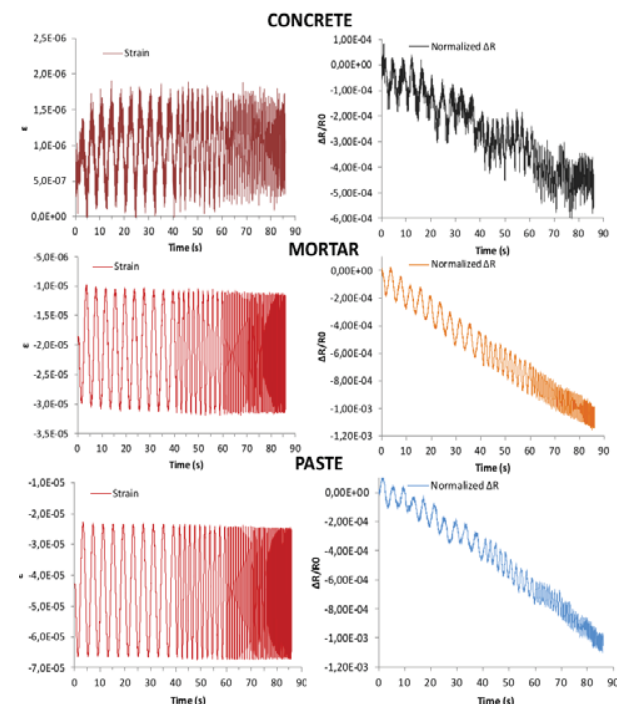


Fig. 9 Results of a strain sensing test for cement paste, mortar and concrete with 0.75% nano-inclusions of MWCNTs, subjected to dynamic loads: strain outputs and normalized variation of electrical resistance

The most advantageous content of nanotubes in a dielectric

matrix, as in cementitious materials, is seen to be nearby the percolation threshold. Research work in the scientific literature confirm this hypothesis. It is explained by the enhanced piezoelectric behavior and sensitivity at the filler concentration closed to the percolation, where applied variations of strains determine a change in the distance between the nanoparticles and alter the tunneling effects between them, highly affecting the electrical conductivity.

The identification of the percolation threshold permitted to select samples with the amount of 0.75% of MWCNTs as nanofillers for the sensing experimentations.

Fig. 7 shows the ratio between the electrical conductivity of the nano-modified and plain cement-based materials, for a MWCNTs content of 0.75%. The relative increase in electrical conductivity grows from paste to mortar or concrete. This is probably due to the influence of aggregates that limit the electrical conductivity of the plain materials. The introduction of conductive fillers in the matrices of mortar or concrete are more effective than in cement paste.

Electrical properties of cement-based transducers with carbon nano-inclusions can be modelled through an equivalent electrical circuit with resistors and capacitors [24], [25]. The relation between the variation of electrical resistance (ΔR) and the axial strain (ϵ) can be expressed as (2):

$$\Delta R / R_0 = -\lambda \epsilon \quad (2)$$

where R_0 is the unstrained initial electrical resistance of the material and λ the gauge factor.

Figs. 8 and 9 report the time histories of the normalized incremental variation of the electrical resistance, $\Delta R/R_0$, and of the strain, ϵ , for cement paste, mortar and concrete with 0.75% of MWCNTs as a function of the two different typologies of applied loads. In particular, Fig. 8 represents the outputs of the cyclic low-varying tests while Fig. 9 shows the results of the dynamic tests at different frequencies of sinusoidal load. The graphs demonstrate that paste, mortar and concrete, sensors with MWCNTs nanoinclusions clearly exhibit strain-sensing capabilities with increasing performance from concrete to paste. The time drifts, noticeable in the electrical resistance signals, were caused by the polarization effect.

The level of noise raises from paste to concrete: It is probably due to the enhancement of the complexity of the materials at the micro-level. All signals maintained a certain degree of linearity with strain, even if the concrete sample exhibits a worst behavior. Figs. 8 and 9 show the time history of the strain and incremental resistance coming from the raw data. Indeed, a residual polarization effect is evident. However, it doesn't represent an effective problem because it can be eliminated with a high pass filtering.

V. RESULTS

The enhanced electrical characteristics of MWCNTs allowed the implementation of innovative conductive reinforced cement-based materials with enhanced properties for structural applications. The increase of electrical

conductivity results from the formation of a three dimensional meshwork of fillers. The greater concentration and homogeneous dispersion of nano-inclusions produces higher conductivity of the matrix, up to the percolation threshold, when the fillers form a continuous electrical conductive network and electrical enhancement is less evident. Nano-modified cementitious materials have both capacitive and piezoresistive behavior. The capacitive part relates to the dielectric nature of the cementitious matrix and is responsible for the polarization effect concurrently with the presence of an applied electric field.

The electrical behavior instead comes from several effects: the intrinsic resistance of nanotubes and of the cement-based matrix, the contacting conduction among the nanotubes, the tunneling and field emission conduction due to nano-size dimensions of the fillers [26]. The self-sensing ability is achieved with the correlation between the variation of the applied stresses or deformations and the variation of electrical resistance or conductivity. However, it is a delicate task to obtain a good dispersion of the nanotubes because they tend to agglomerate, due to their high specific surface and their low solubility within aqueous matrices. In the present research work both physical and mechanical methods have been utilized.

The samples made of paste, mortar and concrete with different percentages of nanotubes content were subjected to electrical and electromechanical tests, in order to investigate their strain-sensing ability. For both low varying and dynamic loads, the nano-modified sensors exhibit a certain self-sensing behavior.

Cement paste and mortar samples showed the best performance, while concrete ones exhibit a higher level of noise. Further investigations about the optimization of the sample preparation process can improve the smart characteristics of cement-based materials with carbon nanofillers.

The results of the experimental campaign demonstrate that new nano-modified cementitious materials with carbon nanotubes appear promising for engineering applications.

VI. CONCLUSION

Nano-modified cement-based sensors represent an innovation in civil engineering. The research works present in literature demonstrates the growing interest of the scientific community about self-sensing cementitious materials with conductive nanofillers.

Future applications are multidisciplinary. They are suitable for monitoring of structures and infrastructures. A distributed sensor system can provide information about the status of the monitored component or structure, identifying anomalies of performance due to incipient damages, such as, for example, before and after a seismic event. They can be useful also to support management and assistance decisions.

Perspective applications are: monitoring of concrete and asphalt pavements, weigh-in-motion sensing via self-sensing cementitious slab, for traffic and crowd management,

monitoring of historical structures, monitoring of energy systems, as wind turbine bases and dams.

Other promising applications are related to the development of smart cities: conductive floors with antistatic capability for data centers in smart grids, thermally conductive cement based materials for de-icing of roads or for geothermal applications, thermal and hygroscopic sensors for local environmental control of structures, concrete with high energetic efficiency through the combination of conductive nanofillers and phase change materials.

ACKNOWLEDGMENT

The authors gratefully acknowledge for their support Prof. J. M. Kenny, Prof. L. Torre, Dr. M. Rallini and the other members of the Group of Materials Science of University of Perugia. This research was partially supported by Regione Umbria, within ESF-funded research-grant Scheme 2007-2013, Axis II "Employability" Objective "e" Axis IV "Human Capital" Objective "I".

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Her research resulted in the publication of several papers in international journals and books, and in contributions in national and international conferences. Three selected papers are:

1. A. D'Alessandro, M. Rallini, F. Ubertini, A.L. Materazzi, J.M. Kenny "Investigations on scalable fabrication procedures for self-sensing carbon nanotube cement-matrix composites for SHM applications" *Cement and Concrete Composites* 65 (2016) 200-213.
2. F. Ubertini, S. Laflamme, A. D'Alessandro. "Smart cement paste with carbon nanotubes" (2016) In: *Innovative Developments of Advanced Multifunctional Nanocomposites in Civil and Structural Engineering*, Edited by K.J. Loh and S. Nagarajaiah, Woodhead Publishing, 97-120
3. A.L. Materazzi, F. Ubertini, A. D'Alessandro, "Carbon nanotube cement-based transducers for dynamic sensing of strain" *Cement and Concrete Composites* 37 (2013) 2-11.