Strain Sensitivity of Carbon Nanotube Cement-based Composites for Structural Health Monitoring

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ABSTRACT

Cement-based smart sensors appear particularly suitable for monitoring applications, due to their self-sensing abilities, their ease of use, and their numerous possible field applications. The addition of conductive carbon nanofillers into a cementitious matrix provides the material with piezoresistive characteristics and enhanced sensitivity to mechanical alterations. The strain-sensing ability is achieved by correlating the variation of external loads or deformations with the variation of specific electrical parameters, such as the electrical resistance. Among conductive nanofillers, carbon nanotubes (CNTs) have shown promise for the fabrication of self-monitoring composites. However, some issues related to the filler dispersion and the mix design of cementitious nanoadded materials need to be further investigated. For instance, a small difference in the added quantity of a specific nanofiller in a cement-matrix composite can substantially change the quality of the dispersion and the strain sensitivity of the resulting material. The present research focuses on the strain sensitivity of concrete, mortar and cement paste sensors fabricated with different amounts of carbon nanotube inclusions. The aim of the work is to investigate the quality of dispersion of the CNTs in the aqueous solutions, the physical properties of the fresh mixtures, the electromechanical properties of the hardened materials, and the sensing properties of the obtained transducers. Results show that cement-based sensors with CNT inclusions, if properly implemented, can be favorably applied to structural health monitoring.

Keywords: Carbon nanotubes, smart sensors, self-sensing materials, structural health monitoring, cement-matrix sensors, strain sensitivity

1. INTRODUCTION

Electrically conductive cement-based materials filled with carbon nanoinclusions are an emerging technology for distributed strain sensing in concrete structures [1-8], whereby the composites exhibit piezoresistive properties resulting in measurable variations of their electrical properties under an applied mechanical deformation [9-12].

Among the various additive particles capable of providing the material with such piezoresistive properties, multiwalled carbon nanotubes (MWCNTs) have excellent electrical properties and provide composites with very high strain sensitivity, and good quality signals under both slowly varying and rapidly varying strain [13-23].

Two major challenges in using MWCNTs are their difficult dispersion in water, originating from the electronic configuration of the tube walls providing high Van der Waals attraction forces among nanotubes, and their relatively high cost compared to other nano- and micro-reinforcements. This paper attempts to address both issues by investigating the use of different surfactants to facilitate nanotube dispersion and by determining an optimal content of nanotubes resulting in paste, mortar and concrete samples with maximum strain sensitivity.

The rest of the paper is organized as follows. Materials preparation is investigated at first and followed by the analysis of the electromechanical properties of the composites. Then, the strain sensing tests are presented and their results are discussed. Finally, the main conclusions are presented.

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2. MATERIALS PREPARATION

2.1 Specimens characteristics

Cement paste, mortar and concrete nanocomposite specimens of dimensions 51x51x51 mm^3 were prepared using different mass contents of MWCNTs. Specimens' geometry are shown in Figure 1 along with the picture of one fabricated specimen. Five stainless steel net electrodes were embedded in the specimens at relative distances of 10 mm. These electrodes were used to measure resistivity and strain sensitivity of the composites and to investigate their homogeneity in space.

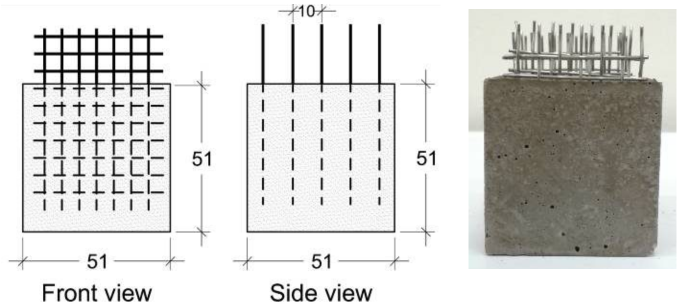

Figure 1.Geometry (left and middle) and sample picture (right) of cement-based nanocomposite specimens

2.2 Fabrication procedure

Nanocomposite specimens were fabricated using the procedure illustrated in Figure 2. First, an equal amount of MWCNTs and surfactant were added to deionized water, proportioned to obtain a final cementitious mix with a water/cement ratio of 0.45. Second, the suspension was mixed via magnetic stirring for 10 minutes followed by sonication for 30 minutes. Third, specimens were casted, modeled, and let cure. Five different types of surfactants were used, summarized in Table 1. Cement paste, mortar and concrete specimens were prepared using different mass contents of MWCNTs with respect to the mass of cement: 0%, 0.25%, 0.50%, 0.75%, 1.0% and 1.5%. The mix designs of the specimens are reported in Table 2.

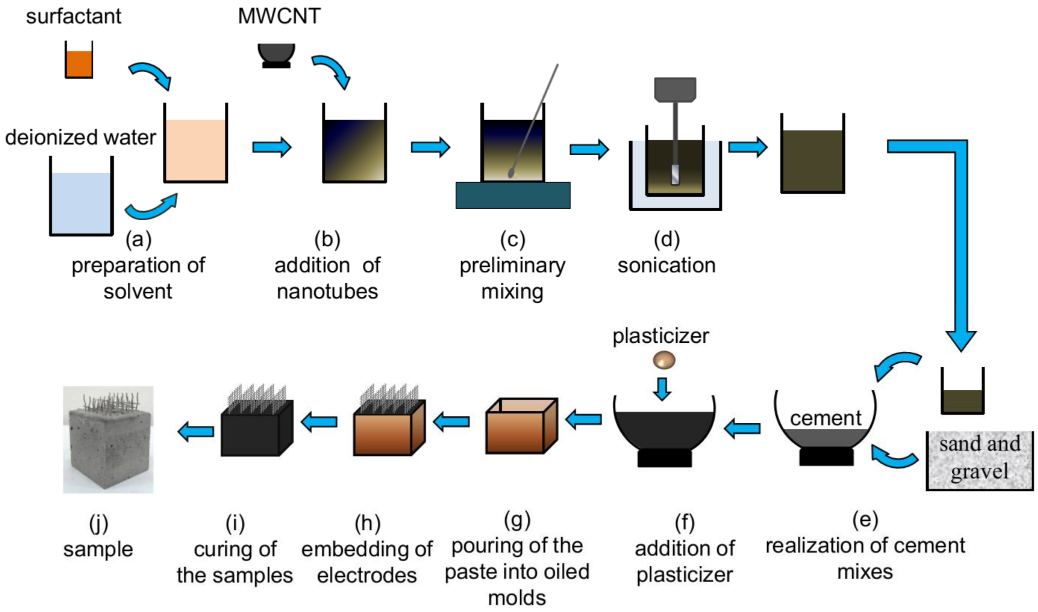

Figure 2. Preparation procedure of cement-based nanocomposite concrete specimens (paste and mortar are prepared without any aggregate and without gravel, respectively)

Table 1. Surfactants used in the experiments

No.	Surfactant	Description
1	ByK 154	Ammonium polyacrylate-based
2	ByK 9076	Alkylammonium salt of a high molecular-weight copolymer
3	NaDDBS	Sodium dodecylbenzenesulfonate
4	SLS	Lignosulfonic acid sodium salt
5	PSS	Polystyrene sulfonates

Table 2. Mix designs of fabricated cement-based composites with MWCNTs (C_0 is the mass of cement in plain cement-based materials, C is the mass of cement in cement-based composites, ΔV_{PA}, ΔV_{MO} and ΔV_{CO} represent the total volume of MWCNTs plus dispersant for composite paste, mortar and concrete, respectively, ν is the ratio between MWCNTs and cement mass).

Components	Paste		Mortar		Concrete	
	Plain (kg/m^3)	**Composite (kg/m^3)**	**Plain (kg/m^3)**	**Composite (kg/m^3)**	**Plain (kg/m^3)**	**Composite (kg/m^3)**
Cement 42.5	$C_0 = 1277$	$C = C_0 \frac{1\text{m}^3}{1\text{m}^3 + \Delta V_{PA}}$	654	$C = C_0 \frac{1\text{m}^3}{1\text{m}^3 + \Delta V_{MO}}$	524	$C = C_0 \frac{1\text{m}^3}{1\text{m}^3 + \Delta V_{CO}}$
Water	574	$0.45C$	294	$0.45C$	234	$0.45C$
MWCNTs	-	νC	-	νC	-	νC
Dispersant	-	νC	-	νC	-	νC
Sand (0-4mm)	-	-	1308	$2C$	951	$1.81C$
Gravel (4-8mm)	-	-	-	-	638	$1.22C$
Plasticizer	-	Var	-	Var	2.62	Var
Water/cement ratio	0.45	0.45	0.45	0.45	0.45	0.45

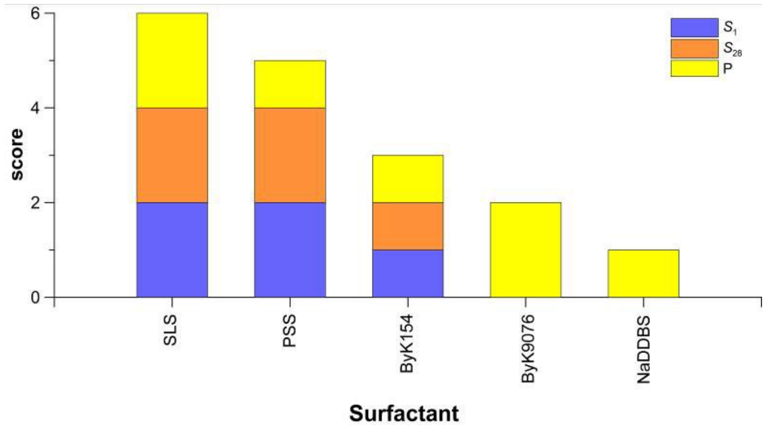

Figure 3. Scores obtained by different specimens in MWCNTs dispersion tests (specimens with zero score are omitted)

MWCNTs were Graphistrength C100, manufactured by Arkema. They are in the form of a black powder with a low apparent density. Their cylindrical structure consists of coils of atomic layers of graphite. Cement was pozzolanic, type 42.5. Polycarboxylate ether polymers were added as plasticizers to the mixes in variable amounts, in order to obtain similar workability for all the mixes. Sand with nominal dimensions ranging from 0 to 4 mm was used as fine aggregate for mortars, while in concrete mixes coarse aggregates with nominal dimension ranging from 4 to 8 mm were also included into the mix.

3. ANALYSIS OF NANOTUBE DISPERSION

To evaluate the quality of dispersion of the MWCNTs, a score J was attributed to each specimen:

$$J = S_1 + S_{28} + P \tag{1}$$

where S_1 is the "initial settling factor", S_{28} is the "final settling factor" and P is the "SEM picture factor". S_1 and S_{28} were assigned a value of 0, 1 or 2 depending upon the assessment of the settling conditions of the suspension in a test tube in the first day and after 28 days from the mixture realization, respectively. A value of $S_i=0$ corresponds to nanotubes fully separated from the water, where $S_i=2$ corresponds to fully dispersed nanotubes, and $S_i=1$ corresponds to an intermediate condition.

The factor P was evaluated by analyzing SEM pictures of the specimens at different magnifications. $P=0$ corresponds to the cases where bundles of MWCNTs were already visible at the lowest magnification factor of 100x. $P=2$ corresponds to SEM images showing bundles of MWCNTs at magnification factors greater than 5000x. A higher value for J signifies a better dispersion of the nanotubes.

Figure 3 shows the scores assigned to the quality of nanotube dispersion in water achieved using the five considered surfactants. Results show that the best quality of dispersion was obtained using the SLS dispersant. Figure 4 shows the corresponding analysis of a test tube after 1 and 28 days and the SEM inspection. Based on these results, specimens with SLS dispersant were fabricated to conduct the following investigations.

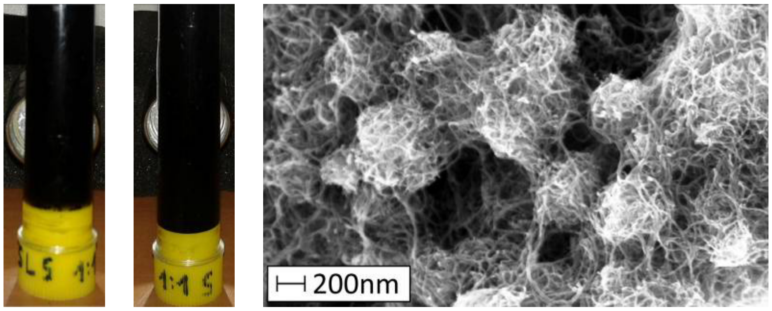

Figure 4. Retroilluminated photography of MWCNTs-water suspensions after 1 (left) and 28 (middle) days, and SEM image of MWCNTs dispersed in water (right)

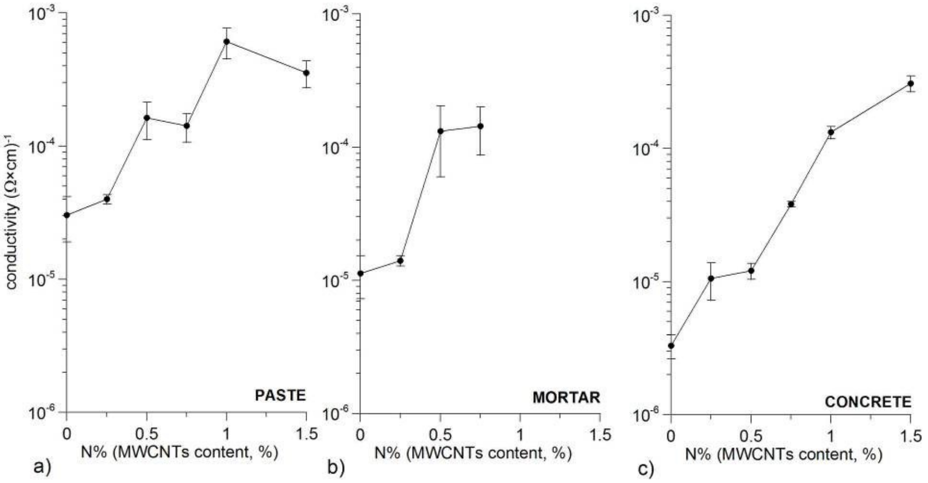

Figure 5. Electrical conductivity obtained from measured impedance of composites with different amounts of MWCNTs.

4. ELECTROMECHANICAL PROPERTIES OF THE COMPOSITES

Tests were conducted to investigate the electrical percolation in cement-based composites. These tests were based on AC current in order to eliminate the polarization effect. In particular, the electrical resistivity of the composites were obtained by measuring the electrical resistance of the specimens through a high precision LCR meter, model HM8018. Measurements were taken using all four pairs of electrodes, spaced 1 cm, for each specimen. This allowed the collection of information on irregularities in signals within each specimen, which is related to the quality of nanotubes' dispersion and to their homogeneity in the volumetric space. Results of the electrical characterization tests are shown in Figure 5. They show a clear percolation threshold between 0.75 and 1% of MWCNTs content for composite concrete specimens. Unsatisfactory results for contents from 1.0% of MWCNTs were obtained in mortar specimens, which indicates that the presence of fine aggregates reduces the effectiveness of the SLS dispersant, conceivably because of excessive liquid phase absorption by aggregates themselves.

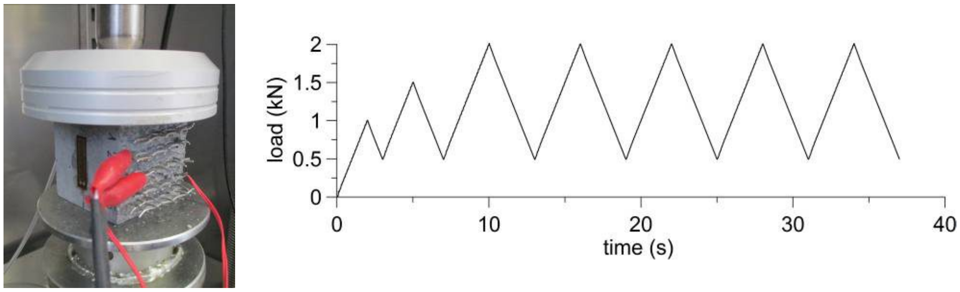

Figure 6. Sensor under uniaxial testing (left) and applied compression load (right)

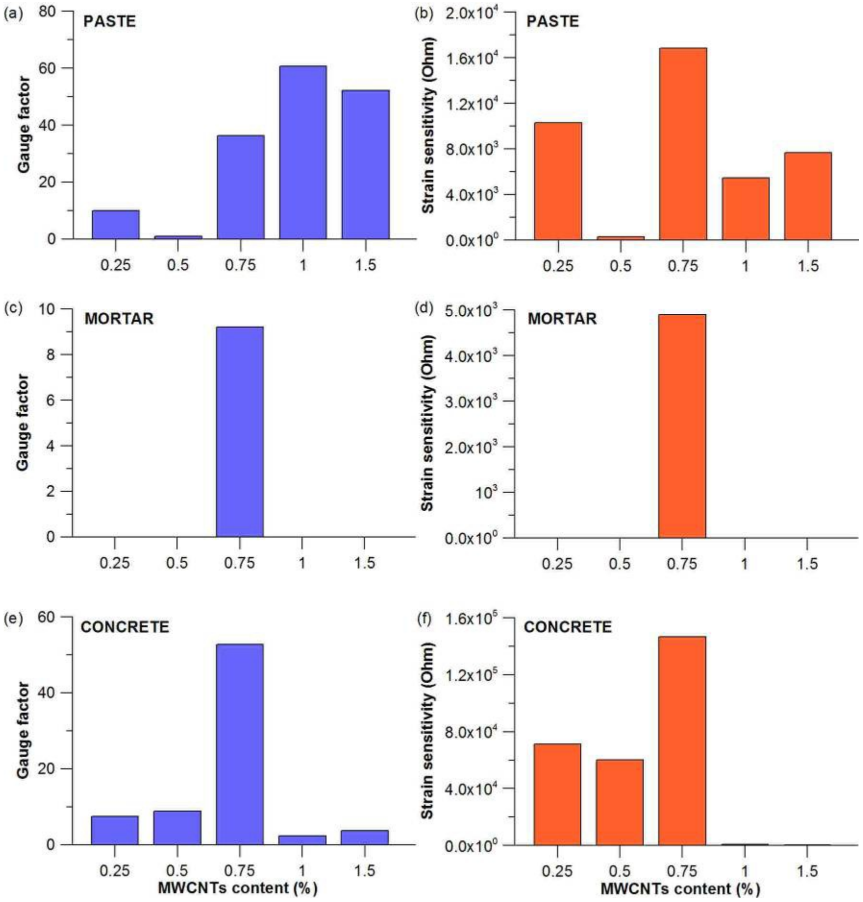

Figure 7. Gauge factor and strain sensitivity of cement-based composites doped with different contents of MWCNTs.

However, the percolation threshold appears evident before this percentage. Percolation is less clearly identifiable in composite paste specimens, probably due to the higher conductivity of cement paste with respect to the other cement materials. Indeed, in mortar and concrete, the presence of aggregates makes these materials less conductive and allows the occurrence of the double percolation phenomenon [24-25].

5. SENSING PROPERTIES OF THE COMPOSITES

In order to test the strain sensing properties of the cementitious composites, specimens were subjected to cyclic axial compression loads in a servo-controlled pneumatic universal testing machine of 14 kN load capacity, model IPC Global UTM14P. Figure 6 shows a specimen under testing and the time history of the applied compression load. The strain sensing ability was investigated by measuring the current passing through the specimens under the application of a constant voltage drop between two active electrodes placed at a distance of 10 mm. The measurement hardware was a high speed digital multimeter, model NI PXI4071, and a source measure unit, model NI PXI4130, provided the stabilized potential difference of 1.5 V. Two 20 mm long electrical strain gauges were also attached at opposite faces of the specimens and their signals were acquired using a data acquisition card, model NI PXIe-4330. Strain-induced incremental variation in electrical resistance of the composites, $\Delta R(t)$, was obtained by dividing the applied voltage, V, by the incremental variation in measured current intensity, $\Delta I(t)$. Under the small strain assumption, the relationship between ΔR and axial strain, ε (positive in compression), was modeled in analogy with electrical strain gauges as

$$\frac{\Delta R}{R_0} = -\lambda \varepsilon \tag{2}$$

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

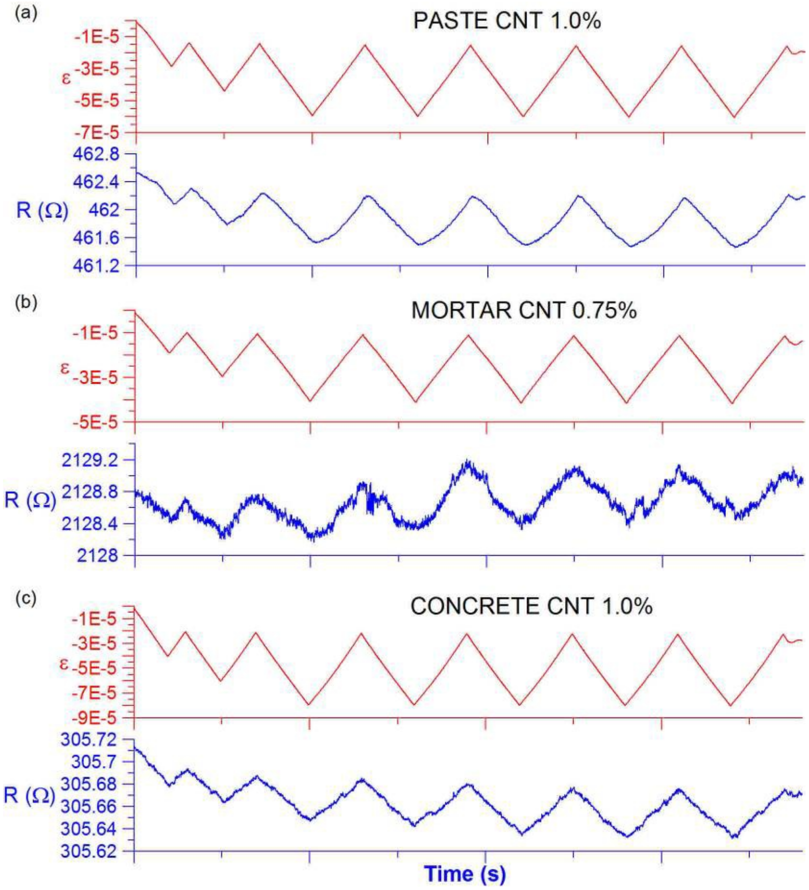

Figure 8. Time histories of applied compression strain and corresponding electrical resistance outputted by cement-based nanocomposite specimens with maximum strain sensitivity.

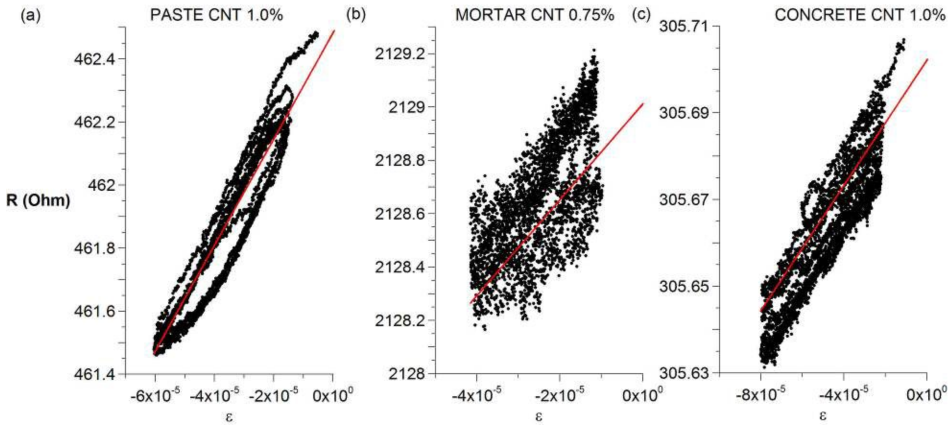

Figure 9. Measured electrical resistance vs. applied strain for cement-based sensors.

Figure 7 shows values of the gauge factor and of the dimensional strain sensitivity ($\Delta R/\varepsilon$) obtained from the strain sensing tests. Results show the existence of an optimal content of nanotubes around 0.75-1.0% mass content with respect to the mass of cement. In all cases (paste, mortar and concrete specimens), the optimal content of nanotubes providing the maximum gauge factor is slightly different from the one providing the highest dimensional strain sensitivity. This is attributed to the value of the unstrained electrical resistivity (Eq. (2)).

It should be noted that the optimal content of nanotubes is seen to be very close to the percolation threshold. This is explained by the enhanced piezoelectric behavior at percolation, where a change in the composite's geometry produces a significant change in the proximity of the nanoparticles, resulting in more sensitive electrical properties with respect to deformations. Figure 8 shows the time histories of applied strain and corresponding electrical resistance of paste, mortar and concrete specimens containing optimal amounts of MWCNTs with respect to sensitivity. Figure 9 plots these results in the ΔR vs. ε plane. Results from Figures 8 and 9 highlight better signal quality and linearity in the case of paste when compared to mortar and concrete.

6. CONCLUSIONS

The paper has investigated the strain sensitivity of cement pastes, mortars and concretes filled with different amounts of MWCNTs. First, the quality of carbon nanotube dispersion in water following the utilization of surfactants and sonication was investigated. The results have allowed to identify the best surfactant for dispersing MWCNTs, evaluated by studying scanning electron microscopy pictures and water nanotube suspensions after 28 days. A clear percolation around a 1% mass content of nanotubes with respect to the mass of cement has been found in the case of concrete specimens, while the same percolation was less evident for pastes and mortars. The results of the strain sensing tests highlighted the existence of an optimal content of nanoinclusions, close to the percolation threshold, resulting in an optimal value for the gauge factors. Cementitious specimens containing these optimal levels of MWCNTs were tested for dynamic strain sensing capabilities. Results showed that all types of specimens could be used to sense dynamic strain, whereas the paste specimens showed enhanced signal-to-noise ratio compared with mortar and concrete composites. This study showed the promise of a simple dispersion technique to produce cementitious sensors using MWCNTs. This has the potential to lead to large volume productions to enable full-scale applications.

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