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Piezoresistive Properties of Cementitious Composites Reinforced by PVA Fibres

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Abstract. The use of fibres to enhance the ductility of cementitious composites has been extensively studied for the past few years. The addition of polymeric or metalic fibres with random orientation to the composite or even natural long and aligned fibres demonstrated a very successful reinforcement capable to reach a high mechanical performance. Other property that has been studied is the use of those composites to work as strain sensors. To develop piezoresistive properties on cementitious composite, the addition of conductive materials is necessary. This research evaluated the incorporation of different volumes of multi-wall carbon nanotubes on the piezoresistive properties of strain hardening cementitious composites (SHCC) reinforced by PVA fibres. Through impedance measurements the opening of the cracks under tensile loading was studied. The characterization of this material can help on the understanding of self-sensing properties, adding value to the SHCC used by the repair industry and will contribute to the continuous infrastructure monitoring.

Keywords: Strain hardening cementitious composites · Mechanical properties · Piezoresistive properties · Smart structures · Self-sensing

1 Introduction

Infrastructure maintenance is a worldwide problem, due to the high complexity of the structures and the cost involved in a durability surveillance. The service life of a large number of existing concrete structures are soon to reach 50 or more years in many countries, bringing problems and high costs for refurbishment to extend their service life. An example can be drafted by the conditions of roads in United States of America and in Brazil, where 32% (American Society of Civil Engineers 2013) in 2013 and 57.3% (INEA-RJ 2014) in 2015 respectively were considered in unsatisfactory conditions. Maintenance surveillance of infrastructure spread in large areas will be always challenging, and resources demanding. Therefore, techniques which improve the efficiency of infrastructure monitoring must be developed.

Most of the infrastructure will need repair interventions during their service life. In the past few years, several projects have been carried out on the development and characterization of different repair materials. Among these, the performance of high ductility cementitious composites have drawn the attention of different sectors,

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bringing conclusions about the importance of the capability of absorbance of high strains of this kind of material (Li et al. 2000).

Emerging as one of the most successful ductile materials in civil engineering, strain hardening cementitious composites (SHCC) are an example of the important role of ductility on performance of such composites. The match between the tailored brittle matrix and ductility of the Polyvinyl Alcohol fibres (PVA) enables these composites to develop multiple cracks when loaded under tension. In the very beginning, the matrix of this class of composite, also called Engineered Cementitious Composites (ECC) in this case, was composed by ordinary Portland cement, silica fume and sand (Li 2003). Afterwards, different compositions for this matrix were developed, like demonstrated by (Zhou et al. 2010), where the incorporation of blast furnace slag and limestone powder was investigated on the cementitious matrix. (Li et al. 1995) demonstrated the importance of low toughness of the brittle matrix of cementitious composites, in order to obtain a pseudo-strain hardening behaviour.

Moreover, several other projects evaluated the performance of different fibres to reinforce cementitious matrix. Carbon (Li and Obla 1994), polyethylene (PE) (Homma et al. 2009), polypropylene (PP) (Zhang et al. 2015), steel (Park et al. 2012) and natural fibres (de Andrade Silva et al. 2009) are usually found on literature as reinforcement for high ductility cementitious composites. Recently the search for hybrid reinforcement was pursued by (Pakravan et al. 2016) and in a multi-scale fibre reinforcement by (Kwon et al. 2015), where the cementitious composites were reinforced by steel fibres and wollastonite micro-fibres. Besides the high mechanical properties, the SHCC's demonstrated a good performance when subjected to accelerated ageing tests and capability to develop autogenous self-healing, confirming extraordinary performance of this composite (Ahmed and Mihashi 2007). (Ranade et al. 2014) investigated the self-sensing properties of ECC loaded in uniaxial tension. In their research, the influence of number of cracks and their average width was correlated with the increase of the electrical resistivity.

The development of smart cementitious composites has been under the spotlight, as well. The addition of electrical conductive fillers in cementitious materials decreases the overall resistivity of the composite. There are many works describing the influence of addition of materials like, nano ZrO_2, Al_2O_3, Fe_2O_3 and TiO_2, ensuring mechanical performance improvement and in some cases lowering the electrical resistivity. However, the focus has been on the carbon nanotubes and nanofibers (Han et al. 2015a, b).

The electrical conductivity of cementitious composites with conductive fillers is represented usually by those three different forms: contacting conduction, tunnelling conduction (and/or field emission conduction), and ionic conduction. The contacting conduction is characterized by the direct contact of conductive particles. Tunnelling conduction happens only when the conductive particles are separated by a maximum distance of 10 nm, and field emission conduction is induced when a strong electrical field is generated. The sharp tips found in nanomaterials, like carbon nanotubes (CNT), are important to create this strong field. Finally, ionic conduction plays a very important role in the case of cementitious matrixes, as well. The water present in the voids might dissolve the ions from the matrix, resulting in ionic conduction through interconnected capillary pores (Han et al. 2015a, b).

The use of carbon nanofibers on cementitious composites has been successfully employed on the development of smart composites for crack detection (Hoheneder et al. 2015). The development of a SHCC which would be able to contribute with the infrastructure health monitoring is investigated on this research program. An experimental routine is proposed to evaluate the performance of self-sensing SHCC.

2 Experimental

The SHCC developed by (Zhou et al. 2010) was used as reference. The mix design of this composite employs ordinary Portland Cement (OPC) CEM-I 42.5 N and blast furnace slag (BFS) as binders, limestone powder (LP) as filler and 2 vol.% of PVA fibres as reinforcement. To investigate the influence of CNT on the piezoresistive and mechanical performance of this composite two levels of incorporation 0.05 and 0.1 wt.% of binder weight were tested. The CNT used were delivered dispersed in aqueous solution with surfactant, with concentration of 3 wt.% by Nanocyl S.A. The properties of the dispersed CNT can be found in Table 1 and the mix design in Table 2.

Table 1. CNT properties.

Average diameter [nm]	Average Length [μm]	Carbon purity [%]	Surface area [m^2/g]	Volume resistivity [Ω.cm]
9.5	1.5	90	250–300	10^{-4}

Table 2. Mix design of the composite (kg/m^3).

	M6	M6 - 2CNT	M6 - 4CNT
OPC	237	237	237
BFS	553	553	553
LP	790	790	790
Water	411	398.2	385.4
PVA	26	26	26
CNT	0	13.2	26.4
Superplasticizer	1.4	1.4	1.4

500 ml of the mixture were mixed in a Hobart mixer according to the following procedure: all the dry components were mixed at low speed for 4 min, then the liquids (water + superplasticizer + CNT solution) were added to the dry components and mixed for 1 min at low speed, followed by 4 min at high speed. The total volume of water, superplasticizer and CNT solution were sonicated for 25 min in advance to enhance the nanoparticle dispersion.

The mixture was casted on dog-bone shape cylinders (see Fig. 1a with dimensions in millimetres). After casting, these moulds were covered with plastic foil during the first 24 h of curing. The samples were demoulded after 24 h of curing and the following 27 days were in the curing room with a relative humidity higher than 95% and 20 °C. Such small specimens were adopted on this research due to the use of CNT and the investigation of the electrical properties of the composite during the mechanical load. Regarding the necessity of employment of small specimens, it is believed that the internal diameter of 16 mm will be enough to promote reasonable dispersion of PVA fibres.

Tensile test was performed using two LVDT's to control the average crack opening. The first 30 µm of crack opening were performed at 0.02 µm/s. From 30 µm to 100 µm the speed was increased to 0.1 µm/s then, from 100 µm to 600 µm the speed was 0.5 µm/s and the test was finished at 800 µm at 1 µm/s. Plexmon 7742 F two component glue was used to attach the specimen to the supporting screws mounted to the Instron machine (see Fig. 1b). Young's modulus of the elastic phase and the tensile strength were calculated from the mechanical test results.

To measure the electrical impedance during the tensile test a function generator was employed delivering a voltage signal of 15 V peak-to-peak amplitude using sine wave with frequency of 500 Hz. The specimen impedance was measured in series with a known standard resistor of 100 kΩ (see Fig. 1d). To separate the resistance and capacitance measurement of the specimen, the phase angle between the current and voltage wave was utilized. Therefore, a real x imaginary plane with the impedance phasor can be used to calculate the real resistance and capacitance during loading.

A two probe electrical measurement was performed. The electrodes were glued on the surface of the specimen on the necking from the external diameter towards the internal diameter, using a conductive paint and a copper wire. In Fig. 1b the sample is prepared to be tested. The specimens were prepared on the 28th day and left under the lab conditions for one day, before the tensile test.

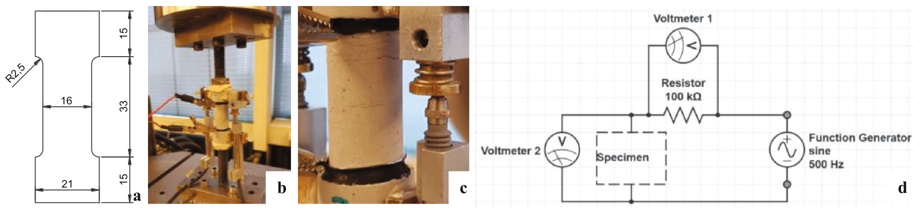

Fig. 1. Cylindrical dog bone shape (a) moulds, (b) prepared for tensile test, (c) showing multiple cracking and (d) scheme of the electric circuit.

The measurement of the electrical impedance, resistance and capacitance were plotted as function of the average width of the crack. Moreover, the curve was divided in four segments to calculate the gauge factors according to Eq. 1, where ΔZ is the impedance changes, Z_0 is the impedance value before the test start and ε is the applied strain during the tensile test. This relation gives a quantitative method for ranking the sensitivity of the composite. The gauge factor from impedance changes is named GF, from resistance RF and capacitance CF. The number after indicates the maximum average crack opening from the analysed interval.

$$GF = \frac{\Delta Z/Z_0}{\varepsilon} \quad (1)$$

The segments correspond to the average crack opening from 0 μm to 6 μm, 6 μm to 20 μm, 20 μm to 100 μm and finally from 100 μm to 800 μm. The analysis of those factors will be helpful to evaluate the influence of different concentrations of CNT employed. To calculate the gauge factor, linear regression from the plot of the values of relative change on the electric properties (Impedance, Resistance and Capacitance) against the strain was adopted.

3 Results and Discussion

Table 3 shows a summary of the mechanical and initial properties of the composite, where E, Z_0, R_0 and C_0 are the modulus of elasticity, initial impedance, resistance and capacitance, respectively. The addition of CNT on the SHCC did not change significantly the modulus of elasticity of the composite. However, a decrease on the tensile strength and ductility was observed. Nevertheless, strain hardening behaviour and multiple cracking was observed for all cases. An example is shown in Fig. 1c. The decrease of the mechanical properties was probably not caused by the CNT, but by the surfactant. This material is employed on the CNT solution to help on the dispersion of the nanoparticles. However, during the composite mixing process foam might be generated, introducing air pockets in the matrix and on the interface with the fibres.

Table 3. Average mechanical and initial electrical properties.

	M6	M6 - 2CNT	M6 - 4CNT
E [GPa]	11.2 ± 0.5	13.7 ± 4.1	12.3 ± 0.7
Maximum tensile strength [MPa]	3.4 ± 0.3	3.2 ± 0.4	2.9 ± 0.2
Z_0 [kΩ]	247.6 ± 44.8	252.8 ± 57.0	180.2 ± 52.1
R_0 [kΩ]	258.8 ± 49.4	259.8 ± 62.7	185.7 ± 55.9
C_0 [ρF]	371.8 ± 29.0	268.0 ± 13.0	416.8 ± 43.6

Measured changes of the modulus of impedance were very similar to those reported by (Ranade et al. 2014). The development of cracks lead to an increase of the total resistance of the composite as can be observed in Fig. 2. The employment of CNT enabled higher gauge factors calculated from the impedance measurement only for crack width up to 100 μm.

Moreover, observing the evolution of the resistance and the capacitance during the tensile test, two different trends can be pointed out. The electrical resistance of the composite increases while the crack width increases and the capacitance goes backwards the opposite direction. By observing the Figs. 3 and 4, it is possible to note that

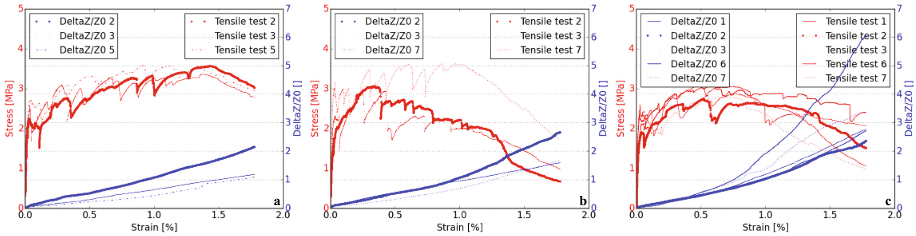

Fig. 2. Fractional change on the electrical impedance during tensile test for: (a) M6, (b) M6-2CNT and (c) M6-4CNT.

the electrical resistance continuously increases while the damage propagates in the composite. On the other hand, the electrical capacitance fractional change tends to find a constant value for wide cracks. Therefore, the electrical capacitance changes are more valuable sensing mechanism for smaller damages then for large crack control.

During the tensile load the CNTs dispersed in the microstructure of the cementitious matrix tend to separate from each other. (Sanli et al. 2016) proposed an electrical model where the matrix between each CNT could be represented by an analogue electric circuit with a resistor in parallel to a capacitor. In this model, when the distance between the CNTs increases, the total impedance of the composite should increase, due

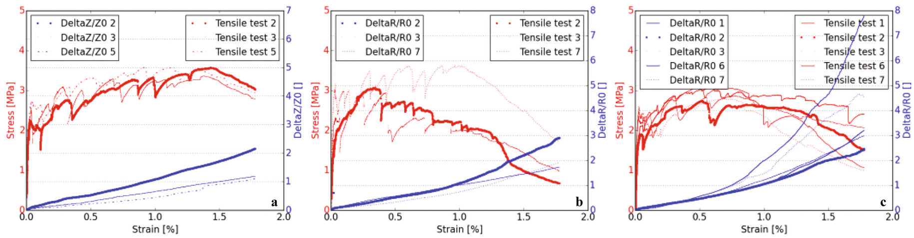

Fig. 3. Fractional change on the electrical resistance during tensile test for: (a) M6, (b) M6-2CNT and (c) M6-4CNT.

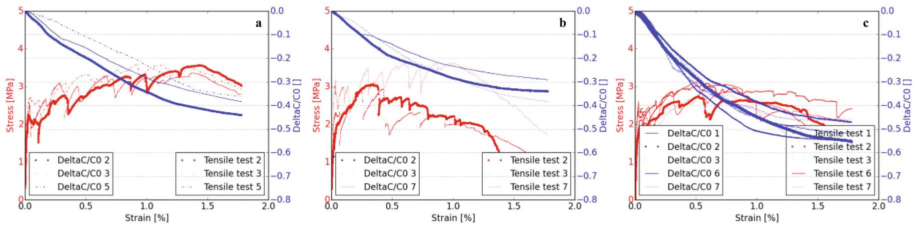

Fig. 4. Fractional change on the electrical capacitance during tensile test for: (a) M6, (b) M6-2CNT and (c) M6-4CNT.

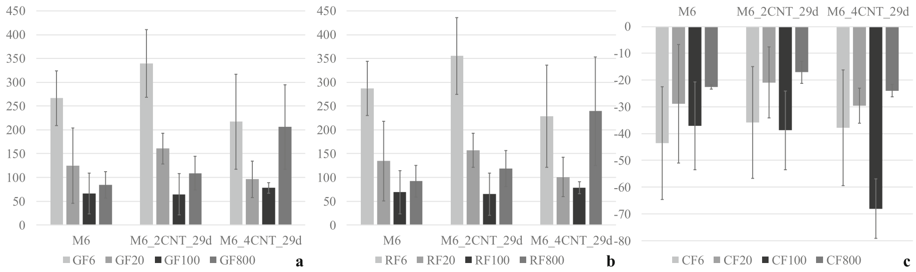

Fig. 5. Average gauge factor calculated from: (a) Impedance modulus, (b) resistance and (c) capacitance.

to higher values of resistance and lower values of capacitance. The result found on this research match with the experimental results and model proposed by (Sanli et al. 2016).

To evaluate the efficiency of CNT employment on cementitious matrix as a piezoresistive sensor the gauge factor reported on Fig. 5 must be taken in account. The electrical resistance of all mix design evaluated on this study increased during the tensile test, demonstrating the capability of the SHCC to be employed as a damage sensor. The same result was found by (Ranade et al. 2014). However, only samples doped with CNT could bring slightly higher GF6 and RF6 for M6-2CNT and considerably higher CF100 and CF800 for M6-4CNT. This might mean that ordinary SHCC are good sensors for detection of small cracks, but CNT doped SHCC sensors could track the development of cracks.

4 Conclusions

The results found in this experimental work lead to the conclusion that the SHCC could be employed as a damage sensor, due to the increase of the modulus of the impedance. However, the use of piezoresistive admixtures as CNT can contribute to the measurement of the crack opening development.

The employment of AC, instead of DC on investigations performed on this field must be taken in account. The results of this paper have clearly shown the damage dependence of the resistance and capacitance. The fully understanding of changes on the total impedance values can clarify the entire mechanism behind it. The results have demonstrated that the changes on capacitance can also be used on measurement of smart cementitious materials.

Further research is needed on the piezoresistive mechanism of fibre reinforced cementitious composites. Older samples must be studied to understand the feasibility of this technique with the development of the hydration, temperature and moisture content. Recently, (Song and Choi 2017) demonstrated that moisture on mortar specimens with CNT lead the composite to lower sensitivity to strain variations.

Moreover, the influence of ageing mechanism like carbonation, freeze and thaw or chloride attack must be comprehended to deliver durable and long term reliable sensors.

Nevertheless, in general the development of a self-sensing SHCC would be very beneficial due to the high importance of those composites for the repair industry, for structures built on seismic region and for a better control of the quality of the infrastructure during their service life.

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