

Variables affecting strain sensing function in cementitious composites with carbon fibers

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Abstract. In this work, cement paste samples with 1% (by cement mass) of a conductive carbon fiber admixture have been studied under uniaxial compression. Three different arrangements were used to measure the resistivity of the samples. According to the results obtained, the resistance should be measured using the four wire method in order to obtain good sensitivity and repeatability. The effect of the load value and the load rate on the fractional change of the volume resistivity has been determined. It has been observed that the gage factor (fractional change in resistance respect to strain) increases when the maximum load is increased, and the loading rate does not affect significantly this parameter. The effect of the sample ambient humidity on the material piezoresistivity has also been studied, showing that the response of the composite is highly affected by this parameter.

Keywords: carbon fiber; cement; piezoresistivity; sensor; strain; stress.

1. Introduction

The need to build structures with non-structural special features while keeping good structural properties is a demand that is gaining momentum. The capability of some structural material to detect its own deformation due to some external loading (without any embedded or attached sensor, which have as disadvantages their high cost, material disturbing, poor durability and limited sensing volume) is indeed one of the most interesting properties of the so called *smart structures*. Some special applications that can be planned for these new materials could be the control of structural vibrations, heavy traffic monitoring, room occupancy detection and safety in buildings, among others (Chen and Chung 1996, Chung 2002).

Self-sensing cement based materials are composite materials with a cement paste matrix and a strong piezoresistive behavior due to the presence of electrically conductive particles or fibers. One of the admixtures that have been more profusely studied is carbon fiber (Garcés *et al.* 2005, 2007, Alcaide *et al.* 2007). The presence of little amounts of carbon fibers in the cement matrix can offer particular properties to the final composite material. The incorporation of carbon fibers produces a new material with a piezoresistive behavior (Chung 2002).

Piezoresistivity refers to the change of volume electrical resistivity when some stress, either tension or compression, is acting inside the structural material producing a measurable strain. This piezoresistive behavior is quantified by the fractional change (in percentage terms) of volume resistance produced by the unity of strain, also known as gage factor (Wen and Chung 2001, Chen

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and Chung 1993, Reza *et al.* 2003, Yao *et al.* 2003).

Previous researchers have reported that in cement pastes with conductive admixtures there is a relationship between the strain produced by some applied stress and the monitored electrical resistance of the material (Chen and Chung 1996, Chung 2002, Wen and Chung 2001, Chung 2001).

It has been demonstrated that when an elastic strain is produced in the material, there exists a response in the electrical resistance that depends on the sign of stress that has been applied to the material. When a specimen of cement paste with a level of carbon fiber below 1% is subjected to a tensile stress, the electrical resistance (measured in the direction of the applied load) increases proportionally to the strain of the specimen as long as the elastic limit is not surpassed (Wen and Chung 1999, Wang and Chung 1998). On the other hand, if a compressive stress is applied, the electrical resistance of the specimen (measured in the direction of the load) decreases proportionally to the strain (Wen and Chung 2001, Reza *et al.* 2003, Wu *et al.* 2005). Both responses, under compressive and tensile stress, are reversible, that is, when the load is removed the electrical resistance of the material recovers its initial value. This aspect is very interesting in order to monitor in situ and continuously the load state of a real structure, which enables the service control and reduces the risk of overloading.

At first, one could think that the dimensional changes in the specimen when it is loaded could be responsible for the observed changes in the electrical resistance. Indeed, there exists some influence of these dimensional variations, but the changes in the electrical resistance are much higher and can not be explained by the geometrical modifications that are produced in the loaded material. In fact, the change in the electrical resistance of the piezoresistive material, when it is subjected to a stress, is due to the modification occurred in the resistivity as an intrinsic property of the material. The monitoring of resistivity allows the observation of this behavior.

The basis of the piezoresistive behavior that has been observed in these composites comes from the variations of the contact resistance between the conductive carbon fibers and the cement matrix (Wen and Chung 2006). When the piezoresistive material is subjected to a compressive stress, this contact resistance decreases, and it produces a decrease in the overall electrical resistance in the direction of the stress. When a tensile stress is applied the phenomena produced is of opposite sign.

If the stress that it is applied to this type of materials surpasses the elastic regime, an irreversible response is observed in the change of the electrical resistance. In this case, the piezoresistive material does not recover completely its initial electrical resistance because at this high stress level two irreversible effects have occurred: the failure of the fiber anchorage to the cement matrix and the breaking of some carbon fibers themselves (Wen and Chung 2006).

The piezoresistive behavior is not only observed in cement matrices incorporating carbon fibers. Other studies have been performed with other conductive fibers, such as steel fibers. Chung *et al.* (Wen and Chung 2003, 2005, Chung 2001) concluded that carbon-fiber (15 μm diameter) cement was a better piezoresistive strain sensor than stainless steel-fiber (8 μm diameter) cement at a similar fiber volume fraction, due to a higher signal-to-noise ratio and better reversibility upon unloading, albeit having a lower gage factor (particularly under tension). The difference in performance of carbon- and steel-fiber cement was attributed to a difference in piezoresistivity mechanism.

One of the most interesting parameters of the piezoresistivity phenomena is the maximum and minimum amount of conductive material that is needed to observe piezoresistivity in cement matrices. The percolation threshold is defined as the amount of fibers that is necessary to form a continuous conductive path through the material, that is, a direct contact between the fibers without

discontinuities. The percolation threshold is usually expressed as volume or mass percentage of the cement in the mix. When the fiber percolation threshold is surpassed, the electrical resistance of the composite decreases sharply, and the resistivity of the material can be as low as several ohm·cm. The percolation threshold mainly depends on the length of the fibers considered, and it is usually in the range from 0.5 to 1 vol% (Chung 2002, Wen and Chung 2005). When the length of the fiber is increased, the percolation threshold decreases. It is not necessary to surpass the percolation threshold to observe piezoresistivity in the composite since it is not necessary for the composite to be conductive (Chung 2002, Wen and Chung 2005).

2. Research significance

This work deals with the influence of some testing procedures and parameters when measuring piezoresistivity on cement based specimens with carbon material admixtures. The influence of the measuring method, how it may affect the results and which is the most appropriate is first considered. Other studied aspect is the repeatability of results in order to have dependable values. Also the influence of ambient conditions (relative humidity) of the specimens was investigated.

3. Experimental

3.1 Materials and specimen preparation

Prismatic specimens of 40 × 40 × 160 mm size were fabricated with Portland cement paste and carbon fiber (CF) Hextow AS4 (Table 1) from Hexcel Corporation. The basic components were:

- Cement type CEM II 32.5.
- Water cement ratio $w/c = 0.5$.
- CF length 10 mm approximately.
- CF mass added to the mix: 1% of the cement mass.
- Methylcellulose (for enhancing the fiber dispersion (Chung 2005)) mass added to the mix: 0.4% of cement mass.

Specimens were cured submerged in water for 28 days before starting the tests. After that curing period, for ambient humidity experiments the specimens were stored, before the tests, in selected ambients of different humidity values: 0%, 60%, 80% and 100%, until constant weight was achieved (approximately 3-4 weeks), in order to ensure that the humidity of the specimens was in

Table 1 Main properties of the carbon fiber used

Diameter	7.1 μm
Precursor	PAN
Carbon content	94%
Tensile strength	4.48 GPa
Tensile modulus	231 GPa
Resistivity	$1.52 \cdot 10^{-3}$ ohm·cm
Density	1.79 g/cm ³

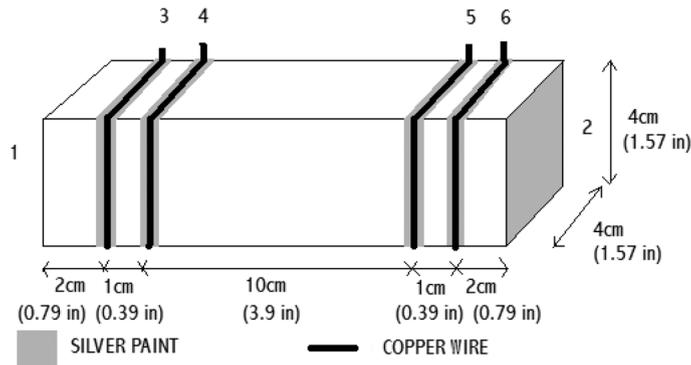


Fig. 1 Specimen electrical configuration. Electrical connection points are identified by numbers

equilibrium with the imposed external humidity. Three specimens were tested for each relative humidity condition.

After the specimen preparation the electrical components were attached. Bands of silver paint were painted around the specimens and then a copper wire was tightly wrapped on each band (see Fig. 1). The end faces of the specimens were also covered with silver paint.

3.2 Testing instruments

Loading of specimens was performed using an electromechanical press model EMI/100/FR, supplied by Microtest SA. Electrical resistance was measured using a digital Keithley 2002 multimeter supplied by National Instruments Inc.

3.3 Description of tests

Tests consisted of monitoring the specimen volume resistance in the longitudinal direction when a cyclic compression load was also applied in the longitudinal direction. The maximum stress value in each cycle was 5, 10, 20 and 25% of the compressive strength (R_c) of the cement paste (that is, 3.12, 6.25, 12.50 and 15.62 MPa, respectively). The loading and unloading rate was 50, 100, 200, 300 and 400 N/s.

The maximum stress value was selected so that the material will behave in the elastic regime; this is about 25% of the compressive strength. Each test consisted on three cycles, the second of which was used to determine the *gage factor*. This parameter is defined as the percentage of volume resistance change of the specimen per unit of applied strain (Eq. (1)) (Wen and Chung 2003).

$$GF = \frac{\Delta R}{R \cdot \varepsilon} \cdot 100 \quad (1)$$

where GF is gage factor, ΔR is the change in resistance, R is the initial resistance, and ε is the applied strain ($\Delta L/L$, being L the length of the specimen).

According to previous references the strain values are very small and for that reason electrical resistance instead of resistivity was measured. The strain measurements have been performed by measuring the relative length change at specific point of the specimen surface.

3.4 Measuring methods

Three different methods have been used and evaluated. The first method (A, four probe method) used four contact points of which current was input at points 3 and 6 while the voltage drop was measured between points 4 and 5 (see Fig. 1). The second method (B, two probe method) used only points 4 and 5 (two probe method), for current input and voltage measurement at the same time. The third method (C, also four probe method) used the end faces of the specimen for current input (points 1 and 2 in Fig. 1) and the voltage drop between points 4 and 5 was registered. The strain was calculated as the mean value of four strain gages that were placed in the central point of each rectangular face of the specimen.

4. Results and discussion

4.1 Influence of the measuring method

After the curing period in a humid chamber, the specimens used in these experiments were stored

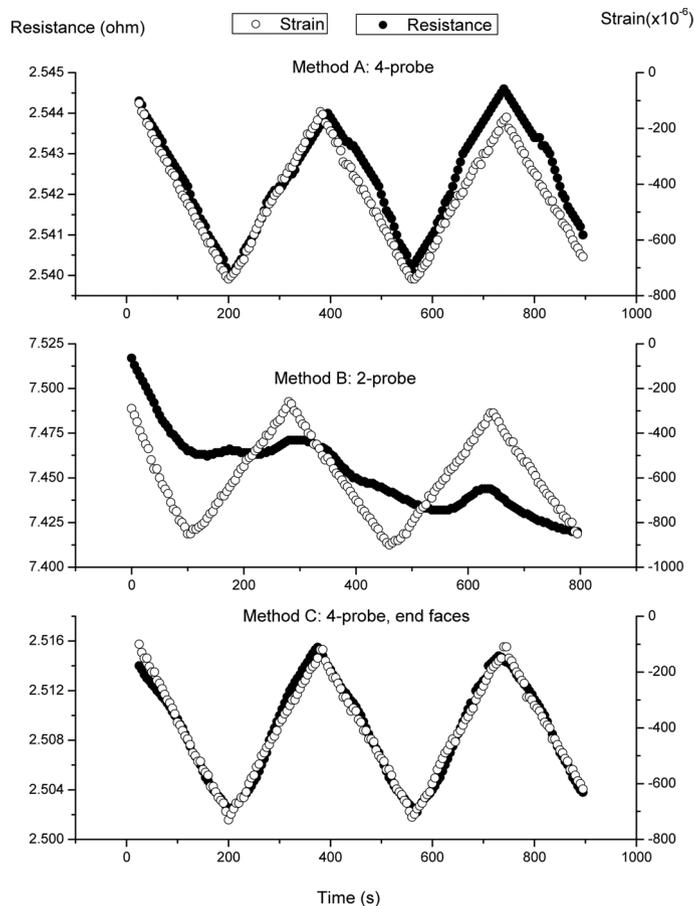


Fig. 2 Results obtained by each of the three measuring methods

in an ambient of 0% relative humidity until constant weight. The initial resistivity of the specimens (using four wire method) was approximately 4 $\text{ohm}\cdot\text{cm}$. Fig. 2 shows the results obtained by each of the three methods. With method A, a good response was obtained and it reverses when deformation reverses. But the sensitivity of this method is quite small because the electrical resistance amplitude along the test is very small ($< 5 \text{ m}\Omega$), so the results are plotted as discrete points. With method B the initial resistance is high due to the inherent contact resistance of the two-probe method, so in this method, in addition to the electrical resistance of the sample, there is another resistance that is being included in the measurement and that arises from the fact that electrons have to move from one material (copper wires) to another (specimen). In the four-probe method the contact resistance is avoided. In addition, as it can be observed in Fig. 2, the initial resistance is higher when using method B (two-probes). The difference between this value and those measured with any of the four-probe methods is the contact resistance. Also, in spite of the high resistance amplitude along the test ($> 100 \text{ m}\Omega$), presuming a good sensitivity, the reversibility is very poor and degrades in consecutive cycling. This lack of reversibility is likely due to an evolution of the contact resistance to lower values during the test, which maybe sharps the change in the resistance during the loading, and reduces the change in the resistance during the unloading. Finally method C provided an acceptable sensitivity (amplitude $> 10 \text{ m}\Omega$) and above all shows a high reversibility with the plots of deformation practically superimposing those of electrical resistance.

The conclusion of these experiments is that the most suitable method for further tests is method C, and for this reason it was the method used in the rest of tests made in this research.

4.2 Result repeatability

In order to verify the repeatability of results 10 additional equal tests using a single specimen were repeated. For these tests, specimens were stored in a 100% RH ambient except for the time of the test performing.

Fig. 3 shows the gage factor values recorded for the set of 10 tests. It can be seen that the values obtained remain rather stable, so good repeatability of the gage factor was achieved. Table 2

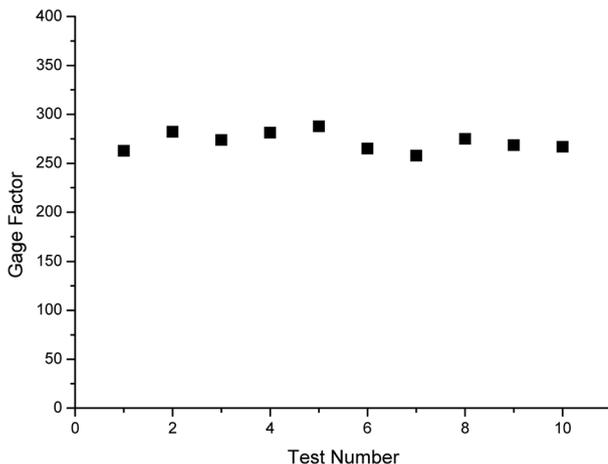


Table 2 Gage factor average values of 10 tests
(SD: Standard Deviation; RSD: Relative Standard Deviation)

	Gage factor
Average	272.1
SD	9.6
RSD (%)	3.5

Fig. 3 Gage factor values recorded for the 10 tests, in order to verify the repeatability of results

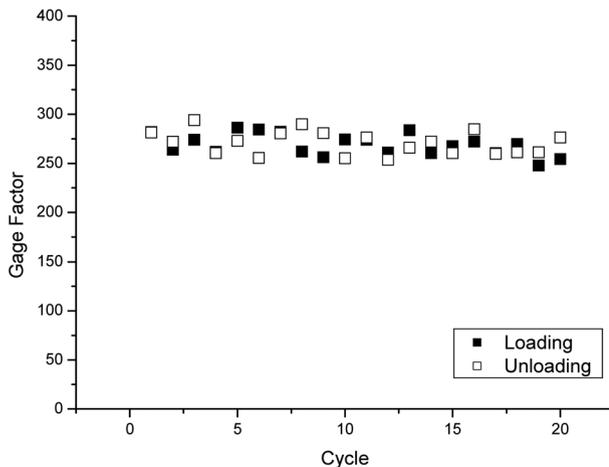


Table 3 Gage factor values for the loading, unloading and total processes are shown (SD: Standard Deviation; RSD: Relative Standard Deviation)

	Loading	Unloading	Total
Average	268.9	270.7	269.8
SD	11.1	12.1	11.5
RSD (%)	4.1	4.5	4.3

Fig. 4 Gage factor values obtained along a 20 cycle test

provides the mean value obtained for the 10 tests. This data show that the tests performed have good repeatability.

Fig. 4 shows the gage factor values obtained along a 20 cycle test. It is clear that the values are stable along the consecutive cycles. It also can be observed that the values obtained in the specimen unloading process are very similar as compared to the ones of the loading increasing process. The small difference in the values calculated may arise from the fact that there is an electrolytic contribution to the conductivity that is due to the ions of the pore solution (Wen and Chung 2007). The movement of these ions due to the electrical field applied and the movement of the overall solution due to the stress applied (Wen and Chung 2006) can probably modify the electrical response of the specimen, producing small variations in the resistance. Also the variation of the strain of the specimen causes small particle displacements that may affect the resistance in the unloading process, taking into account that the cement matrix has rheological behavior such as creep.

In Table 3 the gage factor values for the loading, unloading and total processes are shown. The presented values are the average of the ones obtained in the 20 cycles of loading-unloading of the test.

Also it was observed that there are not practical differences between the gage factor values obtained for different tests and for different cycles within each test. These results imply a good repeatability of the measurements. Nevertheless further tests for a definitive evaluation of this parameter are considered necessary in other experimental conditions. For the rest of tests made in this research it has been adopted the criterion of present the gage factor as a mean value of at least three consecutive cycles with their respective branches of loading and unloading.

4.3 Influence of specimen humidity

Fig. 5 shows the gage factors obtained in tests performed with specimens that had been previously stored in ambients with different RH value. The minimum gage factor values were obtained with an RH of about 40%. In previous works the mechanism of piezoelectric behavior has been described as based on the pull-out of the crossing fibers at the internal cracks of the specimen in areas with tension stress, and the consequent change of the contact resistance between fiber and matrix (Chung 2002).

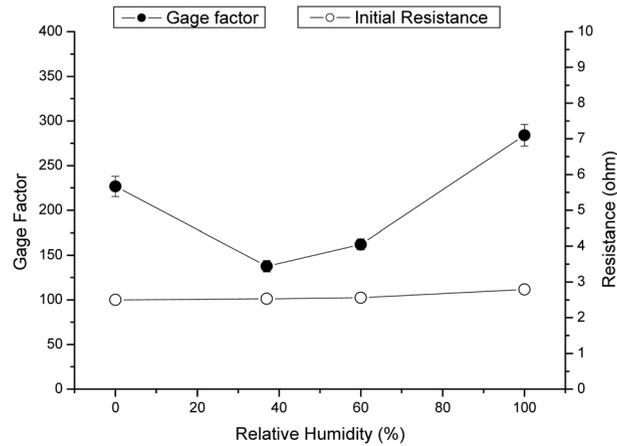


Fig. 5 Gage factors obtained in tests performed with specimens that had been previously stored in ambients with different relative humidity values. Intervals show standard deviation of three replicates

First it can be observed that the electrical resistance of the specimen (before being loaded) is independent of the RH value. This is because with 1% CF content the percolation threshold has been surpassed and there is a continuous electrical path along the specimen (Wen and Chung 2007). On the other hand the highest gage factor values occur when the specimen has been stored in either a saturated or a flat dry ambient. In the case of 0% of RH the electric flow is fully electronic and the piezoelectric behavior can be explained by the contact continuity. In the case of 100% of RH the percolation threshold of water has been exceeded and the current flow is both electronic and electrolytic. Both types of flow contribute to piezoresistivity, but the electronic is higher than the electrolytic. That is why the gage factor for $RH = 100\%$ is somewhat higher than the one for $RH = 0\%$. The specimens in which the water percolation threshold has not been reached the discontinuity of wet areas causes that part of the electrical current is devoted to polarization effects, therefore reducing the piezoresistivity effect and the gage factor values (Cabeza 2003, Cao and Chung 2004).

4.4 Influence of the loading parameters

Fig. 6 shows the results obtained in strain sensing experiments at different loading rates for stress amplitude of 10% of the specimen strength. This kind of experiments was also performed for some other stress amplitudes ranging from 5% to 25% of the compressive strength of the paste. Gage factors were calculated for each loading condition in order to study the influence of the loading rate and the maximum loading stress on the strain sensing response of the paste. In Fig. 7, the calculated gage factors are shown. It can be observed in this figure that when the maximum compressive stress of each loading cycle is increased, a higher gage factor is obtained. Additionally, there is not a significant influence of the loading rate in the gage factors registered. Only in the case of the higher amplitude stress (25% of R_c) it can be observed a decrease in the gage factor.

Fig. 8 shows the evolution of the resistance in the specimens while they are loaded, with a constant loading rate of 100 N/s, up to certain stress and then this stress is maintained for 5 min. As can be observed there is a little shift of the resistance towards lower values while the stress is maintained. This shift of the electrical response can be probably due to the accommodation of the

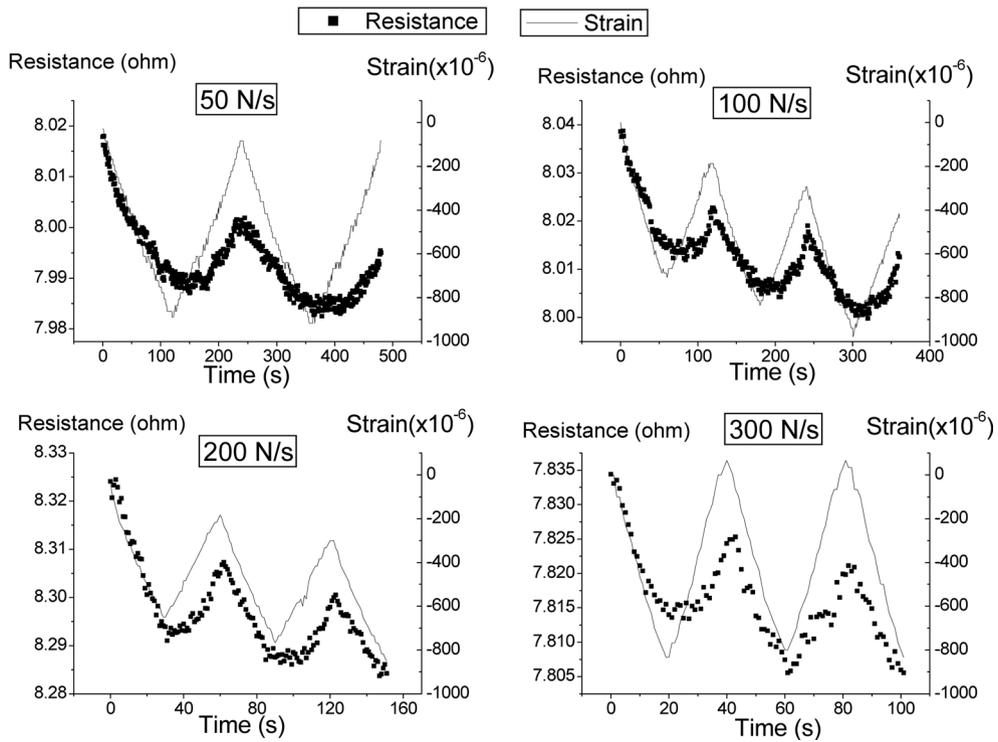


Fig. 6 Results of the strain sensing tests performed with specimens at 100% of RH for various loading rates and 10% of compressive strength as stress cycle amplitude

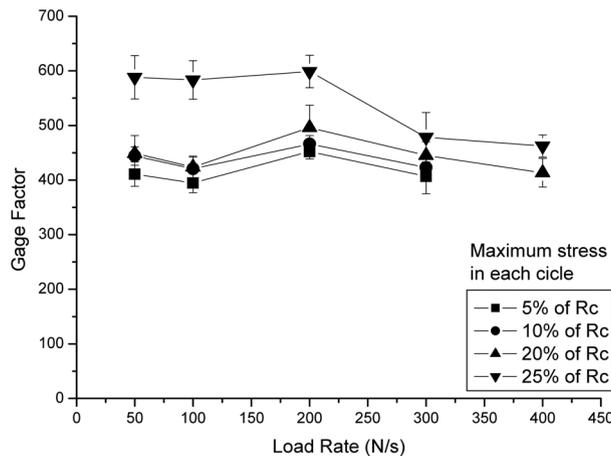


Fig. 7 Gage factors obtained for various loading rates and stress amplitude in cyclic compressive tests. Intervals are standard deviation of 3 replicates. Relative humidity of 100%. R_c = Compressive strength

pore solution in the cement matrix as a consequence of the stress applied (Wen and Chung 2006). This could be an explanation of the variations that are produced in the piezoresistivity behavior when the loading parameters are changed.

Obviously, in real structures is not easy to control both the loading rate and the maximum load

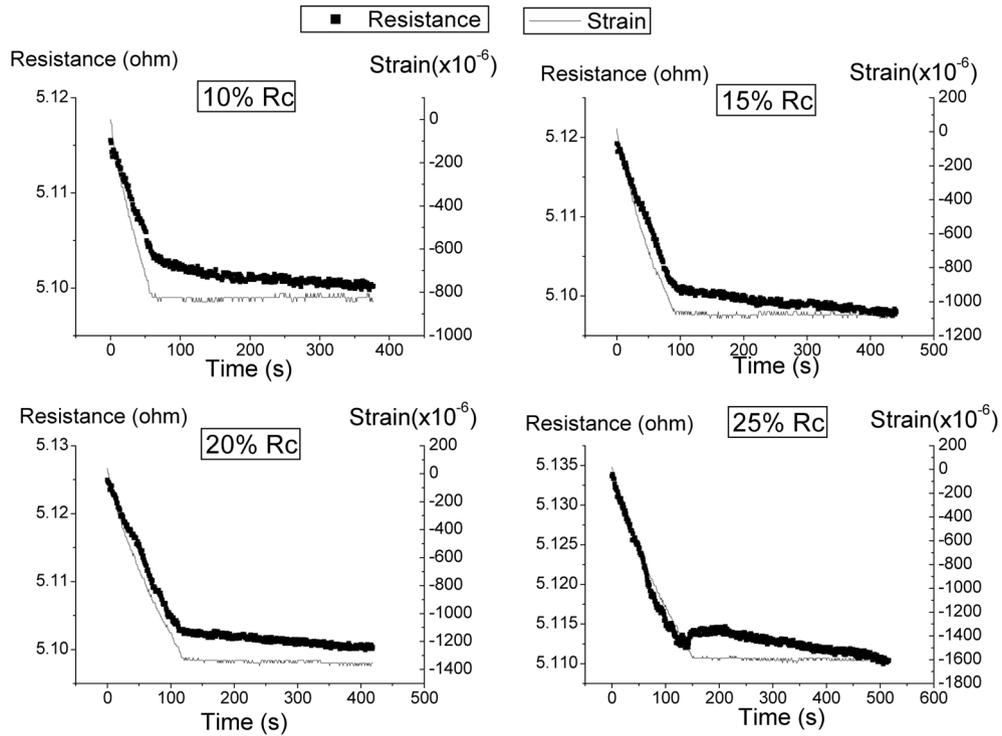


Fig. 8 Results of strain sensing tests for constant maximum load and different final loading stress, performed at 100 N/s

that the material is going to support. However, the strain sensing response of each element of the structure will depend on these parameters as well as others such as the relative humidity condition of the environment. For these reasons, the study of the strain sensing ability of carbon fiber reinforced cement matrices based on the piezoresistive phenomena is a challenging matter that will imply the reliability and reproducibility of the piezoresistive response of these composites.

4.5 Implementation in a computer based structural control system

Structural piezoresistive materials capable of sensing their own strain are much more interesting than standard external sensors attached to structure elements. One of the main advantages of multifunctional conductive cement composites is that they extend throughout the whole structure. There are plenty of systems to track people or vehicles over structures. Usually those methods are video based systems, using image processing algorithms (Shen *et al.* 2006, Ye *et al.* 2000). However they are restricted by poor light conditions. Besides, building a structure with a self-sensing material makes it possible the structural stress monitoring, and moving loads tracking in real time.

Fig. 9 shows an example of a computer based structural monitoring system using multifunctional composites. First of all the material should be calibrated, and the gage factor determined. Also mechanical characterization tests should be performed. Then, by controlling the current intensity input, the potential drop measure between two close points, selected anywhere on the structure, can be related to the stress level. The number of measuring spots can be as high as needed, as the whole

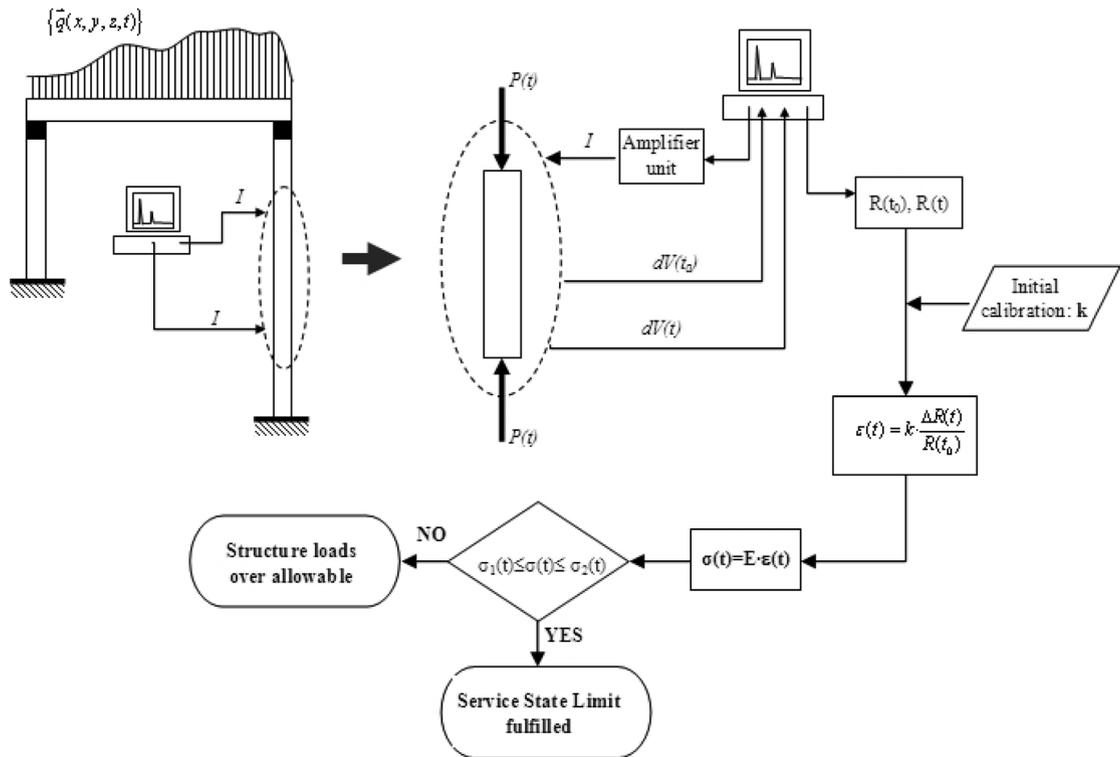


Fig. 9 Flowchart of a computer based structural control system

structure is self-sensing. That concept implemented in an artificial neuronal network would optimized position and number of data measured. Hence reducing the computational cost and algorithm complexity.

Similarly the implementation of intelligent control algorithm based on strain measurements (Katsikeros and Labeas 2009, Spencer *et al.* 2004) on multifunctional composites can be used to have a permanent feedback of the structural behavior for both the stress levels and the real time monitoring of the structural health of constructions such as buildings, bridges, dams, water or other fluid storage tanks, etc. (Song *et al.* 2006).

5. Conclusions

1. Among the studied methods the one of electrical resistance measurement based on 4 wires has been found to be the most suitable, because it is both highly sensitive and reversible, accordingly with prior works found in the literature. Nevertheless, this research has found that the method based on the use of the end faces of the specimen for current input and the measure of the voltage drop between two central points has shown the best performance. Contrarily, the two probe method, although more simple, provides poorer results, because the current input and the voltage drop is taken in the same points and consequently the contact resistance alters the results.

2. Although some dispersion is observed in the first cycles, a good repeatability of the results is

obtained between different tests. Also among cycles within a single test the gage factor obtained as a mean value of at least three consecutive cycles with their respective branches of loading and unloading are very close.

3. The response of the system is significantly dependent on sample humidity. The highest gage factor values occur when the specimen has been stored in either a saturated or a flat dry ambient. In the specimens in which the water percolation threshold has not been reached (RH of 60-80%) the discontinuity of wet areas causes that part of the electrical current is devoted to polarization effects, therefore reducing the piezoresistivity effect and the gage factor values obtained.

4. The loading rate applied to the specimen does not clearly affect the system response in the range under study in this research.

5. When the compressive stress is increased the gage factor increases until the elastic regime is surpassed.

6. The measurement system can be computerized and a control algorithm implemented in a real structure in order to have a permanent feedback of strains that can be used for health structural monitoring.

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