Piezoresistivity in Micro Carbon Fiber Silicone Composites for electrical resistance to Strain Sensing

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Abstract

In order to develop a cost-effective carbon fiber reinforced polymer sensor for compressive strain monitoring, a study was carried out to assess electrical and piezoelectric properties of samples containing five different carbon fiber weight percentages. Testing focused on sensing ability throughout measurement of resistivity: (1) when submitted to uniaxial variable compressive strain; (2) to time prolonged relaxation at constant strain; (3) and influence of environment temperature on measurements. Results enabled the possibility of usage for live monitoring of samples by determining sensitivity values of each sample being tested. Electrical resistance measurements assessment test results, show real time resistivity change in respect to experienced strain. Further piezoelectric properties where determined. An exponential decay function was found in fractional resistance in respect to relaxation due to constant strain testing. The total amount of time needed for measurements to present an error less than 1% at the probes was determined and found to vary up to seven days. Strain reversibility of resistivity measurements varied according weight percentages of carbon fibers used in composite sample being tested. Samples were tested in situ for monitoring of displacement on foundations of a dwelling to be built, placed on foundation's soil. The main objective here was to assess practical questions such as handling and how measurements could be made safely. Results demonstrated successful monitoring during construction phase with easy deployment on site, sensing each construction phase loading.

Keywords: Composite; Stress/strain Sensor; Carbon Fibber Reinforced Polymer; Piezoresistivity; Structures Monitoring

1 Introduction

Smart materials enable the possibility, through an adequate observation of loadings on monitored structural elements, to correct, when necessary, eventual anomalies that might appear during its life cycle. This becomes especially relevant when dealing with carbon fiber composites in high tech industries such as aerospace and automotive industries where maintenance and continuous serviceability status is required as high standards of durability and safety.

The capability of this type of composites act also as stress/strain sensor, opened the possibility of real time monitoring on such type of structural elements[1]. However, carbon fibers composites as any other material does present its limitations when dealing with fatigue or failure [1]. Delamination [2-3], matrix cracking or other types of fatigue might occur on said composites and be difficult to detect or monitor during its service life, mainly, because damage is not easily visible from an outside inspection [4-5].

Previous studies also demonstrated the possibility of carbon fibers laminates itself act as sensing of stress and strain [6-7], and differentiate from those with self-sensing ability of damage [8-12]. Although both can be achieved using carbon fiber polymer-matrix composites by means of resistivity measurements. Wang X. *et al* [1] studied sensing ability during delamination in carbon fiber reinforced polymers (CFRP) during fatigue by means of resistivity measurements and successfully demonstrated an increase of irreversible nature at 33% fatigue life of CFRP, it is also noted electrical resistance, R, increase upon loading and subsequent decrease upon unloading on every cycle indicating strain sensing capability of composites in analysis.

Structural monitoring has nowadays high costs due to materials and equipments used and requires need of specialized labour upon application. Carbon fiber polymer matrix composites containing short carbon fibers, present effective strain sensing capabilities due to composite's piezoresistivity nature. Said piezoresistivity is not only due to electrical conductivity of carbon fibers within composite matrix but also due to change in proximity between adjacent fibers when submitted to strain. Sara Cravanzola [13] characterized multiwalled carbon nanotubes. L. Wang [14] demonstrated piezo effect with carbon nanotubes and studied tunnelling effects of conductive paths on composites at study. Timothy M. *et al* [15] demonstrated improved results on micro carbon reinforced composites (MCRP) incorporating nickel nano-particles improving sensing capabilities for low values of strain.

1.1 Objectives

This study objectives, consist on development of MCRP strain sensors that are same time low cost and easy deployment, not requiring specialized labour. Samples tested are composited with a non-conducting polymeric matrix and conducting carbon fiber filler. When submitted to tension or compression loading, present change in resistivity [15-17]. This change is related to imposed strain and thus deformation can be plotted as a function of measured *R*.

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Previous research focused on piezoresistivity behaviour of MCRP when submitted to time changing stress / strain loading. Wang and Chung [18], Todoroki and Yoshida [19] and Xiaoping S. [16], investigated piezoresistance of MCRP composites when submitted to compressive loading. Park *et al* [20-21] proposed a model to describe change in electric resistance in fiber direction taking account fiber breakage due to tensile strain.

Little attention has been given to behaviour of this type of composite submitted to constant strain deformation for prolonged periods of time. The present study will focus on behaviour of electric resistance, *R* when samples are imposed to a constant deformation of 2,0 mm. During testing is also monitored the effects of temperature on *R* measurements. Is also investigated the behaviour when samples are being submitted to triangular loading for strains up to 5mm max deformation.

2 Materials and Experimental Work

On previous studies, several types of conducting fillers have been tested, namely metallic particles, carbon black and carbon fibers, commonly used, presented diameter ranging from ~10 μm to about 0.01μm.

MCRP composite matrix at study was made of silicone polymer (Silastic nº145, Dow Corning, Co.) mixed with carbon fibers of volume resistivity 150 μΩ.m and diameter of 20.7μm (Kureha Chemicals, Co.). Average length of producer fibers determined to have 1554 μm, referred here as L15. From initial fibers, were made new, using a crushing method on a cylindrical chamber at 6000 PSI compression loading during sixty seconds. Resulting fibers measured an average length of 311 μm, referred here as S03 (table 1).

Composite's samples were produced by mixing base polymer with a catalyst in a 10:1 ratio until resulting paste had uniform consistency. Carbon fibers were added and mixed into polymer paste, in a given percentage of polymer mass until a good dispersion was obtained. Final composite was then moulded into a cylindrical shape mould with 44mm diameter and later cured on a temperature controlled room at 40±2ºC for 24h (fig. 1).

Table 1 – Electrical resistivity values for samples studied; different fiber types and concentrations

Carbon fibers type (avg. length)	Concentration (% in weight)	Fiber volume fraction (m^3/m^3)	Electrical resistivity (Ω.m)
L15 (1554 μm)	5	0,023	12±1
	10	0,046	5,05±0,05
	13	0,060	0,56±0,04
S03 (311 μm)	40	0,184	11±3,54
	50	0,230	11±1,5

Five MCRP mixtures were prepared, each with different fiber weight percentages, dispersed randomly within matrix composite. L15 samples were moulded with micro carbon fibres weight percentages of 5%, 10% and 13%; S03 samples moulded with 40% and 50% weight percentages (%wt) (table 1).

In order to enable measurements on samples, was added specially designed electrodes at each extremity. Consisted of a very thin steel fiber mesh with a 1.0 gr weight, welded to a typical 2.0 mm diameter copper wire, placed during moulding at distance of 10mm from each extremity (fig. 1).

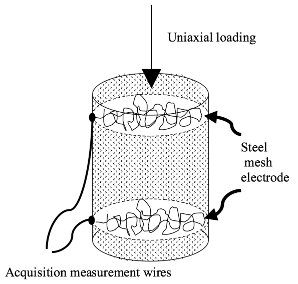

Figure 1 – simple schematic of samples being tested

This type of electrode presents advantage over typical surface silver paint as is not prone to detach from surface. The nature of fiber mesh used, enables a large number of contact points to be made available while sample is able to maintain electrode to composite conductivity through all operation, but more importantly, is able to endure more when handling on everyday usage.

Measurements of *R* were made using the two-probe method, 50mm apart, measured along applied deformation axis. Direct current (DC) was used, which is fit for this type of composites, as there isn't the problem observed on concrete samples of false polarization potentials on the contact zone of electrodes [22]. This type of two-probe electrodes was selected due to simplicity on manufacture as well as in everyday usage as sensing unit on a construction site when used in monitoring. Logging of results, were made with data logger Agilent 3970A. Operating measure time is less than a second and requires an output test current of 1mA with neglectable ohmic heating during operation.

Experimental testing was divided into two types of tests. Study of sensing ability began testing samples with a triangular loading during ten cycles on which sample's deformation was made vary from 0 mm to 5 mm. It was used the Lloyd LR30K testing equipment with load control and controlled displacement rate of 1,2mm/min. Arrangements were made in order to have electrical insulation from the loading equipment to avoid any possible interference on logged results. It was used samples without any prior test history before demoulding.

A handspike deformation apparatus with deformation control was used, with resolution of a hundredth of millimetre for study constant axial displacement when samples where kept without any strain variation for prolonged periods of time.

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3 Measurements of resistivity

Piezoresistivity in MCRP composites are characterized when resistivity changes in accordance with imposed strain, which translates into sensing ability by means of measurement of one another. Positive piezoresistivity sensing is defined by R increase with increased strain, whereas negative piezoresistivity translates sensing behaviour of a composite by decrease of R with increased strain [23]. Moreover, is important to evaluate reversibility nature of R to define strain sensing from irreversible measurements or damage sensing.

The principle behind a resistive sensor can be described by a conductive wire with resistivity, ρ, with electrical resistance kept constant regardless its deformation. When applied a traction force, starts to elongate its length, L, at same time the cross section, A, decreases. The electrical resistance, R of a conductive wire that varies directly with its length, can be described as follows:

$$R = \rho \times \frac{L}{A} \tag{1}$$

The variation of electrical resistance, ΔR, due to strain on a composite, is given by the difference between initial electrical resistance, R_0 and resistivity measured at a deformation, ΔL.

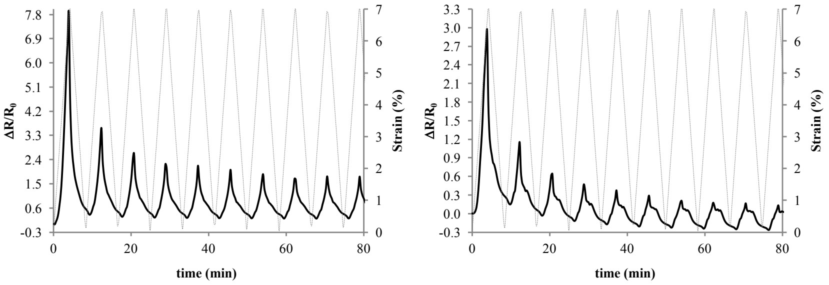

Figure 2–S03 sample with 40wt% of fibers (left side) and 50 wt% of fibers (right side) experimental results

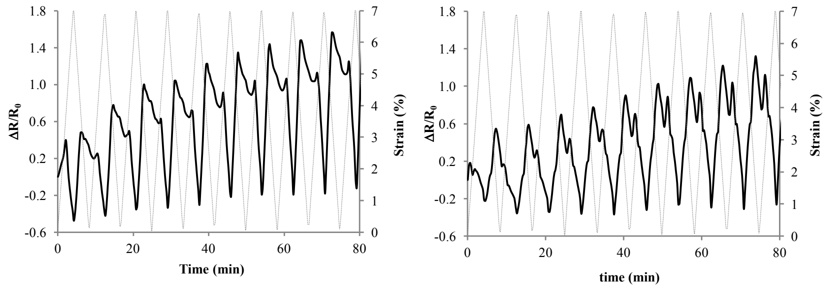

Figure 3–L15 samples with 5wt% (left side) and 10 wt% of fibers (right side) experimental results

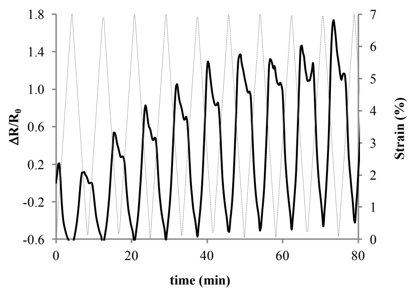

Figure 4–L15 samples with 13wt% experimental results

Consider hook's law to evaluate equation 2, the fractional increase in electrical resistance, $\Delta R/R_0$, can be related with fractional increase on applied displacement deformation of sample at study according to the following:

$$\frac{\Delta R}{R_0} = \kappa \cdot \frac{\Delta L}{L} \tag{2}$$

$$\kappa = \frac{\Delta R}{R_0} \cdot \frac{1}{\varepsilon} \tag{3}$$

where, $\varepsilon\, is$ strain to be sensed according to above equation 4 and κ is strain sensitivity (or gauge factor) for any given sample at study.

To evaluate the reversible nature of R on L15 and S03 samples, was imposed, during ten triangular cycles loading and unloading, a longitudinal axial deformation up to 5mm at a rate of 1,2mm/min.

Figures 2 to 4 present factional change results of samples at test. Triangular load / unload is shown in dashed line throughout all referred figures. Figures 3 and 4 depict results obtained, relating fractional resistance, $\Delta R/R_0$, with strain experienced on L15 samples. These samples are characterized by negative piezoresistivity. When axial compression is applied although reducing the effective axial length, low content of carbon fibers, results on overall increased distance of composite conductive paths which results on a decrease in sample's conductivity.

Analysis results show samples to have irreversibility behaviour (table 2), initial verified by Shoukai Wang and D.D.L. Chung [23]. Irreversibility can be attributed to a phenomenon of micro cracking on composite related to compression during loading phase. When unloading starts during testing, fibers are rearranged into new positions, altering resistivity of sample's composite. S03 type samples (fig. 3), unlike L15 type ones, have high content of carbon fibers and are comprised with shorter length. The high content and also being approximately one fifth in length, produces a composite compact and more prone to micro cracking, as noticed differences in $\Delta R/R_0$ measurements on initial loading cycles. Once micro cracking occurred, rearrangement of fibers in the polymer matrix is the dominant affect, altering piezoresistance measurements, changing measured R at same time.

For first time loading, is expected sample's composite matrix to develop micro cracking while rearrangement of fibers is occurring, until maximum stress amplitude is reached. Micro cracking is followed by rearrangement of fibers, as dominant effect on composite matrix during testing. Stress in the vicinity of the interface of composite at test is also important but a complex task requiring further research.

Irreversibility values determined shown on table 2, are in respect to 7th cycle of testing. Considered at this stage micro cracking already took place and only rearrangement of carbon micro fibers is in place. Values of irreversibility from the 7th to 10th cycle where determined to be less than 0.05.

Table 2 – Piezoresistance and stress properties of samples

		R_0 (Ω)	$\lvert\Delta R/R_0\rvert$ Reversible	$\lvert\Delta R/R_0\rvert$ Irreversible	Stress amplitude (MPa)	Strain Sensitivity
L15	5%	131	0,26	0,01	0,22	3,8
		84	0,83	0,02	0,23	11,8
		147	0,27	0,12	0,26	3,9
	10%	47	1,26	0,10	0,30	18,0
		59	1,16	0,40	0,30	16,6
		58	1,48	0,14	0,26	19,2
	13%	7	3,66	0,48	0,41	52,3
		72	1,29	0,22	0,40	18,4
		6	3,63	0,04	0,28	51,8
S03	40%	88	0,37	0,01	0,23	5,3
		1154	0,64	0,02	0,16	9,1
		189	0,40	0,04	0,16	5,7
	50%	113	0,22	0,03	0,39	3,2
		144	0,18	0,00	0,53	2,5
		69	0,50	0,02	0,42	7,2

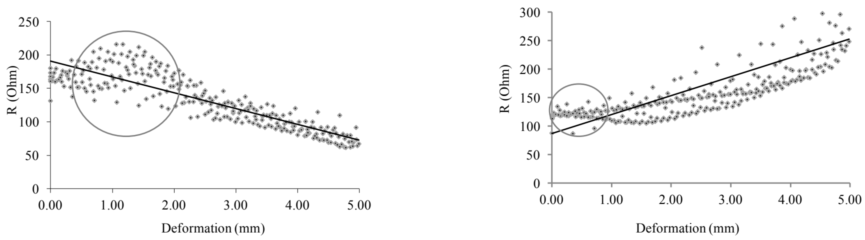

Figure 5 – Scattering of 10-cycle R measurements for L15 with 5wt% (left) and S03 with 40wt% (right)

L15 sample's results show a more pronounced increase in R during approx. 2/3 of cycle testing. Such behaviour is explained by lower initial quantities of fibers on composite matrix, associated with rearrangement of fibers and change in contact points within matrix, altering conductive paths on composite's sample and in turn, its overall resistivity.

On both graphics shown in figure 5 is plotted all data for the 10 cycles of testing as function of sample's deformation (mm) while measurements of electrical resistance are being logged at electrode's extremities. For L15 samples, for near zero deformation, is observed a local decrease in R followed by final increase until zero deformation is attained (fig.3 and 4). Same effect can be detected on figure 5, left side one, on area with wider dispersion of results for initial deformation (denoted with a circle). This behaviour can be explained due to construction of composites at study, in particular how electrodes are embedded in. Dispersion values can be correlated to location where electrodes are inserted on sample height. Since electrodes are constituted of small steel mesh involving composite matrix, forces fibers to rearrange differently, in respect to remainder portion of the composite associated with fiber breakage and rearrangement, from cycle to cycle, produces changes on the conductive paths within sample's composite.

In respect to S03 samples on same figure 5, right side one, while fiber breakage isn't occurring, is observed lower measurement dispersion values (denoted with a circle). As soon as fiber breakage effects and rearrangement, start to occur (at higher strain values), increased dispersion of values observed means the conductive paths are also changing, from cycle to cycle, including the ones that are made with electrode's steel mesh. Typical and similar results were observed on other type of samples: L15 with 10%wt and 13%wt as well as on samples type S03 with 50%wt in respect to the ones presented on figure 5.

On both cases, relaxation of fibers that were previously under tension may also be producing so-called piezo effect lowering or increasing R according to sample type, before zero deformation is reached (figures 2, 3 and 4).

Strain sensitivity analysis values where determined from eq. 3 using experimental data obtained from the last (10^{th}) cycle of testing. As an example, for a L15 sample with 5wt% carbon fibers, a reversible strain of 7% resulted on reversible stress of 0.22MPa with reversible $\Delta R/R_0$ of 0.26. Strain sensitivity defined as $\Delta R/R_0$ per unit of strain was determined to be 3.8 (table 2).

4 Resistivity during Relaxation

At present, piezoresistive behaviour of MCRP when subjected to external load or deformation is fairly accepted [24-26]. However, past research works focused mainly on analysis of behaviour during triangular loading environments [15-16]. Under such conditions, results show this type of carbon fiber construction for sensing, to have good piezoresistivity agreement with strain. However little has been done testing *R* behaviour when samples are being submitted to a prolonged period of time at constant strain. It is known that polymer based materials intrinsic viscoelastic properties include the relaxation phenomenon and when it occurs, composite matrix carbon fibers tend to be rearranged in closer proximity altering the piezoresistivity behaviour of it while strain is maintained the same.

Relaxation effect is particularly important on the sensing ability of samples as these may not be constantly subjected to strain variation, rather be in a state of constant strain for prolonged periods time. Think for instance sensor's setup to measure displacement of foundations in a concrete structure. On such conditions is important to quantify resistivity during long periods of unchanged strain.

Table 3- $\Delta R/R_0$ after seven-day testing, 2.86% strain

	Carbon Fibers wt%	$\Delta R/R_0$
L15	5	-0.49
	10	-0.40
	13	-0.34
S03	40	-0.65
	50	-0.50

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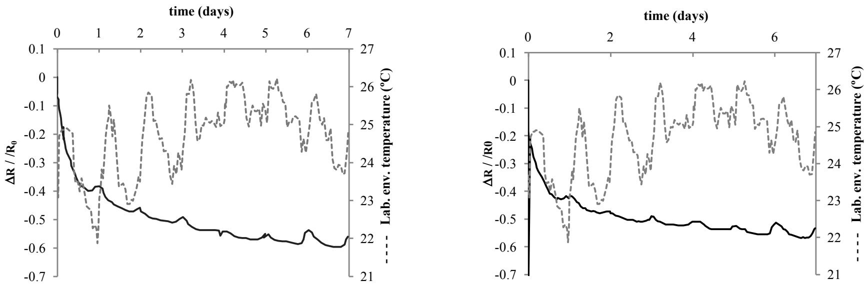

Figure 6 –Behaviour samples type S03-40wt% (left) and type S03-50wt% (right).

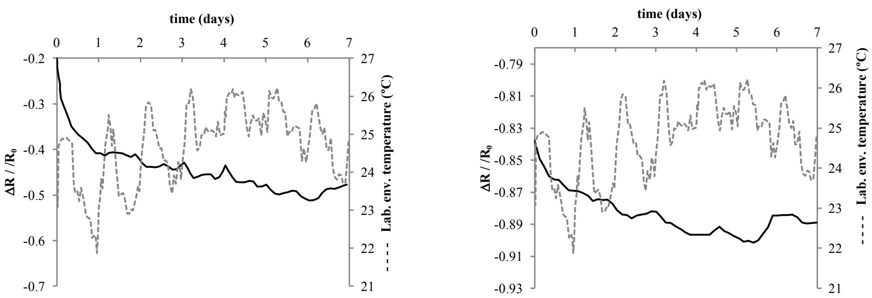

Figure 7 - Behaviour samples type L15-5wt% (left) and type L15-10wt% (right).

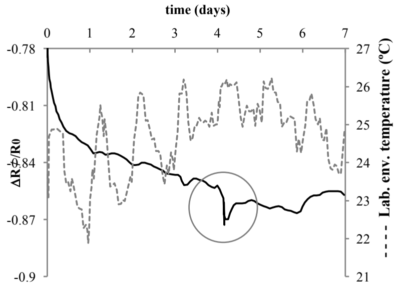

Figure 8 –Behaviour samples type L15-13wt%

To evaluate variability of R when subject to those conditions, MCRP samples were tested by imposing on samples an axial deformation of 2mm (2.86% strain) during seven days while R was being monitored over given period time. Determined ${}^{\Delta R}/_{R_0}$. values are presented on table 3 and concern fractional change at the end of test. Figures 6 to 8 present factional change results of samples at test during a 7-day period relaxation. Laboratory temperature load / unload is shown in dashed line throughout all referred figures. L15 relaxation behaviour is depicted on figures 7 and 8, the ones related to S03 samples, depicted on figure 6. All samples presented similar R behaviour in a decreasing trend. This is explained due to the viscoelastic properties of the composite material used to produce samples at test. Table 3 summarizes the final variation of $\Delta R/R_0$ measurements from the imposed strain. Change was largely reversible during subsequent unloading; however, it was noticed R returned to a slightly but higher initial value. Such behaviour may be explained due to damping effect of composite since it's made of silicone rubber. This same variation in R may also indicate micro cracking have occurred, as one can expect for higher values of stress.

Fractional resistance change decreased overall, during 7 days of testing. L15 samples, $\Delta R/R_0$ values range between -0.49 for samples with 5wt% fibers content and -0.34 for samples with 13wt% fibers content. S03 sample's $\Delta R/R_0$ values varied between -0.65 and -0.50, respectively to samples with 40wt% and 50wt% fibers content. Is noticed largest variation to occur within first two days of testing. On average, $\Delta R/R_0$ was -0.50, for all sample types.

Due to temperature dependence, it's observed daily R increase peaks in all MCRP samples. This behaviour is in agreement with temperature amplitudes that laboratory experienced during testing. Although observed temperature variations affected R behaviour, increasing to a local daily maximum, overall results show that decreasing trend overtime (figures 6 to 8) to stay unchanged. Experimental data was found to best fit the following equation (eq.5):

$$\frac{d}{dt}\left(\frac{\Delta R}{R_0}\right) = a\times e^{bt} + \left.\frac{\Delta R}{R_0}\right|_{t>0} \qquad (4)$$

where t, is time in days and $\left.\frac{\Delta R}{R_0}\right|_{t>0}$, b are constants represent respectively amplitude and decay rate. Equation 5 allows one to evaluate the amount of time needed to minimize R change between two consecutive days (table 4).

Table 4 - Regressions values for relaxation for equation 5

Designation	wt%	a	b	$\Delta R_1/R_0$	r^2
L15	5	- 107/174	2/227	- 88/423	0,83
	10	- 278/321	1/176	- 222/265	0,76
	13	293/359	1/95	- 313/399	0,85
S03	40	- 199/526	60/653	103/743	0,78
	50	- 158/635	61/837	- 31/984	0,75

Table 5 presents results in order R measurements to have less than 1% variation between daily measurements. From results presented in table 5, was determined time needed to achieve 1% maximum change on two consecutive days.

Table 5 – Time (days) needed for R measurements with less than 1% variation between daily readings

	Carbon Fibers wt%	days
L15	5	5
	10	6
	13	7
S03	40	6
	50	7

On figure 8 is show a fast decrease in fractional resistance between days 4 and 5. Such behaviour is explained by works inside the laboratory were experimental campaign was ongoing, sensing the fall of a huge steel H profile in the vicinity of the laboratory. Regardless, due to viscoelastic properties of the silicone composite, relaxation trend, resumed after the damping effect ended.

5 In situ applicability assessment

After characterization on laboratory conditions, a new set of MCRP composites samples were moulded for testing practical usage in situ while performing as self-sensing material or sensor. To do so, a to be constructed two-storey dwelling was selected. MCRP samples were placed on terrain just below where each concrete foundation was being casted.

Was observed during installation the necessity to accommodate samples more thoroughly during casting of first concrete elements to avoid damage to connecting wires of the sample composite.

Composite samples were placed on the geometrical centre of each slab foundation to be monitored with top surface in direct contact after casting. Measurements began right after proper placement on terrain and continued during construction until all gross works completed. Table 6 lists major construction phases and built elements at each measurement.

L15 with 5wt% and S03 with 40wt% where selected for in situ applicability and sensing testing and results are shown on figure 9. Sensing behaviour of samples as construction of the building took place was detected.

Table 6 – Measurements made during construction phase of the dwelling

Measurement nº	R measurement on total elements built
1	Composite samples setup and placement Concrete foundations casted
2	1m height of concrete wall
3	(Unchanged)
4	Concrete wall raised to 2m height
5	Floor slab casted and pillars
6	2nd floor slab casted
7	(Unchanged)
8	Exterior and interior ceramic brick walls built

The largest variation in strain of measurements made was observed when foundation elements where casted mainly because foundation soil was excavated before casting and therefore not fully compacted.

Followed additional construction of concrete foundations and wall structural elements until exterior and interior ceramic brick walls were in place (table 6). Results are shown on figure 9 and present agreeable sensing behaviour when compared with laboratory experimental results.

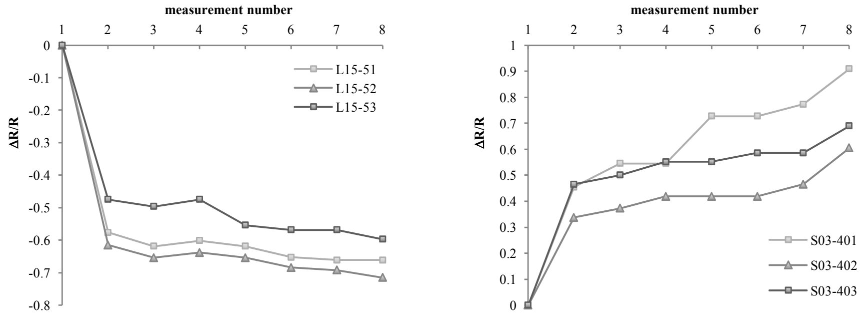

Figure 9 – L15-5wt% (left) and S03-40wt% (right) soil to foundations measurements.

6 Conclusions

This study objective consisted on development of MCRP sensors that are at same time, low cost and easy deployment, not requiring specialized labour. Research work focused on piezoresistivity behavior upon axial compression of cylindrical samples to strain. Testing focused on five types of MCRP composites, three with carbon fibers average length 1554μm (L15) comprised with 5%, 10% and 13% in weight content; two with carbon fibers average length 311 μm (S03) with 40% and 50% weight content of carbon fibers. For each of the composites studied, during triangular loading, L15 samples found to have negative piezoresistivity whereas S03 samples presented positive piezoresistivity. This reverse behavior is explained by differences in length of fibers used plus differences on weight percentages used on each composite type.

Samples found to have fractional change to resistivity between cycles that can be explained by:

(1) Occurrence of micro cracking on initial triangular loading cycles;

(2) Bending of fibers in the composite upon loading, producing piezoelectric effects, altering ρ;

(3) Rearrangement of fibers in the composite altering piezoresistivity and conductive paths within composite;

As for any this type of composites, connectivity is sensitive between composite and measurement equipment on setup. Further development is needed to correct observed dispersion on initial deformation and improve quality of measurements (fig. 5). Such shortcoming can be mitigated by placing composite sample with initial 2mm pre-load.

Time prolonged constant strain experimental data show composite's samples relaxation to decay exponentially. Samples with highest value of $\Delta R/R_0$ of -0.49 was determined to be for samples type L15 with 5% weight content of carbon fibers; and -0.65 for samples type S03 with 40% weight content. Results show the needed to have a database of values with 7 days logging results so detection can be made of unchanged strain less than 1% change in electrical resistance for two consecutive days. Such results make this type of composite more suitable for monitoring loading requests with slower solicitations such as displacements on foundations, especially at early stages of construction. When used in a set or group, for instance, my enable early determination of eccentricities for vertical structural elements.

In situ testing helped access applicability of this type of composites in a real construction environment. The type of self-sensing composite manufactured and tested found to be adequate for usage on an in-situ environment, able to endure when handling during setup and placement on foundation terrain while all gross works preparation is happening at same time. However, further research and development is required to assess more accurately strain under such conditions.

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