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Electromechanical Testing of Smart Lime Mortars for Structural Health Monitoring

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Abstract. Masonry structures are characterised by low tensile strength and limited ductility. Excessive static or high-cyclic loading or seismic excitation can lead to large localised strains and cracking. It is therefore essential to monitor the response of masonry structures to external loading, especially in the case of historic buildings and infrastructure. The present work aims at designing novel smart intervention materials for multifunctional application in historic masonry structures as a means of SHM, simultaneously structurally and chemically compatible with the in-situ material. The materials investigated consist of lime mortars mixed with different conductive micro- and nanofillers dispersed in the binder. Smartness stems from the materials' enhanced piezoresistivity, namely the constitutive relation between strain and electrical resistivity. Through application as a repointing agent in existing structures, these materials can be used as deformation and damage sensors. Electromechanical testing employing cyclic compression was conducted on mortars with different doping levels of three conductive fillers: graphite powder, carbon nanotubes and carbon microfibres. The electromechanical study involved the determination of the piezoresistive gauge factors of the different mixes for determining the optimal doping level for each employed filler. The mortars were evaluated in terms of piezoresistive sensitivity and structural application scalability.

Keywords: lime mortar · carbon nanotubes · carbon microfibres · graphite · smart materials · piezoresistivity

1 Introduction

Lime mortars are the preferred choice as intervention materials in historic masonry structures [1]. This is due to the chemical and mechanical compatibility between intervention and existing materials. This has motivated the extensive physical and mechanical investigation of the properties of lime mortars with various combinations of binders and aggregates [2–4].

Structural health monitoring (SHM) of historic structures is an important aspect in the evaluation of their structural performance [5] and a key factor in the correct timing of sustainable preventive maintenance. However, this complex task often requires the design and installation of externally mounted sensors [6]. The interference with the function or appearance of historic structures caused by these sensors is not desirable from a conservation engineering perspective.

Smart materials as a structural intervention (repair/strengthening) agent are a promising solution in the area of conservation engineering. Their multifunctional properties allow them to be used as an integrated sensor of features such as temperature and strain [7]. This multifunctionality can be accomplished through the doping of the base material with electrically conductive fillers [8, 9]. The conductive fillers enhance the piezoresistive performance and the sensing capabilities of the matrix material [10]. While modification of lime mortars for the enhancement of their mechanical properties has been attempted in the past [11], a systematic study of the piezoresistive enhancement of this material through modification has not been previously attempted.

This paper presents the electromechanical testing of a series of lime-based mortars modified through different types of conductive carbon micro- and nanoscale fillers. The mortars were subjected to mechanical loading while changes in their electrical resistivity were monitored. The purpose of this investigation was to determine the content of fillers for optimising the piezoresistive behaviour of the material. Additional consideration is given to practical aspects of the doping process, which differed depending on the type of filler used. It has been found that the successful design of a high-performance multifunctional material is the first step in designing and implementing smart structural interventions for SHM on historic masonry structures.

2 Methodology

For the purposes of the testing campaign, plain, or unmodified, and modified mortars were produced. The unmodified mortars were made of moderately hydraulic natural hydraulic lime (NHL 3.5) as a binder mixed with a siliceous sand, graded 0 – 3 mm grain size as an aggregate. The modification of the mortars was accomplished through the inclusion of three types of conductive micro- and nanofillers: graphite (G), carbon nanotubes (CNT) and carbon microfibres (CMF). G is a powder consisting of oblate

particles with low aspect ratios. CNTs are carbon atom lattices forming high aspect ratio fibrous tubes. CMFs are elongated strands of carbon fibre tows resulting from precision cutting. Being carbon-based, all these fillers are characterised by very high electrical conductivity [12]. They can be incorporated in varying doping levels in the lime binder. All the different mortar types investigated, along with the filler type and doping percentage by mass, are detailed in Table 1. G requires higher doping content for achieving percolation compared to CNTs and CMFs. This is due to G having a much lower aspect ratio than the latter two fillers, which hampers the formation of a conductive network throughout the bulk material.

Table 1. List of different mortar mixes: filler type and content percentage against the binder by mass.

Mortar	G/b (g/g %)	CNT/b (g/g %)	CMF/b (g/g %)
G0F0T0	0.0	0.0	0.0
G1F0T0	1.0	0.0	0.0
G2F0T0	2.5	0.0	0.0
G3F0T0	5.0	0.0	0.0
G4F0T0	10.0	0.0	0.0
G5F0T0	20.0	0.0	0.0
G0F1T0	0.0	0.1	0.0
G0F2T0	0.0	0.2	0.0
G0F3T0	0.0	0.3	0.0
G0F4T0	0.0	0.4	0.0
G0F0T1	0.0	0.0	0.01
G0F0T2	0.0	0.0	0.05
G0F0T3	0.0	0.0	0.10
G0F0T4	0.0	0.0	0.20

G: graphite , CNT: carbon nanotubes, CMF: carbon microfibres, b: binder

The volume ratio of lime binder over sand aggregate was 1:3 for all mortars. No cement or other additives were included in the mortar, in keeping with conservation engineering guidelines and empirical application rules regarding lime mortars. Assuming that electrical current primarily passes through hardened unmodified or modified NHL paste, these volume proportions for the dry ingredients, common for repointing mortar in masonry, mean that the volume ratio of the bulk material being modified through conductive fillers is relatively low. Following an initial investigation of the workability of the mortar for different water content, a water/binder ratio of 1.6 by mass was used for all mortars in order to obtain sufficiently workable mortar even after the incorporation of filler that might have negatively affected it.

The conductive fillers need to be evenly dispersed in the binder for achieving percolation and piezoresistive enhancement. G and CMFs can be incorporated in the binder and evenly dispersed through ordinary mechanical mixing prior to the addition of water. Conversely, CNTs need to be dispersed in the water used in each mortar batch through ultrasonic homogenisation, which can be costly, time-consuming and can be carried out on water batches of limited volume. Therefore, in this investigation G and CMFs were incorporated in the binder through mechanical mixing while CNTs were dispersed in the water using ultrasonic homogenisation. No chemical dispersants were incorporated in the water as they might interfere with the binder-filler interface and block electrical current transmission.

Following the mixing of the dry ingredients, fillers and water in a standard mortar mixer, the fresh mortar was cast in $50 \times 50 \times 50$ mm cubic moulds and compacted using a tamper rod. Subsequently, two stainless steel mesh electrodes were inserted into the fresh mortar in the mould. To minimise the disturbance to the mortar, the lateral wires were removed from the embedded portion of the electrodes and the mortar was lightly compacted again after insertion. The moulds were then sealed in airtight polyethylene bags and stored in the laboratory for 7 ± 2 °C in order to avoid moisture loss. The specimens were demoulded after 4 days, at which point the mortar was sufficiently hardened so that it could be carefully handled without damaging the cubes. An illustration of the mortar cubes, the arrangement of the embedded electrodes and the loading direction for electromechanical testing is shown in Figure 1a and b.

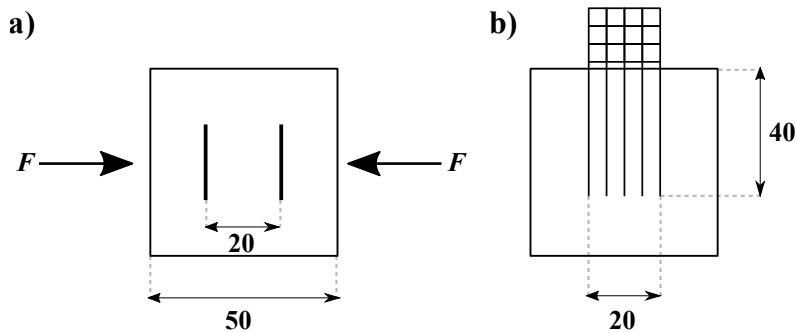


Fig. 1. Schematic of electromechanical test setup: a) embedded electrodes with respect to loading force F . b) Cross section of specimen with embedded mesh electrode. Dimensions in mm.

The mortar cubes were subjected to electromechanical testing at the age of 49 days. This testing consisted of the application of repeated compressive loading

at different magnitudes in sequence: 2 cycles at $[-0.2, -0.4]$ N/mm², 2 cycles at $[-0.2, -0.6]$ N/mm² and 2 cycles at $[-0.2, -0.8]$ N/mm². The compressive load was applied using a UTM 14P press by IPC Global. The deformations of the specimens were measured using strain gauges, complemented by LVDTs attached to the load plates for acquiring reliable measurements after compressive cracking was observed on the mortar.

The specimens were supplied with a ± 2 V square wave voltage with a frequency of 1 Hz using a Rigol DG1022 function generator. Using alternating instead of direct current assists in substantially reducing polarisation in the specimens, while the low voltage magnitude and low frequency simplify and facilitate field applications [10]. The electrical current I in the circuit was monitored using an NI PXIe-4071 digital multimeter during the application of the compressive load. The mortar specimen acted as the resistor in the circuit. The resistance R between the embedded electrodes was calculated using Ohm's first law:

$$R = \frac{V}{I} \quad (1)$$

The resistivity ρ of the bulk material was derived from the resistance R using Ohm's second law:

$$\rho = R \frac{A}{L} \quad (2)$$

where A is the cross section of the specimen (equal to 25mm²) and L is the distance between the electrodes (equal to 20mm).

The overall arrangement of the electrical circuit is illustrated in Figure 2, with V representing the function generator, I the multimeter and R the mortar specimen.

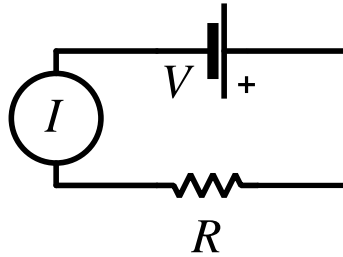


Fig. 2. Circuit diagram for electrical measurements.

The piezoresistive effect linking the observed strain ε , namely the relative change in length, and the measured relative change in resistivity $\Delta\rho/\rho$ is expressed as the gauge factor λ :

$$\lambda = (1 + 2\nu) + \frac{\Delta\rho/\rho}{\varepsilon} \quad (3)$$

where ν is the Poisson's ratio of the material. The terms in parentheses in Eq. 3 can be disregarded. Higher λ values indicate higher piezoresistive sensitivity of the material, hence enhanced performance as a smart sensor of strain through electrical measurements. The gauge factor λ was calculated through linear regression modelling between the observed values for ε and $\Delta\rho/\rho$ during the electromechanical tests.

3 Results

The development of the resistivity of the mortars was monitored during the 49 days of hardening. The results of these measurements are illustrated in Figure 3 for the different filler types at different doping levels. The modified materials were characterised by lower resistivity with higher filler content (the exception being the measurements on G specimens at 14 days due to a large drop of temperature on the day of measurement for some specimens). The resistivity increased as curing progresses due to loss of moisture from the open pore system in the bulk material. The decrease in resistivity for higher filler content become less pronounced as curing progressed.

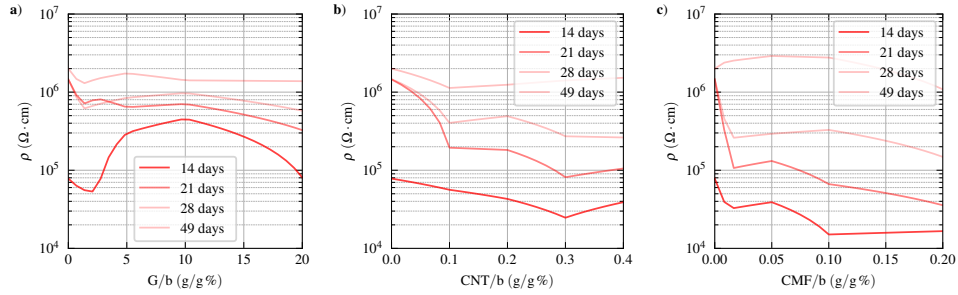


Fig. 3. Development of mortar resistivity for different filler types and content: a) G, b) CNT and c) CMF.

The results of the electromechanical testing are detailed in Table 2. The gauge factor λ is the measure of the piezoresistive sensitivity (Eq. 3) of the material while

the resolution δ , equal to the 95% confidence interval in the strain domain, expresses the sensing linearity; lower values of δ indicate enhanced sensing linearity. For G the maximum value for λ was achieved for 10% filler content, with an improvement of 179.92% being registered. For CNTs an improvement of λ by 48.57% was achieved for 0.2% content. Finally, CMFs resulted in an increase in λ by 154.30% for 0.10% content. Higher values of filler content than the ones mentioned led to a decrease in λ , signifying overpercolation of the bulk material due to an excessively high amount of conductive filler or inhomogeneity of the material. Therefore, G and CMFs were roughly matched in sensitivity improvement achieved.

Table 2. Results of electromechanical tests. Percentile difference between modified and unmodified mortar in parentheses.

Mortar	λ (-)	δ (-)
G0F0T0	52.3	4.632×10^{-3}
G1F0T0	97.5 (+86.42%)	2.926×10^{-3} (-36.83%)
G2F0T0	114.3 (+118.55%)	2.239×10^{-3} (-51.66%)
G3F0T0	81.5 (+55.83%)	3.079×10^{-3} (-33.53%)
G4F0T0	146.4 (+179.92%)	1.196×10^{-3} (-74.18%)
G5F0T0	58.5 (+11.85%)	4.334×10^{-3} (-6.43%)
G0F1T0	76.5 (+46.27%)	2.634×10^{-3} (-43.13%)
G0F2T0	77.7 (+48.57%)	3.549×10^{-3} (-23.28%)
G0F3T0	61.8 (+18.16%)	4.732×10^{-3} (+2.16%)
G0F4T0	44.7 (-14.53%)	5.842×10^{-3} (+26.12%)
G0F0T1	59.4 (+13.58%)	3.837×10^{-3} (-17.16%)
G0F0T2	94.3 (+80.31%)	2.472×10^{-3} (-46.63%)
G0F0T3	133.0 (+154.30%)	1.995×10^{-3} (-56.93%)
G0F0T4	63.0 (+20.46%)	4.191×10^{-3} (-9.52%)

G led to the maximum achieved improvement in linearity through a reduction of δ by 74.18% at 10% filler content. CMFs achieved a 56.93% reduction in δ at 0.10% content. These two filler contents coincide with the contents leading to the maximum increase in sensitivity. Conversely, CNTs led to a maximum improvement in linearity equal to 43.13% at 0.1% filler content. Therefore, the optimal filler content for CNTs appears to lie between 0.1% and 0.2%.

Finally, the piezoresistive behaviour of the mortar is illustrated in Figure 4 showing the results of the G3F0T0 mortar mix as an example, with all specimens exhibiting a similar response. The cycles up to 0.4 N/mm^2 compressive stress showed substantial linearity, while the cycles up to 0.6 N/mm^2 showed signs of hysteresis. The cycles up to 0.8 N/mm^2 showed clear indications of damage through residual

strains. The specimens were also substantially cracked at the end of the final load cycle. The presence of cracks can lead to the reduction of the effective cross-section A of the specimen as defined in Eq. 2, thus resulting in a decrease in the resistance of the specimen. While this cracking can interfere with the strain sensing capabilities of the smart sensor, residual changes in the resistance following mechanical loading can also serve as an indicator of the damage.

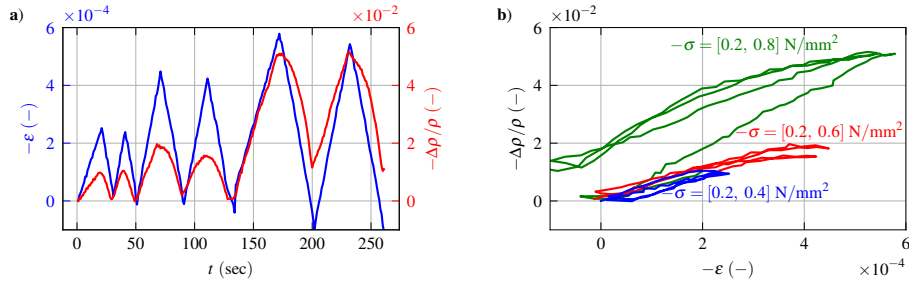


Fig. 4. Piezoresistive behaviour of G3F0T0 mortar: a) strain and relative resistivity vs. time b) strain vs. relative resistivity change.

4 Conclusions

In this paper smart lime mortars doped with three different types of conductive fillers at different filler content levels were produced and subjected to electrical and electromechanical testing. The different mortars were compared in terms of resistivity, piezoresistive sensitivity and sensing linearity in comparison with the unmodified material.

Graphite and carbon microfibres were easily incorporated and dispersed in the binder, making use of ordinary mechanical mixing equipment identical to the equipment used in mixing unmodified mortar. Thus these fillers can be readily used in large-scale applications. Conversely, carbon nanotubes were dispersed using ultrasonic homogenisation equipment, which can be prohibitive in their application in anything other than small batches of mortar.

The greatest improvement in sensitivity and linearity compared with the unmodified material was obtained by graphite. However, this is achieved for high levels of doping. Carbon nanotubes provided only a modest improvement in the piezoresistive properties of the mortar. Finally, carbon microfibres provided a maximum increase

in sensitivity roughly equal to the one obtained with G, albeit with somewhat less improved linearity, but with a very low level of doping.

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