A novel measurement system for self-sensing graphite-cement composites

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Abstract. Carbon-based conductive fillers have been incorporated into cement matrix to develop smart self-sensing materials with piezoresistive properties. However, accurately measuring the sensing property of the cement composite without compromising its mechanical performance is not easy to achieve in practical engineering. Therefore, in this study, a novel experimental setup for measuring the self-sensing properties of conductive fillers embedded cementitious composites was developed. This multi-functional measurement system is able to measure specimens under compressive and flexural stress with different loading profiles, apply various loading rates, obtain the electrical properties, and measure the strain using both LVDT and Particle Image Velocimetry (PIV) or Digital Image Correlation (DIC) techniques with all the data synchronised to one file sharing the same time stamp controlled by Python codes. In this study, the piezoresistivity and the performance on damage detection of the cementitious composites with low graphite concentration (5%) in a bulk form were investigated through monotonic compressive and flexural tests. Experiment results include the specimens' stress, strain and Fractional Change in Resistivity (FCR). Data analysis showed that the set-up and methodology developed in this study are effective to test self-sensing cementitious composites, and the graphite-cement composites used in this study have a stable piezoresistivity and able to detect damage upon failure.

1 Introduction

Carbon-based conductive fillers have been incorporated into cement matrix to develop Intrinsic Self-Sensing Concrete (ISSC), also called intrinsically smart, piezoresistive or pressure sensitive concrete. It refers to a structural material that incorporates conductive fillers such as carbon fibre (CF), steel fibre (SF), graphite, graphene and carbon nanotube (CNT) into conventional concrete which is able to monitor itself without the need of embedded, attached, or remote sensors while maintaining or improving its mechanical properties and durability [1]. Compared with conventional concrete structures which usually require additional sensors for structural health monitoring, the advantages of self-sensing concrete include good flexibility, high sensitivity, enhanced mechanical property, natural compatibility, identical durability with concrete, easy installation, and convenient maintenance [2]. Since the self-sensing concrete has both mechanical properties and sensing ability, it is not necessary to have additional sensors for monitoring purposes. In such way, this new engineering material can not only contribute to the field of structural health monitoring [3-5], but also be applied to other fields including traffic monitoring and military security [6, 7].

Graphite powder is one of the conductive fillers that is used in practical engineering due to its good conductivity and low cost [8, 9]. Due to its good electrical conductivity and multifunctionality (e.g., Joule effect, thermo-electric effect, and piezoresistive effect), the conductive cementitious composites can be widely used in the fields of intelligent buildings, health monitoring, disaster and damage prevention, etc. Previous studies have confirmed the decreased electrical resistivity of cement-based composite material after the addition or inclusion of graphite [10-12]. Generally, the addition of graphite will result in decreased compressive strength, flexural strength and bending strength of cement-based material [13-15], which can be attributed to the weak bond between graphite particles and cementitious matrix and the increased porosity of the hardened cement composites [16]. On the other hand, improvements in the fracture toughness, plastic behaviour, vibration damping ability and thermal crack-resistance ability have also been reported in previous studies [17].

Previous studies have demonstrated that graphite with small particle size exhibits good electrical conductivity and the potential to be used in cementitious composites to monitor the loading, deformation, and damage [18], in addition to its benefits on functional properties of the cementitious composites [19]. However, in this case, since comparing to the nano-scaled graphene, the graphite's particle size is still in a micro-scale, its electrical percolation threshold is attained at relatively high percentage (40 wt%) of additive in the matrix [20].

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Such high amount of hydrophobic additive has a significant impact on the rheological properties of fresh mixture and the basic mechanical properties of hardened composites become worsened [21]. These shortcomings could be overcome by using low graphite concentration, but this needs to be under the condition that the electrical performance of the composites cannot be compromised.

In this study, a novel experimental setup for measuring the self-sensing properties of conductive fillers embedded cementitious composites was developed. This multi-functional measurement system is able to measure specimens under compressive and flexural stress with different loading profiles, apply various loading rates, obtain the electrical properties, and measure the strain using both LVDT and Particle Image Velocimetry (PIV) or Digital Image Correlation (DIC) techniques with all the data synchronised to one file sharing the same time stamp controlled by Python codes. Moreover, the piezoresistivity and the performance on damage detection of the cementitious composites with low graphite concentration in a bulk form were investigated through monotonic compressive and flexural tests. Experiment results include the specimens' stress, strain, and Fractional Change in Resistivity (FCR). Data analysis showed that the graphite-cement composites have a stable piezoresistivity and able to detect damage upon failure.

2 Materials and methodology

This section provides a description of the materials, sample preparation, experimental set-up and procedures that were used in this study.

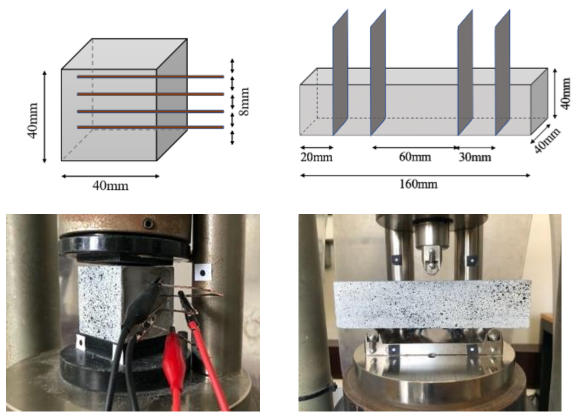
2.1 Materials and sample preparation

The materials used in this study include Portland cement and graphite. Portland cement CEMI 52.5R (rapid hardening cement) supplied by White Star Cement conforming to BS EN 197-1:2011 was used in the experiments. A commercial product of natural graphite with carbon content ⩾ 99.4%, specific surface area 12 m^2/g and particle size 18.5-29.5 μm supplied by Versarien PLC was used in this study. In this study, the cementitious composites were in form of cement paste with low graphite concentration, i.e., 5%, in a bulk form. All specimens used a w/c ratio of 0.45.

The preparation process of cementitious composites filled with graphite is as follows:1) Dry mix the graphite and cement at low speed for 3 minutes using a laboratory bench-scale mixer, Kenwood 1500 W; 2) Add water into the mixture; 3) Mix at low speed for 90 s, stop for 30 s when all the paste adhering to the wall and bottom part of the bowl was removed and placed in the middle of the bowl, and then restart the mixer at low speed for further 90 s; 4) Pour the mixture into the moulds and put the mould with the mixture on the vibrating table for 30 s to eliminate bubbles; 5) Incorporate electrodes in the mixture; 6) Demould after 24 hrs; 7) Cure the specimens for 28 d separately in a water tank at a temperature of 20±2 °C. Three specimens in each group were casted for piezoresistivity tests.

2.2 Experimental procedures

The electrical resistivity of the specimens was measured by the four-probe method with DC current and embedded electrodes [22]. In this set up, the outer two electrodes were used to supply the electric current flowing through the entire sample and the inner two electrodes were used to measure the corresponding voltage across a given sample length (Fig. 1). Cubic specimens with dimensions 40×40×40 mm for compressive tests and prismatic specimens with dimensions 40×40×160 mm for bending tests were prepared and all the tests were undertaken after 28 days' water curing to establish the effect of hydration on the electrical resistivity performance. For cube specimens, the electrodes were equally spaced and made of copper wire with a diameter of 1 mm; for prism specimens, the electrodes were made of a perforated steel sheet of a thickness 0.55 mm and hole size 3 mm supplied by RS Components.



(a) (b)

Fig. 1. Four-probe electrodes arrangement of (a) cube and (b) prism specimens.

The specimen's electrical resistance was calculated using Ohm's law:

$$R = \frac{V}{I} \tag{1}$$

where I is the electrical current, V is the measured voltage and R is the electrical resistance.

Electrical resistivity ρ was calculated by equation 2 below:

$$\rho = \frac{RA}{L} \tag{2}$$

where L is the length between the inner electrodes and A is the cross-sectional area.

Fractional Change in Resistivity (FCR) was then calculated following equation 3 below:

$$FCR = \frac{\Delta\rho}{\rho} \times 100\% \quad (3)$$

The stress sensitivity (S) against electrical resistivity was defined as:

$$S = \frac{\Delta\rho/\rho}{\Delta\sigma/\sigma} \quad (4)$$

where σ is the stress.

To study the piezoresistive behaviour of the graphite-cement composites, compressive tests were conducted, over which the electrical performance and strain of the specimen were continuously recorded by the four-probe method and Particle Image Velocimetry (PIV) technique. The compressive stress was calculated as:

$$f_c = \frac{P}{A} \quad (5)$$

where P is the compressive load and A is the cross-sectional area of the sample.

For three-point bending tests, BS EN 196-1 (2016) was followed for all the tests. The flexural stress was determined based on BS EN 196-1 (2016) following equation 6 below:

$$R_f = \frac{1.5 \times F_f \times l}{b^3} \quad (6)$$

where R_f is the flexural stress, b is the width of the sample in mm, F_f is the load that is applied to the middle of the prism and l is the distance between the supports.

2.3 Particle Image Velocimetry

The Particle Image Velocimetry (PIV) or Digital Image Correlation (DIC) method is an image-based deformation measurement technique which combines a range of advances in image analysis algorithms and techniques best suited to engineering applications [23]. This technology has been used in this study as an important step to analyse the strain and crack development under loading, as well as the relationship between the strain, stress, and electrical resistance of the specimen.

As part of the PIV preparation process, to create adequate texture on the surface of the specimen for strain calculation, the specimen was painted white with black points added on top by knocking a brush full of black paint (Fig. 1). After that, a raspberry pi camera was placed in front of the specimen. Calibration needs to be carried out by setting calibration points on the testing equipment to convert measurements from image space to real movements on the front plane of the sample. In a typical PIV–DIC analysis, a region of interest (RoI) is first defined within the initial ("reference") image of the model and populated with a mesh of subsets (or "patches") of user- defined size. The specific software used for this study is referred to as GeoPIV-RG [24] and is an update of the GeoPIV program (which represents the typical algorithms currently used in research and is described by White et al. [25]).

3 Results and discussions

Recent investigations show that it is possible to produce smart concrete capable of self-monitoring and self-sensing when suitable additives are added [1-4]. To further delineate the piezoresistive effects of graphite-cement composites, particularly its ability of damage detection and performance of self-sensing, a comprehensive investigation on such materials under compression and tension has been carried out in this section.

3.1 Monotonic compressive tests on graphite-cement specimens

To verify the relationship between the compression and electrical performance, compressive tests on graphite-cement bulk composite cubes with embedded electrodes were carried out at a loading rate of 2400 N/s in this section. Figure 2 presents the fractional change in resistivity (FCR) - stress curve of the three tested cubes.

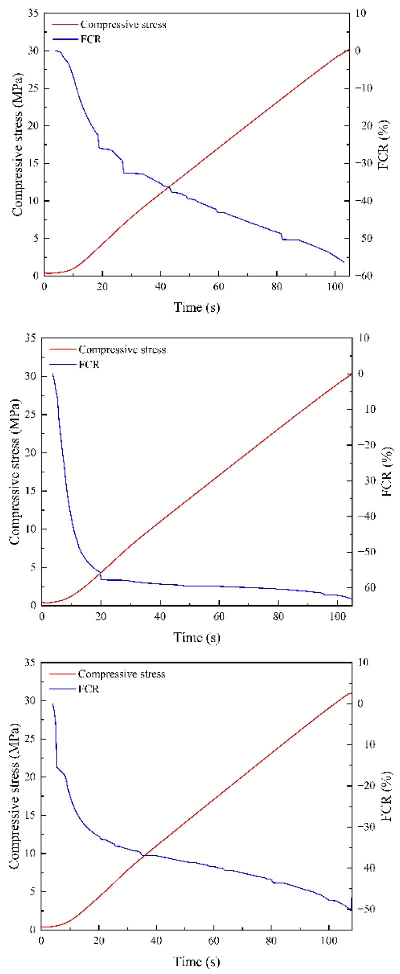


Fig. 2. Three piezoresistive tests on cubes with 5% graphite under compression.

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According to figure 2, all three cubes' electrical resistivity became smaller under compression, which fitted the theory - the distance between conductive fillers (graphite in this case) became shorter under compression, which made the sample more conductive and its electrical resistivity smaller. Up to -60% FCR was detected during the tests, which contributed to an average compressive stress sensitivity of 1.9 %/MPa. Such stress sensitivity is higher than a 1-10 wt% carbon black (CB) cementitious composite with a stress sensitivity of 0.17-0.29 %/MPa [26]; similar to a composite containing 1-2 wt% graphene nanoplates (GNPs) which has a stress sensitivity of 1.47-1.83 %/MPa [27]. Figure 3 presents the PIV results of one of the above-mentioned specimens presenting the linear strain in y direction, which shows the crack development of the cube till its final failure.

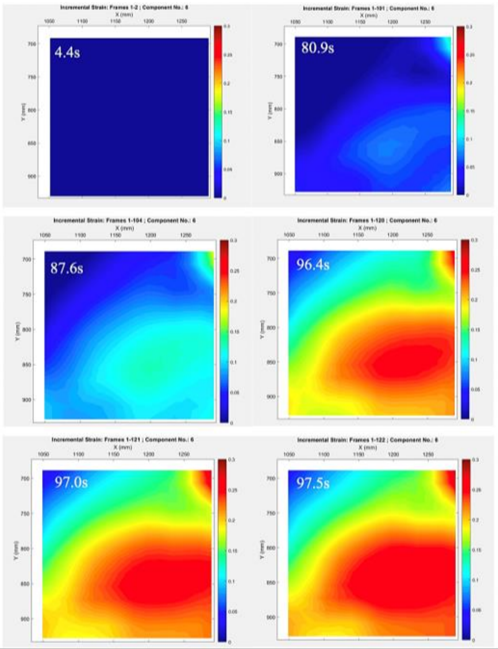

Fig. 3. PIV results of surface strain distribution of specimen over the tests under compression

3.2 Three-point-bending tests on graphite-cement specimens

To investigate the piezoresistive performance of graphite-cement composites under flexural stress, three-point-bending (3PB) tests at a loading rate of 25 N/s were conducted on graphite-cement bulk prisms, during which the stress, electrical performance and strain of the specimens were continuously recorded using the four-probe method and Particle Image Velocimetry (PIV). Figure 4 presents the test results on three 5% graphite embedded specimens, according to which, all the specimens' electrical resistivity increased with increasing flexural stress, which is as expected, i.e., under bending, the bottom of specimen was under tension which caused an increase in the resistivity. Up to 18% FCR was detected during the tests, which contributed to an average flexural stress sensitivity of 3.1 %/MPa. Figure 5 shows the PIV results of the linear strain in x direction, which shows the crack development of the prism till its final failure.

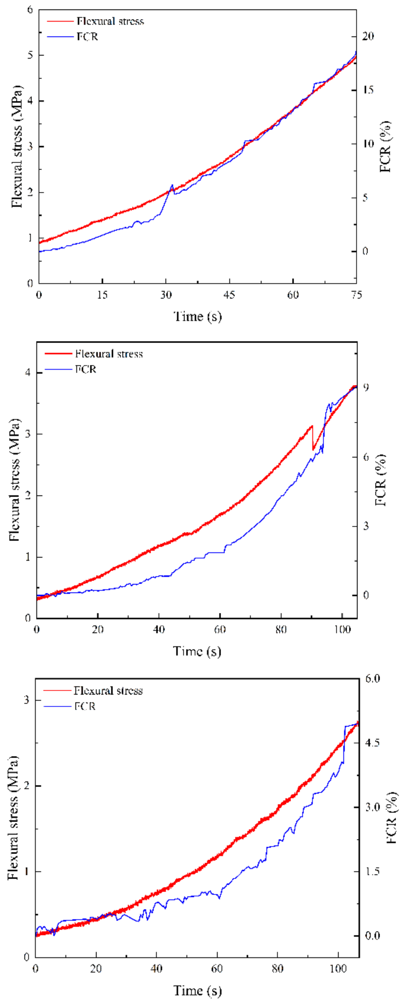

Fig. 4. Three piezoresistive tests on cubes with 5% graphite under bending.

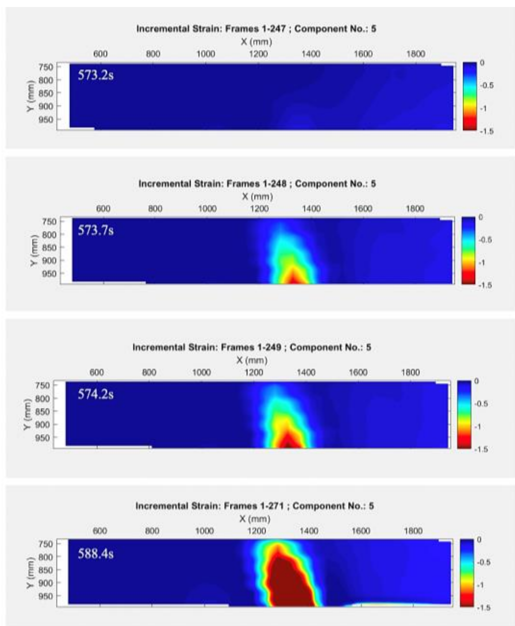

Fig. 5. PIV results of surface strain distribution of specimen over the tests under three-point bending

4 Conclusions

In this study, the piezoresistivity and the performance on damage detection of the cementitious composites with low graphite concentration (5%) in a bulk form were investigated through monotonic compressive and flexural tests. Experiment results show a decrease on the FCR of up to 60% for compressive tests, and an increase of up to 18% for flexural tests upon failure. The average stress sensitivities of the composites are 1.9 %/MPa for compression and 3.1 %/MPa for tension.

Data analysis showed that the experimental set-up and testing methods developed as well as the PIV technology applied in this study are effective to test self-sensing cementitious composites. The graphite-cement composites used in this study have stable piezoresistivity and satisfactory sensitivity, and are able to detect damage upon failure.

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