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Self-sensing performance of cementitious composites with functional fillers at macro, micro and nano scales

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ABSTRACT

Self-sensing cementitious composites have attracted substantial attention as a multifunctional construction material for structural health monitoring (SHM). This study aimed to develop self-sensing cementitious composites using a combination of macro, micro and nanoscale conductive fillers, as the hybrid fillers can take advantage of different types of conductive fillers and create a synergistic effect. Magnetite aggregate (MA), carbon fibre (CF) and carbon nanotube (CNT) were combined and used as the conductive fillers for the fabrication of self-sensing cementitious composites, where the mechanical properties, electrical properties and piezoresistive performance were studied. The MA at 100 wt% achieve the optimal mechanical properties, leading to a 5% increment in compressive and a 25% increment flexural strength with a value of 37.3 and 5.7 MPa. Additionally, multiple reinforcing effects were achieved when combining different types of functional fillers, which a single filler cannot achieve. The best conductive filler combination is MA, CF and CNT hybridisation, each at 100, 0.3 and 0.05 wt%, respectively. A 17% improvement in terms of compressive strength can be observed. And the piezoresistive response can achieve a maximum fractional change in resistivity of 44.7% and demonstrates enhanced linearity, repeatability, signal to noise ratio and stability.

1. Introduction

Structural health monitoring (SHM) can provide real-time monitoring of the structural performance and predict the service life of the structures, which is essential, especially for those infrastructures under the threat of aggressive environments [2]. Self-sensing cementitious composites can be used for future smart building development and fulfil the demand for SHM [1].

The self-sensing cementitious composite is a highly complex structure, which achieves the self-sensing capacity through the incorporation of conductive fillers. The conductive fillers can induce the piezoresistive effect of the cementitious composites, refer to the phenomenon of electrical resistance changes react to external stress [3,4]. Apart from the conductive filler, the self-sensing cementitious composites own multiphase includes the filler phase, matrix phase and the interface between fillers, which all affect the quality of piezoresistive behaviour of the composites [5].

The self-sensing cementitious composites exhibit an enhanced mechanical bonding and durability, compared with the traditional SHM technique [6,7]. However, some challenges include the high costs and dispersion of the conductive fillers, which have limited the scalability and the potential application of self-sensing cementitious composites [8]. To address these issues, hybrid fillers can take advantage of different types of conductive fillers and create a synergistic effect on electrical performance. In addition, the hybrids filler in different forms could achieve an enhanced mechanical capacity and reduce the required amount of fillers [9].

Common conductive fillers include steel fibre (SF) [10,11], carbon fibre (CF) [12,13] and graphite powder (GP) [14,15] have been widely investigate. SF at 0.36 vol% can achieve a gauge factor (defined as the ratio of the fractional change in electrical resistivity (FCR) to the fractional change in strain) of 720, which is 2 times greater than CF [10]. However, the corrosion of SF can block the conductive path with the increasing time of curing [16]. Among the conductive fillers in micro-scale, CF is one of the most effective conductive fillers. Due to its high conductivity and large aspect ratio, it can easily form the conductive network within the cement matrix and has been widely applied in self-sensing cementitious composites. CF could be selected as the ideal micro-scale conductive fillers [17]. Fan et al. [14] combined the fibrous filler (CF) with the particle fillers (GP) and achieved a piezoresistive

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Table 1
Chemical composition and physical properties of cementitious materials.

OPC		GGBFS		SF	
CaO	63.40%	FeO	1.3%	Silicon as SiO_2	98%
SiO_2	20.10%	CaO	38–43%	Sodium as Na_2O	0.33%
Al_2O_3	4.60%	SiO_2	32–37%	Potassium as K_2O	0.17%
Fe_2O_3	2.80%	Al_2O_3	13–16%	Available Alkali	0.40%
SO_3	2.70%	MgO	5–8%	Chloride as Cl^-	0.15%
MgO	1.30%	TiO_2	1.5%	Sulphuric Anhydride	0.83%
Na_2O	0.60%	MnO	0.5%	Sulphate as SO_3	0.90%
Total Chloride	0.02%	Hydraulic Index	1.7–1.9%		
Physical Properties		**Physical Properties**		**Physical Properties**	
Specific Gravity	3.0–3.2 t/m^3	Bulk Density	850 kg/m^3	Bulk Density	625 kg/m^3
Fineness index	390 m^2/kg	Glass Content	>85%	Relative Density	2.21
Normal consistency	27%	Angle of Repose	Approx. 35°	Pozzolanic Activity at 7 days	111%
Setting time initial	120 min	Chloride Ion	<0.025%	Control Mix Strength	31.3 MPa
Setting time final	210 min			Moisture Content	1.10%
Soundness	2 mm			Loss of Ignition	2.40%
loss on ignition	3.80%				
Residue 45 μm sieve	4.70%				

performance with enhanced repeatability and stability, which indicated that functional filler in particle form could bridge the adjacent fibre and improve the surface bonding between fibre and the surrounding matrix. With the development of nanotechnology, nanomaterials such as carbon nanofiber (CNF) [18], carbon nanotube (CNT) [19,20], carbon black (CB) [21,22], have gathered increasing attention and have been adopted as the hybrid filler with CF [23–26]. CNT is a nanoscale fibrous material, which owns the benefits of a large aspect ratio, high conductivity and excellent mechanical performance compared to particle-shaped nanofillers [27]. In addition, CNT can enhance the mechanical strength of the cementitious composites owing to its reinforcing ability, and accelerate the cement hydrates and contribute to the calcium silicate hydrates (C-S-H) formation [28,29]. However, CNT is difficult to form a continuous conductive network due to the ease of agglomeration. Han et al. [30] adopted the hybrid fillers in the nanoscale (CNT and CB), which achieved an improved piezoresistive performance. However, the sensitivity of the piezoresistive performance needs to be further improved. Several studies have been conducted on improving the piezoresistive performance by adding CF to the cementitious composites with the incorporation of CNT [24,31,32]. The self-sensing cementitious composites should be designed based on the percolation threshold to ensure an adequate concentration of conductive fillers, which refers to the filler concentration when the adjacent fillers begin to touch one another [33]. Lee et al. [23] illustrated that the percolation threshold of CF and CNT was 0.5–1 vol% and 1 vol%, respectively. A gauge factor of 160 can be achieved by 1 vol% of CF and 0.5 vol% of CNT. The gauge factor and FCR represent the sensitivity of the self-sensing cementitious composites, which is one of the most important factors to evaluate the sensing performance. The sensing performance can also be evaluated by other factors, such as stability, linearity, repeatability and signal to noise ratio. Kim et al. [31] indicated that the combination of CNT and CF could achieve a conductive network with enhanced stability, owing to the interconnectivity of CNT. The incorporation of CNT can enhance the repeatability and accuracy of the piezoresistive response compared to the composites with CF alone [24]. Luo et al. [32] reported that a combination of 0.5 wt% of CF and 0.1 wt% of CNT can achieve a piezoresistive response with improved linearity and consistent variation toward applied stress.

Although previous research has developed self-sensing cementitious composites with a reasonable sensing performance, the performance can be further improved. The lack of aggregate in the self-sensing cementitious composite may result in autogenous shrinkage and affect the mechanical strength of the composites. The self-sensing cementitious composites generally do not incorporating aggregate to ensure the connectivity of the conductive network as the addition of aggregate may block the conductive path and adversely affect the piezoresistive performance [34]. Magnetite aggregate (MA) (Fe_3O_4) is a common mineral that owns a semi-conductive property, which has been used for electromagnetic interference shielding and has the potential to be used as the aggregate for self-sensing cementitious composite fabrication [35].

Table 2
Properties of 45–50 silica sand.

45–50 Silica Sand			
Chemical composition		**Sieve size %**	**Retained**
SiO_2	99.86%	850 μm	0%
Fe_2O_3	0.01%	600 μm	0.3%
Al_2O_3	0.02%	425 μm	11.9%
CaO	0.00%	300 μm	40.8%
MgO	0.00%	212 μm	31.60%
Na_2O	0.00%	150 μm	12.6%

The main objective of the current research is to develop a self-sensing cementitious composite that could achieve an improved piezoresistive response. Besides, the possibility of replacing natural limestone aggregate with MA in the fabrication of self-sensing cementitious composites for better sensing performance has also been evaluated. The combination effect of conductive fillers with three different scales, including macro, micro and nanoscale, were examined. The mechanical properties, electrical resistivities, and piezoresistive behaviour of cementitious composites with hybrid MA, CF and CNT were investigated. The performance and percolation threshold of mono and hybrid conductive

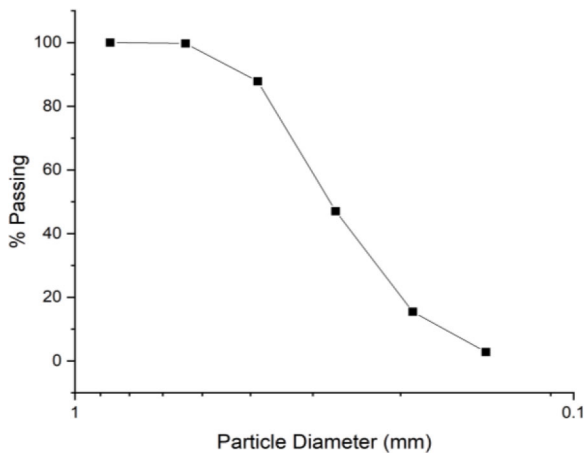


Fig. 1. The particle distribution curve of fine silica sand.

Table 3
Material properties of the functional fillers.

Magnetite Aggregate		Carbon Fibre		Multi-wall Carbon Nanotube	
Diameter (mm)	1–2	Length (mm)	3	Purity (%)	95
Chemical composition		Diameter (mm)	7×10^{-3}	Outer diameter (nm)	8–13
Al (%)	0.9	Aspect ratio	428.6	Length (μm)	50–150
Ca (%)	0.1	Density (g/cm^3)	1.8	Specific gravity (g/cm^3)	2.1
Fe (%)	39.1	Electrical resistivity (Ω•cm)	1.55×10^{-3}	Specific surface area (m^2/g)	200–300
Mn (%)	0.06	Tensile strength (MPa)	4137	Wall number	>8
Si (%)	0.2	Tensile modulus (GPa)	242	PH	6–7
Ti (%)	6.2	Carbon content (%)	95		
O (%)	46				

fillers were determined. Scanning electron microscopy (SEM) analysis was also conducted to evaluate the performance and dispersion of functional filler within the matrix.

2. Materials and methods

2.1. Materials

Ordinary Portland cement (OPC) and supplementary cementitious materials such as ground granulated blast furnace slag (GGBFS) and densified silica fume (SF) were used as binder materials for self-sensing cementitious composites manufacture. The properties of the materials are summarised in Table 1, which are in accordance with AS3972, AS3582.2 and AS3583. 45–50 fine silica sand with the chemical composition and particle size distribution outlined in Table 2 and Fig. 1 was used as the fine aggregate.

Three types of functional fillers, one in macro scale, one in micro-scale and one in nanoscale, were used as the conductive functional fillers to investigate the hybrid effect of functional fillers in this study. The MA with a diameter of 1–2 mm was selected as the macro scale functional fillers due to its high iron content (see Table 3, which is believed to be effective in improving the electrical performance of the cementitious composites. Unsized polyacrylonitrile (PAN) based CF with the properties outlined in Table 3 was selected as the micro filler. MWCNT was incorporated as the nanoscale filler, which is designed to create the interconnection between CF. The SEM images of the functional fillers used in this study are shown in Fig. 2. The CF was not in cluster form indicating that no sizing has been applied to the surface of CF. CNT was fond in agglomerate form without any dispersing treatment. Polycarboxylate-based high range water reducing agent (HRWRA) was adopted to ensure the dispersion of the functional fillers and adjust the workability of the mixture.

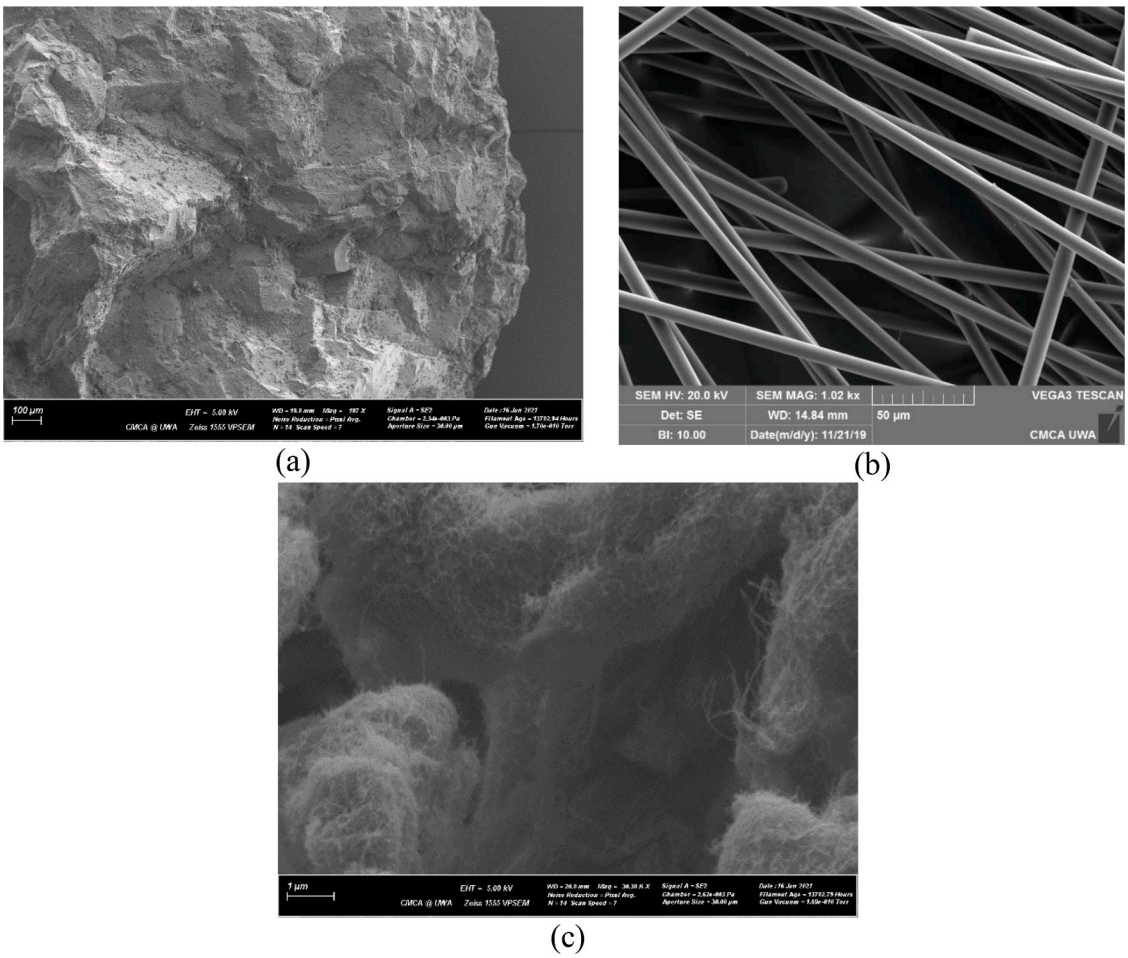


Fig. 2. SEM images of (a) magnetite aggregate, (b) CF and (c) CNT.

Table 4
Mixing proportion of self-sensing cementitious composites.

Mix	W/B ratio	Portland cement	GGBFS	Silica fume	45/50 silica sand	Filler types	Filler content (wt.% of binder)
CM	0.4	1	1.2	0.11	0.84	–	–
CF	0.4	1	1.2	0.11	0.84	CF	0.7
MA0.5	0.4	1	1.2	0.11	0.84	MA	50
MA1	0.4	1	1.2	0.11	0.84	MA	100
MA1.5	0.4	1	1.2	0.11	0.84	MA	150
MA2	0.4	1	1.2	0.11	0.84	MA	200
CNT0.05	0.4	1	1.2	0.11	0.84	CNT	0.05
CNT0.1	0.4	1	1.2	0.11	0.84	CNT	0.1
CNT0.5	0.4	1	1.2	0.11	0.84	CNT	0.5
CNT1	0.4	1	1.2	0.11	0.84	CNT	1
MA1CF0.1	0.4	1	1.2	0.11	0.84	MA/CF	100/0.1
MA1CF0.3	0.4	1	1.2	0.11	0.84	MA/CF	100/0.3
MA1CF0.5	0.4	1	1.2	0.11	0.84	MA/CF	100/0.5
MA1CF0.7	0.4	1	1.2	0.11	0.84	MA/CF	100/0.7
CF0.3CNT0.05	0.4	1	1.2	0.11	0.84	CF/CNT	0.3/0.05
CF0.5CNT0.05	0.4	1	1.2	0.11	0.84	CF/CNT	0.3/0.05
MA1CF0.3CNT0.05	0.4	1	1.2	0.11	0.84	MA/CF/CNT	100/0.3/0.05
MA1CF0.5CNT0.05	0.4	1	1.2	0.11	0.84	MA/CF/CNT	100/0.5/0.05

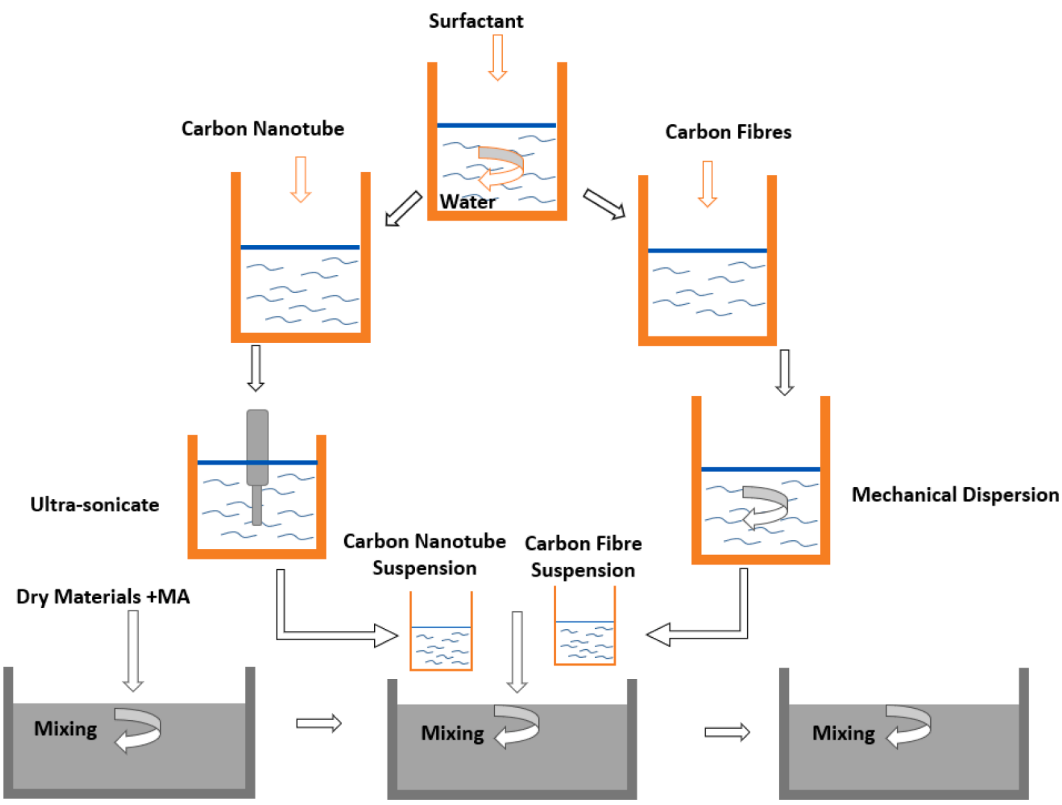


Fig. 3. preparation procedure of the self-sensing cementitious composites.

2.2. Sample preparation

An experimental programme was designed to investigate the mechanical, electrical and piezoresistive properties of self-sensing cementitious composites that contained conductive functional fillers at various scales. Table 4 summarises the mixture proportion investigated in this study. The water/binder (W/B) ratio was kept at 0.4 for all specimens. The previous study has investigated the piezoresistive behaviour of cementitious composites with CF at various concentrations, which indicated that CF at 0.7 wt% of the binder endows the optimal piezoresistive performance [13]. Therefore, the composites containing CF at 0.7 wt% and the composites without any functional fillers were examined as the controls. MA was added into the composites at 50, 100, 150 and 200 wt% of the binder to preliminary determine the concentration of MA. Then, MA was combined with CF at different concentrations to investigate the combined effect of MA and CF. Finally, CNT was added to the hybrid MA and CF mix to investigate the performance of the composites containing function fillers in three different scales. CNT was also investigated individually as a control at various concentrations to evaluate the combined effect of functional fillers.

As the first step in preparing self-sensing cementitious composites, the binder materials and fine silica sand were dry mixed for 2 min using a Hobart mixer at a low speed of 60 RPM. Preliminary treatment was applied for functional fillers before adding them into the dry materials to ensure the proper dispersion. Different mixing methods were considered based on the types of functional fillers, which were summarised in Fig. 3. The synchronous admixing method was used for mixes involving MA, where MA was first mixed with dry materials until homogenous before

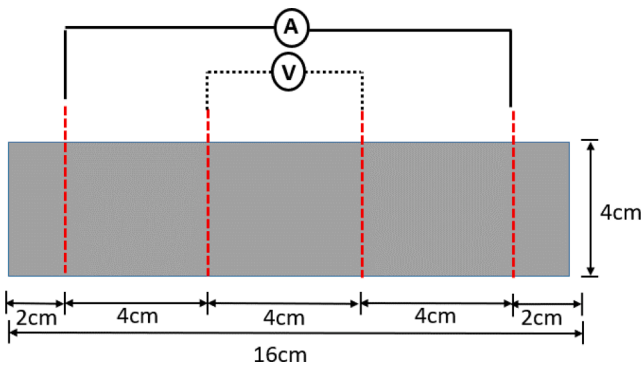


Fig. 4. Electrode configuration of electrical resistivity measurement.

adding water and surfactant. The latter admixing method was applied for mixes containing CF/ CNT, where CF/CNT need to be first mechanical dispersed in the aqueous solution prepared by HWRWA and mixing water. For hybrid fillers, a collaborative mixing process was used for sample preparation. However, CNT is shown in agglomerate from as illustrated in Fig. 1c, which is difficult to disperse properly due to the high Van der Waal force. Therefore, ultra-sonication was introduced for CNT dispersion [36,37]. CNT was added into the aqueous solution containing and sonicated by a 500 W probe ultrasonic processor for 10 min at a cycle of 20 s. The prepared functional filler suspension was then poured into the dry mix and first mixed at low speed, then moderate speed (124 RPM) until homogeneous.

After mixing, the fresh mixture was poured into the oiled moulds, and the copper meshes were embedded into the specimens prepared for electrical properties measurement. External vibration was applied for all prepared specimens to eliminate the air bubbles also enhance the bonding between copper meshes and matrix. The specimens were demoulded after hardening and kept in a moisture-controlled room with the temperature-controlled at 25℃ and humidity at 95% until the day of testing.

2.3. Mechanical properties tests

The compressive and flexural strength of hardened specimens after 28 days of curing were measured using a Baldwin testing machine and Instron 5982, respectively. The compression tests were performed on 50 mm cubes following ASTM C109 [38], and 3-point bending tests were conducted on 40 mm × 40 mm × 160 mm prisms to determine the flexural strength following ASTM C348 [39]. The compressive and flexural strength were obtained from the average testing results of three specimens to ensure an accurate value. The loading rate was controlled at 0.5 mm/min for both compressive and 3-point bending tests.

2.4. Electrical resistivity tests

Specimens were taken out from the curing room at a curing age of 28 days for electrical resistivity measurement. A digital multimeter, Keithley 2100, was used to measure the direct current resistance of the specimens. Four probe method was considered in the measurement as it can eliminate the contact resistance and provide a more accurate result. The outer two probes are used for current input, and the inner two probes are used for voltage output (see Fig. 4). It should be noted that unwanted fluctuation and continuous time drift can be observed in the measured electrical resistance induced by the polarisation effect [40]. When a direct current electric field was applied on the specimens, the negative and positive ions start moving toward the positive and negative electrodes, respectively. The movement of ions creates an internal electric field with a direction opposite to the applied direct current, which leads to an increase in the measured resistance. Samples were kept in an oven for 24 h prior to the test to eliminate the free water to reduce the effect caused by the polarisation effect, which has been found effective in reducing the polarisation.

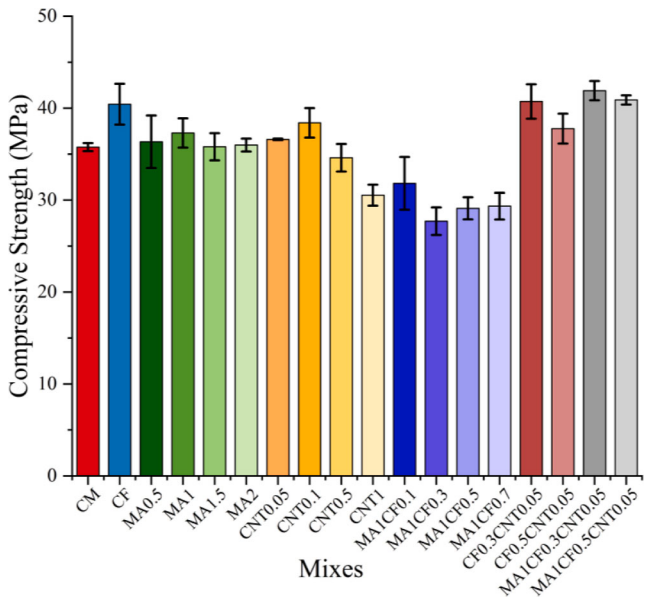


Fig. 5. Compressive strength of self-sensing cementitious composites after 28 days of curing.

2.5. Piezoresistive behaviour

The piezoresistive performance was conducted within the elastic region on the 40 mm × 40 mm × 160 mm prisms. The effect of filler types and concentration on piezoresistive performance were studied under a cyclic compression with a magnitude of 10 MPa and a loading rate of 0.5 mm/min. As the loading beyond the elastic region can lead to irreversible damage and affect the repeatability and stability of the piezoresistive behaviour, the magnitude of loading is determined based on the compressive strength within the elastic region. The cyclic compression was applied perpendicular to the embedded electrodes using Instron 5982. The resistance was continued measured during the loading process, using the digital multimeter. The relationship between the applied cyclic compression and the corresponding fractional change in resistivity (FCR) were studied to determine the piezoresistive performance.

The electrical resistivity(ρ) (Ω · cm) and the fractional change in resistivity (Δρ) (%) of the cementitious composites can be determined using the following equations (Eq. (1) and Eq. (2)).

$$\rho = \frac{\mathrm{RA}}{\mathrm{L}}\# \tag{1}$$

$$\Delta\rho = \frac{\rho_m - \rho_0}{\rho_0} \times 100\%\# \tag{2}$$

R is the electrical resistance; A is the area of the electrodes; L is the distance between the internal two probes; ρ_m and ρ_0 are the measured resistivity and initial resistivity, respectively (Ω · cm).

2.6. Microscopy analysis

A high resolution, field-emission variable-pressure (VP) scanning electron microscope, Zeiss 1555 was used for taking SEM images of the fillers and cement matrix. The samples for microscopy analysis were selected from the inner core of the compressive samples at 28 days. The samples were platinum coated to ensure a high surface conductivity, which is related to the quality of the images.

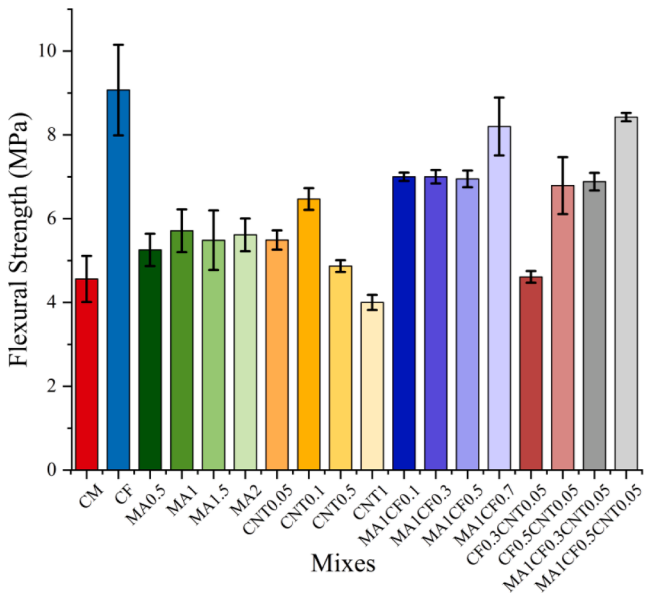


Fig. 6. Flexural strength of self-sensing cementitious composites after 28 days of curing.

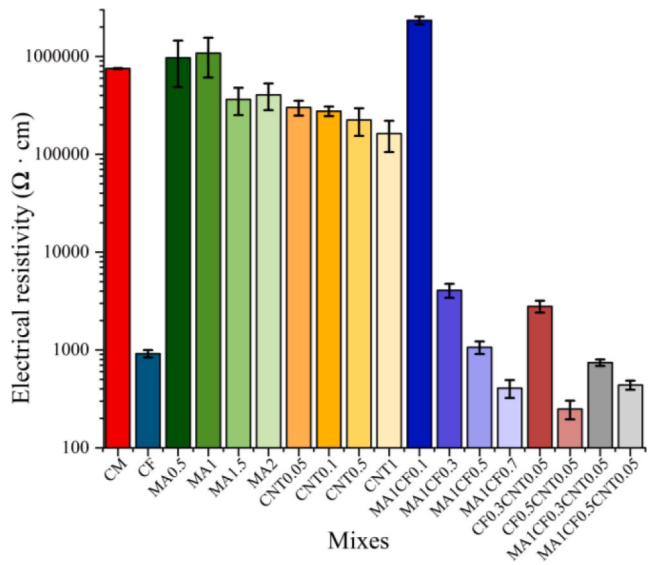


Fig. 7. Electrical resistivity of self-sensing cementitious composites after 28 days of curing.

3. Results and discussion

3.1. Mechanical properties

3.1.1. Compressive strength

The 28 days compressive strengths of self-sensing cementitious composites were presented in Fig. 5. The maximum compressive strength of cementitious composites containing MA can be achieved when the concentration of MA is at 100 wt% of the binder. The addition of MA can fill the pores and result in a denser matrix. Besides, the high strength of MA itself can create enhanced skeleton support in the matrix [41]. However, the compressive strength starts to decrease with the increasing MA concentration. This reduction in compressive strength may result from the water absorption of MA [42,43]. The high water absorption of MA can inhibit the cement hydration and the formation of calcium silicate hydrates (C-S-H), which affect the strength of the composites. Moreover, bleeding and the segregation of the MA can happen owing to the high water absorption and density of the MA, which affect the strength of the composites [44].

A similar trend can be observed in composites containing CNT. The optimal compressive strength was achieved by CNT0.1, which revealed a 7% of increment. CNT can act as individual fibres and exhibit an anchor effect within the matrix. Besides, the addition of CNT can accelerate the C-S-H gel formation and contribute to the compressive strength [45]. However, due to the large specific surface area, CNT has a high water absorption. A large content of CNT can significantly reduce the flowability of the mixture and result in a higher amount of air voids in the hardened composites, consequently affecting the compressive strength. Besides, CNT can form agglomerates at a high concentration due to the strong van der Waals force between particles, which act as the defect in the cementitious composites [46]. Moreover, the CNT fields and clumps might cover the surfaces of cement particles, which inhibit the cement hydration and hider the formation of cement hydrates [47].

The MA concentration was designed to be 100 wt% when combined with the other functional fillers. When MA was combining with CF, the compressive strength first reduced and then increased with the CF addition. The reduction can be explained by the water absorption and segregation of MA and the poor distribution of CF. However, when CF is added at a high content, the bridging effect of CF dominates and results in an increase in compressive strength. The combination of CF and CNT present an enhanced compressive strength. The CNT can enhance the bonding between CF and the surrounding matrix, thus contributing to the reinforcing effect of CF [48,49].

The optimal compressive strength was achieved for composites containing hybrid MA, CF and CNT. Both CNT and CF can prevent the formation of cracks by the bridging effects. CNT can accelerate the formation of C-S-H gel and improve the bonding between CF and surrounding matrix [45,46]. MA can fill the air voids and contribute to the density of the matrix and create enhanced skeleton support [41]. The hybridisation of functional fillers can combine the reinforcing effect of different fillers, which a single filler cannot achieve.

3.1.2. Flexural strength

Fig. 6 shows the flexural strength results of the developed cementitious composites. The addition of CF significantly increases the flexural strength of the composites, which was 100% higher than the control mix. MA also presented a slight increase in flexural strength when the concentration is at 100 wt%. However, no enhancement can be observed in the flexural strength of the composites containing hybrid MA and CF, compared with composites containing CF alone. This might result from the reduced flowability of the mixture, which adversely affects the dispersion and the bonding strength of the CF. Cementitious composites containing solely CNT at 0.1 wt% demonstrate an improved flexural strength, which is attributed to the bridging effect of CNT and the improved hydration reaction caused by the CNT. In contrast, an excessive amount of CNT can lead to a severe reduction in flexural strength, which the agglomeration of CNT can explain. The obtained results indicated that a high flexural strength could also be achieved at a lower CF addition when combined with the CNT. The CNT attached on the surface of CF could enhance the interface between the CF and the cement hydrates by improving the mechanical interlocking between CF and matrix [50]. Besides, the attached CNT could form the additional nucleation sites, which contribute to the formation of C-S-H gel on the surface of CF [51]. The formation of C-S-H gel can improve the hydrophilia and roughness of the CF surface, thus contributing to the bonding strength of CF [49].

3.2. Electrical resistivity

Fig. 7 depicts the electrical resistivity of self-sensing cementitious composites at 28 days of curing. CF is most effective in reducing the electrical resistivity of the composites among all the functional fillers. Besides, MA and CNT can also improve the electrical conductivity of the

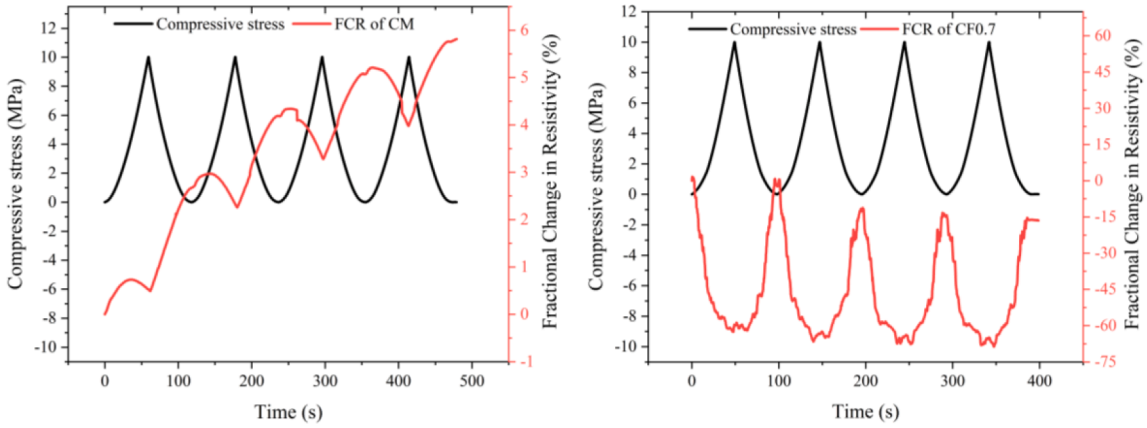


Fig. 8. Piezoresistive response of (a) control mix and cementitious composites with 0.7 wt% of CF.

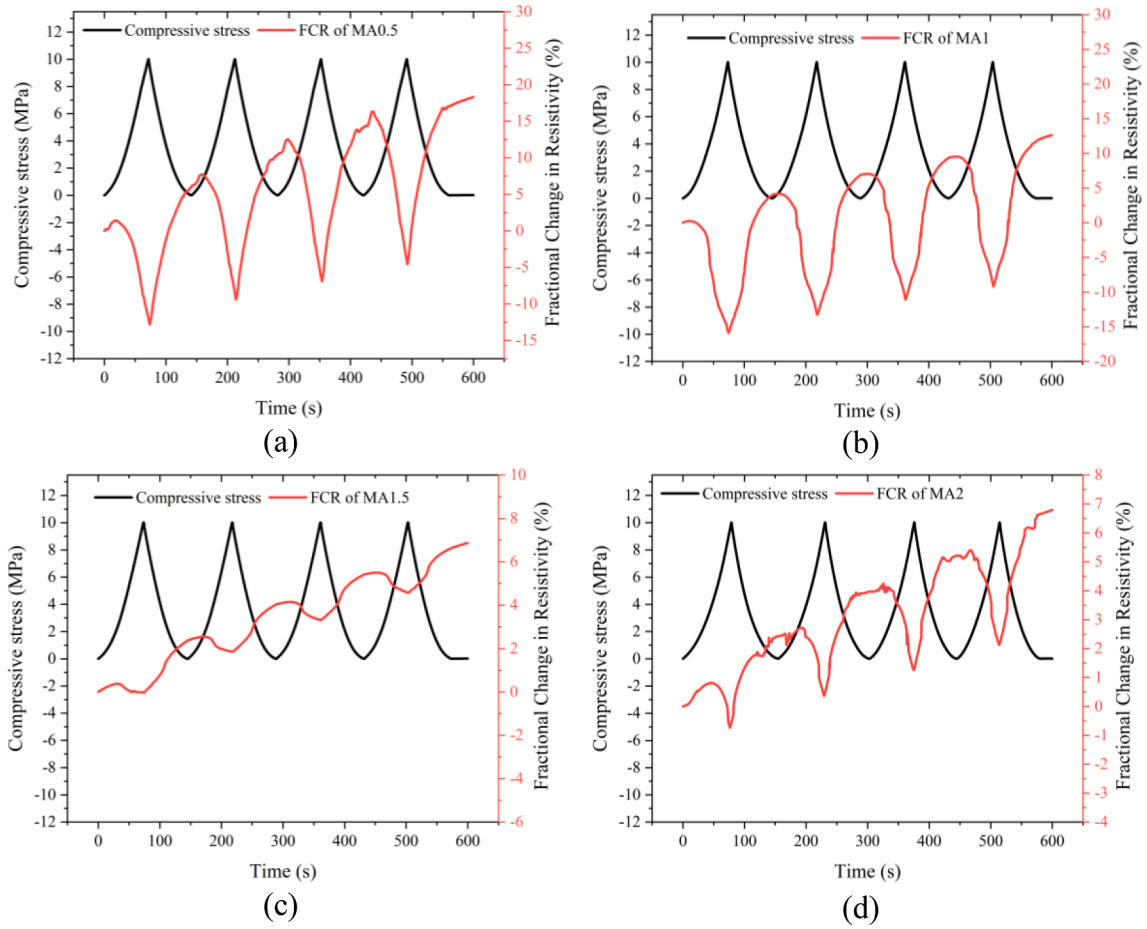


Fig. 9. Piezoresistive response of cementitious composites with (a) 50, (b) 100, (c) 150 and 200 wt% MA under cyclic compression.

composites owing to the high conductivity of the functional fillers. However, the electrical resistivity of composites containing MA first increase and then reduces with the MA addition. The increase in electrical resistivity is attributed to the pore filling effect of the MA. MA can densify the cementitious composites by filling up the pores, resulting in higher electrical resistivity, consistent with the compressive strength. The combination of MA and CF can lead to a low resistivity, as MA can bridge the adjacent CF and contribute to the formation of the conductive networks. Hybrid CF and CNT exhibit a synergistic effect, which can be explained by the obtained electrical resistivity. When the CNT at 0.05 wt % is incorporated into the composites together with CF at 0.5 wt%, the composites can achieve much lower resistivity compared to the composites containing mono CF at 0.7 wt%. For the composites containing MA, CF and CNT, MA1CF0.3CNT0.05 presents an electrical resistivity lower than CF0.3CNT0.05, which indicated the combination of three different types of functional fillers could contribute to the conductive network. However, MA1CF0.5CNT0.05 exhibit an electrical resistivity slightly larger than CF0.5CNT0.05. This lightly increases in resistivity may owing to the excess amount of overall functional fillers. All three types of functional fillers have a water absorption effect, leading to poor workability of the mixture and increasing the number of air voids in the specimens. The existence of the air voids can block the conductive path

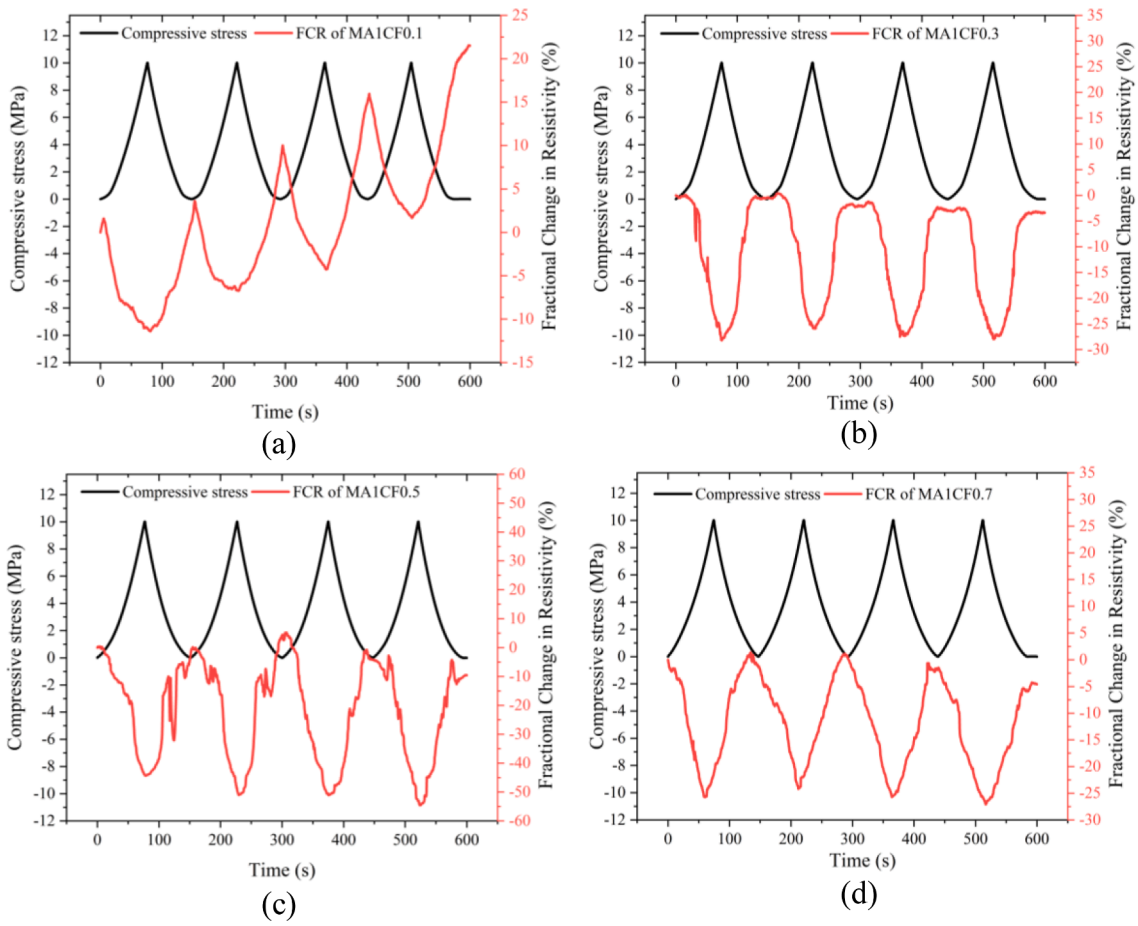


Fig. 10. Piezoresistive response of cementitious composites with MA and (a) 0.1, (b) 0.3, (c) 0.5 and 0.7 wt% CF under cyclic compression.

and consequently increase the electrical resistivity.

3.3. Piezoresistive behaviour

Figs. 8–12 show the piezoresistive response of cementitious composites with different types of fillers. The FCR was represented by the vertical axis on the right-hand side, and the compressive stress was represented by the vertical axis on the left-hand side. The FCR obtained from all tested specimens various synchronously with the applied cyclic compression. The FCR is reduced during the loading phase and increase during the unloading phase.

3.3.1. Piezoresistive response of composites containing magnetite aggregate

The FCR of both the control mix (Fig. 8a) and composites containing MA (Fig. 9) demonstrate a noticeable time drift, which may result from the polarisation effect [52]. When a continuous DC electrical field was applied, the positive ions started moving toward the negative electrode, and the negative ions started moving toward the positive electrode. The movement of ions generated a polarization induced electric field within the composites, which has the direction opposite to the applied electric field. The generated electric field will lead to a continuous increase in the electrical resistivity of the composites. No continuous conductive networks formed within the cementitious matrix for control mix, and the existence of MA showed less contribution toward the conductivity, the polarisation effect can still be observed. Composites containing MA at 50 and 100 wt% of the binder can achieve an FCR of around 25%. MA1 performed a sensing behaviour with a smoother piezoresistive curve, which exhibits a less noise ratio. The piezoresistive performance with the increasing amount of MA has been adversely affected by the poor workability and large numbers of air voids caused by the excessive amount of MA. Although the effect of polarization cannot be neglected, composites containing MA (Fig. 9) demonstrate an enhanced piezoresistive response by comparing with the control mix (Fig. 8a). The conductivity of the composites needs to be improved to reduce to polarisation effect on the sensing performance, as the composites with high conductivity are less likely to retain the internal electric field. As indicated in Fig. 8b, no polarisation can be observed, as the continuous conductive networks have been developed within the matrix. This is consistent with the result of the electrical resistivity described in section 2.4, as the percolation threshold of CF is at 0.7 wt% [13]. The composite can achieve optimal sensitivity when the conductive filler is incorporated into the cementitious matrix with the concentration at the percolation threshold. Therefore, 70% of FCR can be observed for CF0.7. However, the initial FCR of each loading cycle has been reduced with the increasing number of loading cycles. This reduction affects signal repeatability, which may result from the deformation and debonding of CF. The bonding strength of CF should be improved to achieve high signal repeatability.

3.3.2. Piezoresistive response of composites containing hybrid magnetite aggregate and carbon fibre

MA with an amount of 100 wt% of the binder was selected to be combined with CF at various concentrations, and the piezoresistive performance of composites containing hybrid MA and CF was summarised in Fig. 10. FCR of around 25% can be achieved by MA1CF0.1 (Fig. 10(a)), which is similar to MA1. Besides, a significant polarisation can also be observed. This indicated that the addition of 0.1 wt% of CF is inadequate to form the continuous conductive networks. The sensitivity of the piezoresistive response of the composites first increased and then reduced with the increasing CF addition. The maximum FCR with a

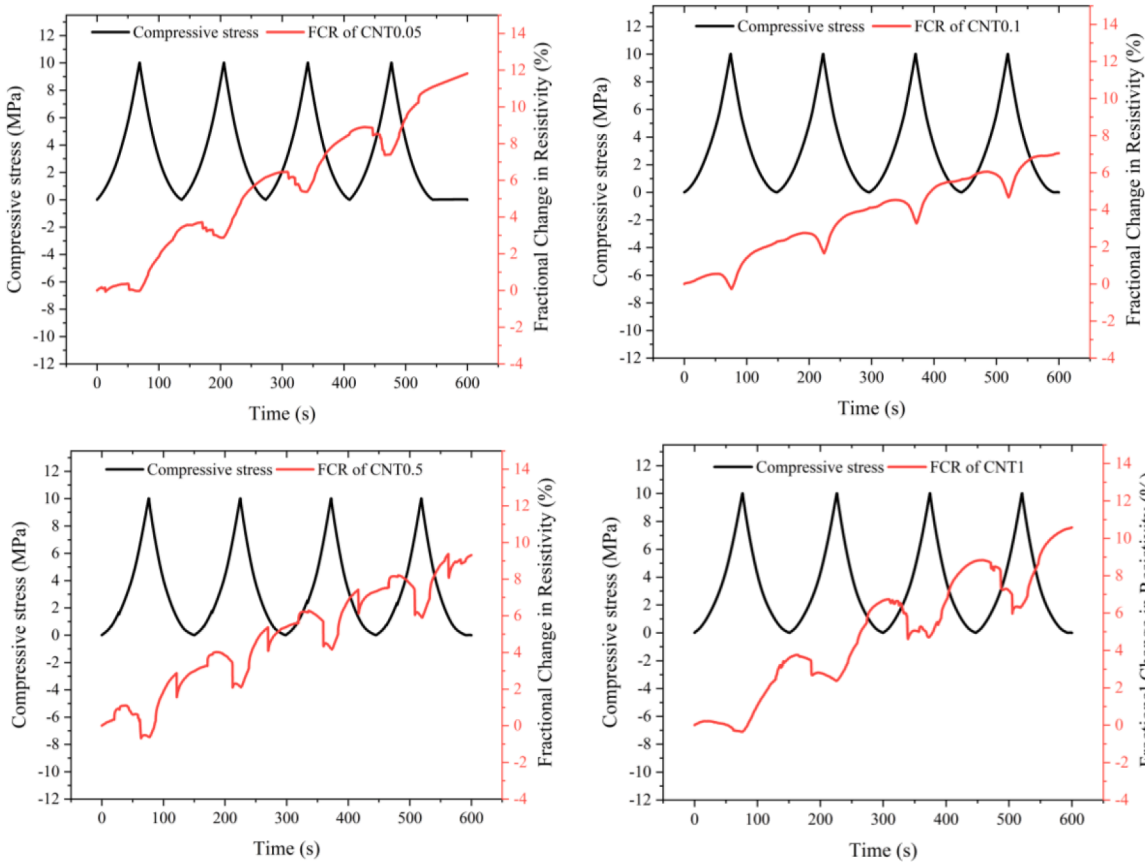


Fig. 11. Piezoresistive response of cementitious composites with (a) 0.05, (b) 0.1, (c) 0.5 and 1 wt% CNT under cyclic compression.

value of around 55% was reached when the CF addition was at 0.5 wt%. When the concentration of CF further increases, a stable conductive network forms, which is difficult to deform when subjected to external loading and consequently resulting in a reduction in the sensitivity. This result indicated that the optimal CF concentration could be between 0.3 and 0.7 wt%. The highest sensitivity can be achieved with less CF incorporation, indicating that MA can contribute to the formation of conductive networks. Besides, the MA can fill the pores and result in a denser matrix, contributing to the bonding strength between CF and the surrounding matrix, consistent with the slight improvement in composite strength. The repeatability and linearity of the piezoresistivity response for composite containing hybrid MA and CF have been found to be improved in comparison with the composite containing CF alone because of strong bonding strength (Fig. 8b). Also, a slight reduction at the initial FCR of each loading cycle can still be observed with increasing loading cycles, which can be further improved. The composites containing MA at 100 wt% hybrid with CF at 0.3 and 0.5 wt% were selected for further improvement.

3.3.3. *Piezoresistive response of composites containing carbon nanotube*

CNT was selected to combine with the MA and CF due to its high conductivity and inherent piezoresistive characteristics [53]. The piezoresistive response of composites containing CNT at 0.05, 0.1, 0.5 and 1 wt% was summarised in Fig. 11. When CNT was incorporating at 0.05 and 0.1 wt%, lightly improvement in sensitivity can be observed. However, there is no improvement in polarisation, indicating that although CNT can slightly improve the piezoresistivity and conductivity of the composites, the number of CNT is inadequate to form the conductive path. When CNT was added at a larger dosage, the CNT tended to form agglomeration within the matrix, and the signal noise of the piezoresistive response was also found to increase. The CNT agglomerate can deform under the external loading; however, this deformation is irreversible, and the CNT agglomerate cannot recover to its original form, which also results in a poor piezoresistive response [54]. Besides, the increasing amount of CNT can significantly reduce the workability of the fresh mixture and result in increasing numbers of air voids within the matrix, consequently leading to significant signal noise. In addition, polycarboxylate-based HWRWA was used as the surfactant. Although it can contribute to the CNT dispersion, the existence of surfactant between CNT can wrap the surface of CNT and block the contact between CNT [55].

3.3.4. *Piezoresistive response of composites containing hybrid magnetite aggregate, carbon fibre and carbon nanotube*

Fig. 12a and b present the piezoresistive response of composites containing hybrid MA, CF and CNT with different CF addition. Compared with MA1CF0.3, the additional 0.5 wt% of CNT has led to an increase in FCR (Fig. 12a). Besides, the piezoresistive response also demonstrates enhanced linearity, repeatability, signal to noise ratio and stability. This indicated that the CNT is able to contribute to the conductive network and prevent the CF from debonding when it is well dispersed within the matrix. However, the maximum FCR has been found to be reduced when CNT is combining with MA at 100 wt% and CF at 0.5 wt% by comparing with MA1CF0.5. This indicates an excess amount of functional fillers within the matrix when CNT was added into the mix. As can be observed, the quality of the piezoresistive response with the additional CNT has slightly improved compared with MA1CF0.5. This proved that the CNT could result in a more stable conductive network. However, due to the excess amount of functional filler, the CNT dispersion has been adversely affected, and CNT can form agglomerates within the matrix. These agglomerates can deform when subjected to an external load and result in a change in electrical resistivity. However, this deformation is irreversible, and it is difficult for them to recover to their original form, which results in poor

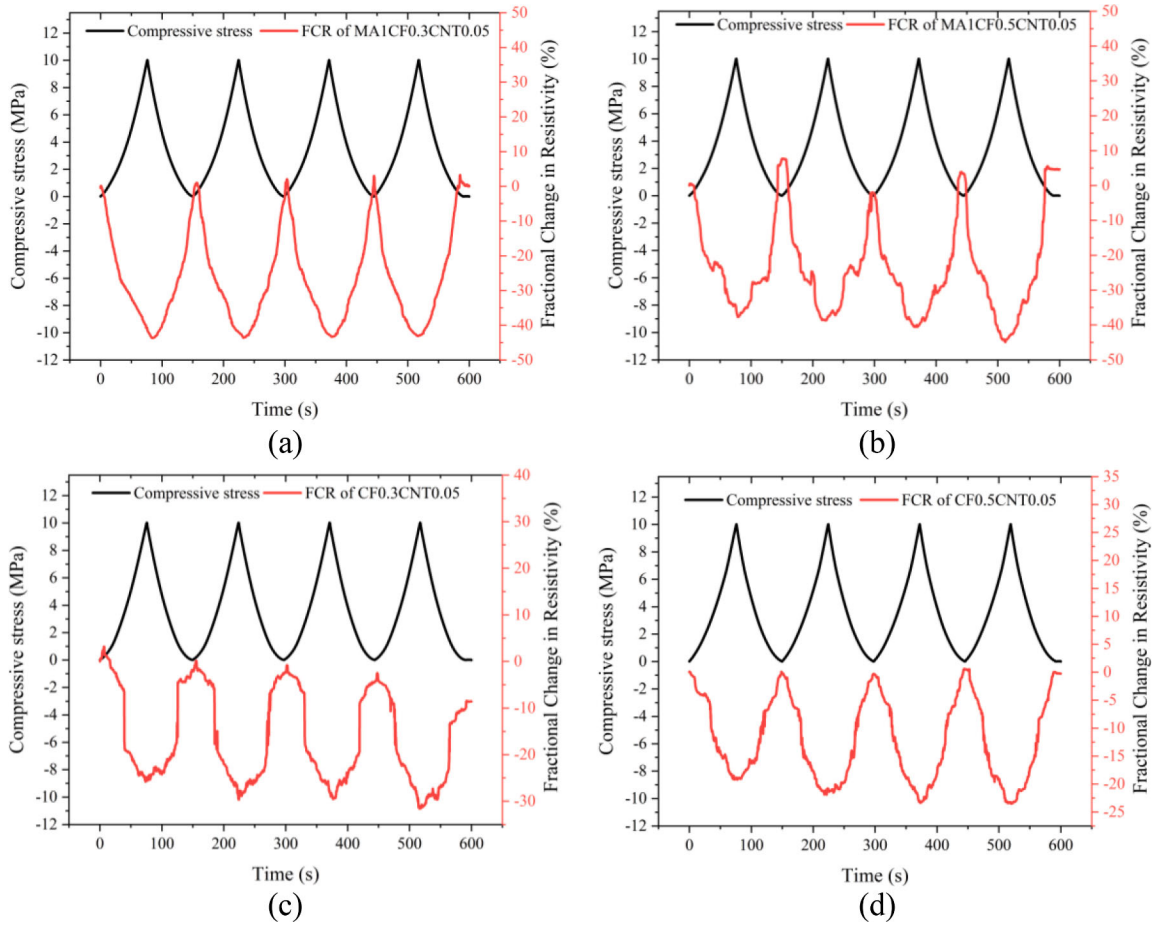


Fig. 12. Piezoresistive response of cementitious composites with hybrid MA, CF and CNT under cyclic compression; (a) composites with 100% of MA, 0.3 wt% of CF and 0.05 wt% of CNT; (b) composites with 100% of MA, 0 0.5 wt.% of CF and 0.05 wt% of CNT; (c) controls with 0.3 wt% of CF and 0.05 wt% of CNT; and (d) controls with 0.5 wt% of CF and 0.05 wt% of CNT.

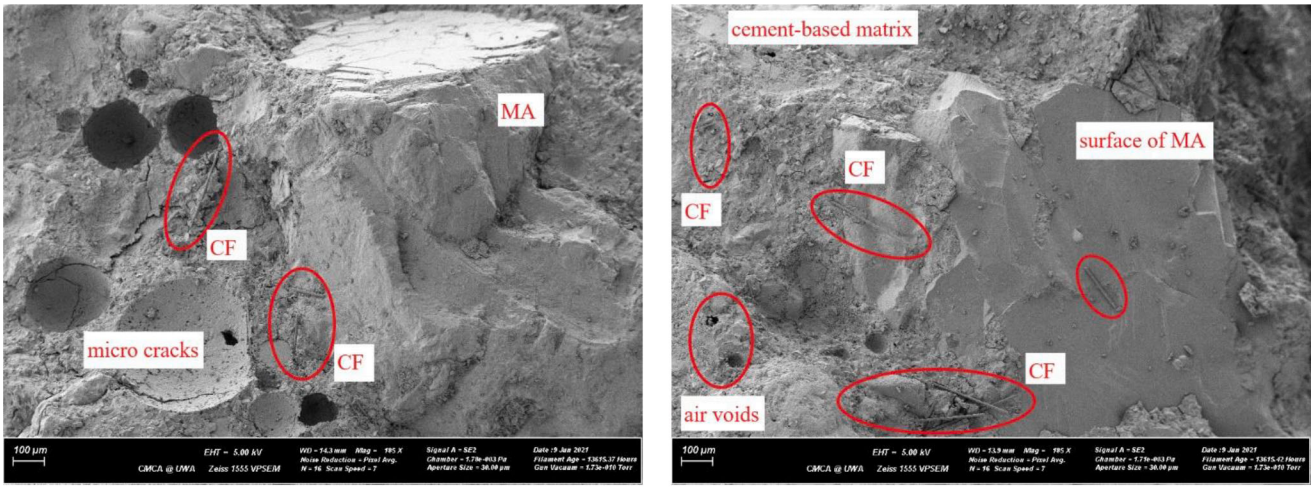
Table 5
Literature of piezoresistive performance of cementitious composites.

Ref.	Filler type	Filler content	Matrix	Compressive stress (MPa)	FCR (%)	FCR/stress	Gage Factor (FCR/strain)
[23]	CF	0.5 vol%	Cement paste	12	72	6	405.3
	CF and CNT	0.1 and 0.5 vol%			25	2.08	160.3
[14]	CF and GP	1 and 20 wt%	Cement mortar	6.25	1.6	0.26	63
[30]	CNT/CB (40/60)	6 wt%	Cement paste	10	23.2	2.32	225
[57]	CNT	0.05 wt%	Cement paste	14	–	–	189
	CNF	0.1 wt%			–	–	228
[19]	CNT	0.1 wt%	Cement paste	4	4.5	1.13	–
		0.3 wt%			1.4	0.35	–
	CNF	0.1 wt%			5.3	1.33	–
		0.3 wt%			1.9	0.48	–
[59]	CNT	0.25 wt%	Cement paste	8	14.5	1.81	–
		0.5 wt%		10	12.5	1.25	–
[56]	GNP	6 vol%	Cement paste	20	15	0.75	–
[58]	GNP	3 wt%	Cement paste	10	30	3	–
	Graphite plate	3 wt%			15	1.5	–
This study	MA, CF and CNT	100, 0.3 and 0.05 wt%	Cement mortar	10	44.7	4.47	–

repeatability [54]. Fig. 12c and d present the piezoresistive behaviour of composites containing hybrid CF and CNT, which were designed as the control groups to determine the effect of MA. The composites can only achieve an FCR at around 25%, indicating the conductive network has not been well established with the absence of MA. Besides, poor repeatability and significant signal noise can be observed in Fig. 12c, resulting from the loose bonding between matrix and functional filler.

The hybridisation of MA, CF and CNT achieve the optimal piezoresistive performance. The hybrid functional fillers can combine the effect of different types of conductive fillers and achieve an improved property, which cannot be achieved by a single filler. CNT can contribute to the conductive network and prevent CF from debonding. MA could connect the adjacent functional filler, which contributes to the conductive network formation. The MA can fill the air voids and reduce the possibility of the conductive network been blocked by the air voids. Besides, MA could enhance the performance of CF by contributing to the matrix density.

Table 5 summarised the piezoresistive behaviour in other



(a) (b)

Fig. 13. SEM images of cementitious composites containing MA and CF.

(a) (b)

Fig. 14. SEM images of composites containing CNT at 0.05 wt%

experimental studies of the self-sensing cementitious composites. In this study, the maximum FCR was achieved by CF at 0.7 wt% with a value of 70%. Besides, although the additional CNT can contribute to the piezoresistive performance in terms of repeatability and linearity, the sensitivity can be reduced. These findings are inconsistent with Lee et al. [23], where an FCR of 72% can be achieved by 0.5 vol% of CF. However, the FCR was reduced to 25% by the hybridisation of CF and CNT. The nanoscale conductive fillers such as CNT, CNF, GNP and graphite plate exhibit piezoresistive characteristics; however, the sensitivity evaluated by the FCR is much smaller compared with CF [19,56–59]. This indicated that CF is one of the most effective conductive fillers, which can achieve a good FCR at a low concentration. The previous literature indicated that the hybridisation of conductive fillers in a different form could improve the quality of the sensing performance, the sensitivity of the signal can be further [14,30]. In this research, the optimal piezoresistive performance was achieved by combine 100 wt% of MA, 0.3 wt% of CF and 0.05 wt% of CNT. A piezoresistive performance with an FCR of 44.7 %, good linearity, repeatability and stability can be achieved when subjected to external compressive stress at 10 MPa.

3.4. Microscopy analysis

Fig. 13 shows the SEM images of cementitious composites containing hybrid CF and MA. CF has been found to be embedded within the cement matrix, which is able to bridge the cracks and inhibit the growth of the cracks. As shown in SEM images, MA shows a smooth surface; however, no cracks and gaps can be observed between the MA and surrounding matrix, proving that the MA has strong compatibility with the cement matrix. Moreover, MA can bridge adjacent CF, indicating that MA can connect the CF and contribute to the conductive path formation. Therefore, a continuous conductive path can be achieved with CF at a lower concentration when MA is incorporating in the mix. However, air voids can be observed within the matrix as both the MA and CF performed the water-absorbing characteristic, consequently leading to a reduction in the workability of the fresh mix.

Figs. 14 and 15 demonstrate the morphology of CNT when incorporated into the cementitious composites at 0.05 wt% and 1 wt%, respectively. As shown in Fig. 14(a), individual CNT can be observed embedded into the cement matrix when the CNT is incorporated at a concentration of 0.05 wt%. Cement hydrates were formed and wrapped in the individual CNT, which result in high bonding strength between the CNT and cement matrix. Fig. 14b presents an SEM image of the

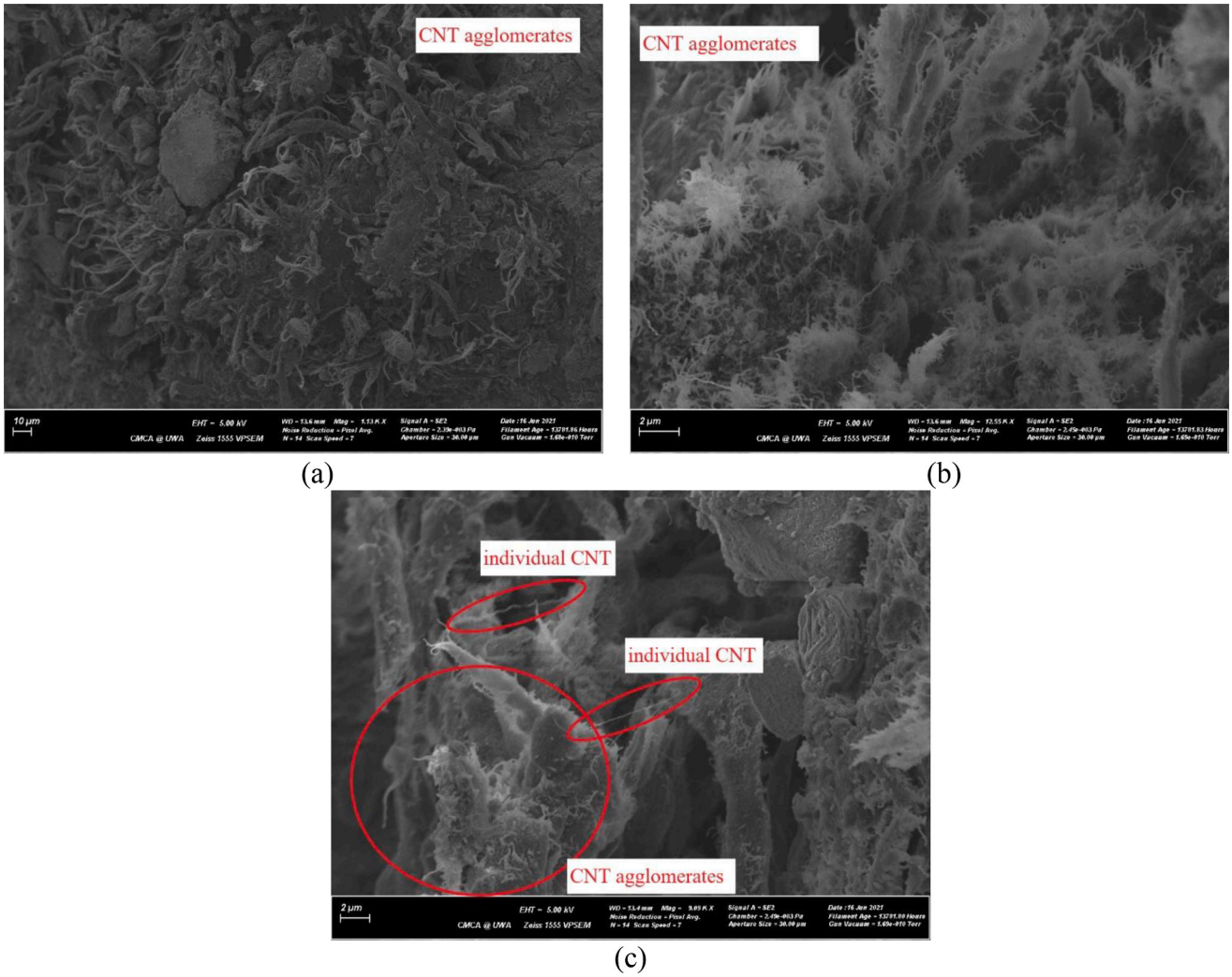


Fig. 15. SEM images of cementitious composites containing CNT at 1 wt%.

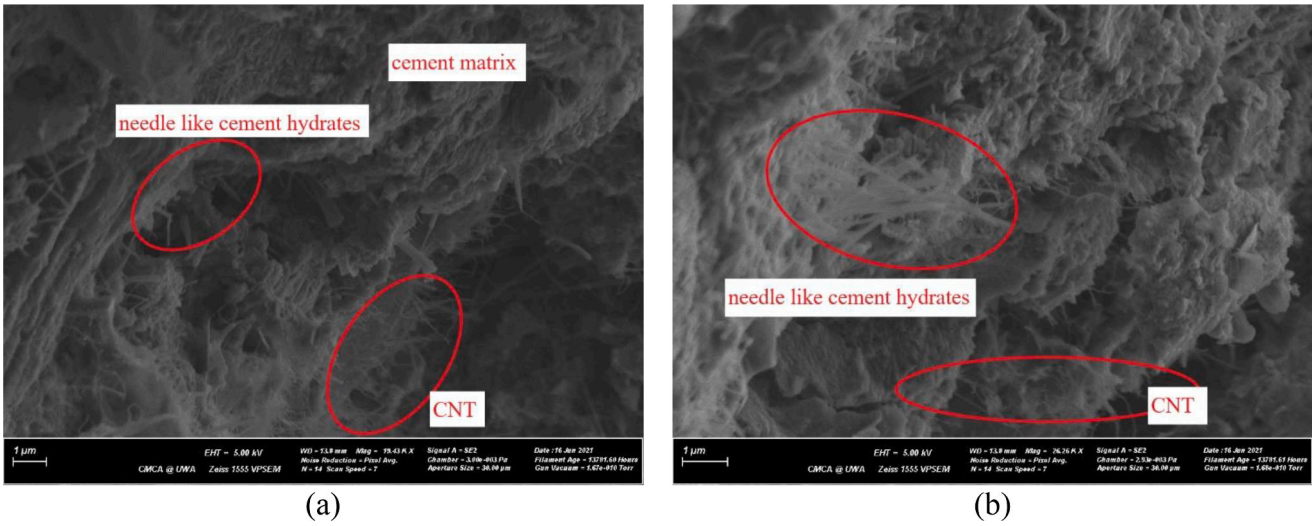


Fig. 16. SEM images of well-dispersed CNT when combined with CF within the matrix; (a) composites with 0.05 wt% of CNT and 0.3 wt% of CF, and (b) composites with 0.05 wt% of CNT and 0.5 wt% of CF.

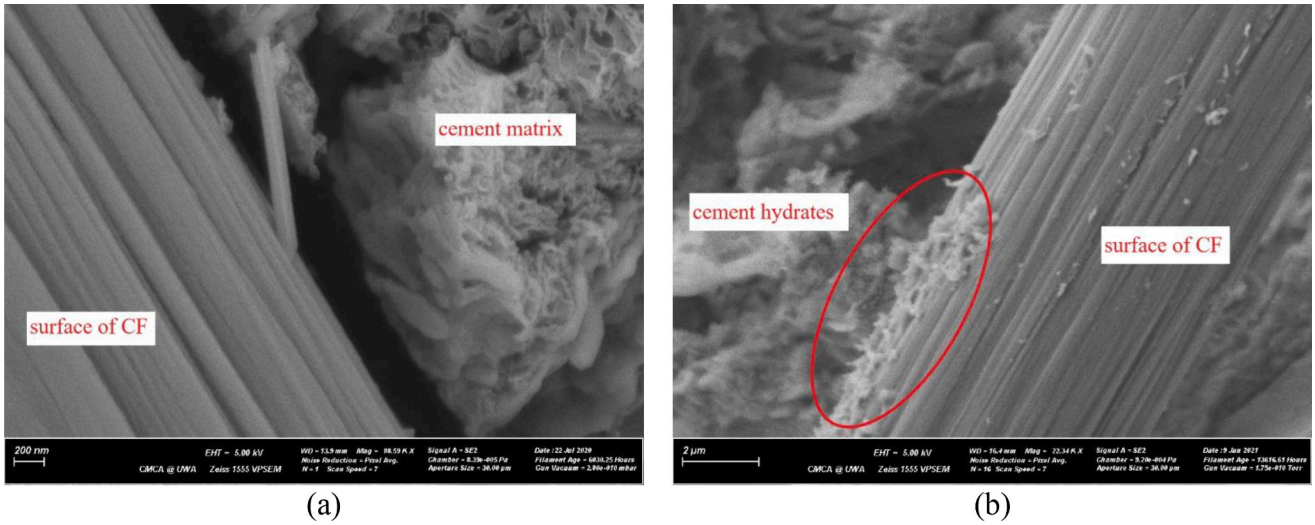


Fig. 17. The surface condition of CF when (a) the CF is adding alone in the cementitious composites, and (b) then CF is hybrid with CNT.

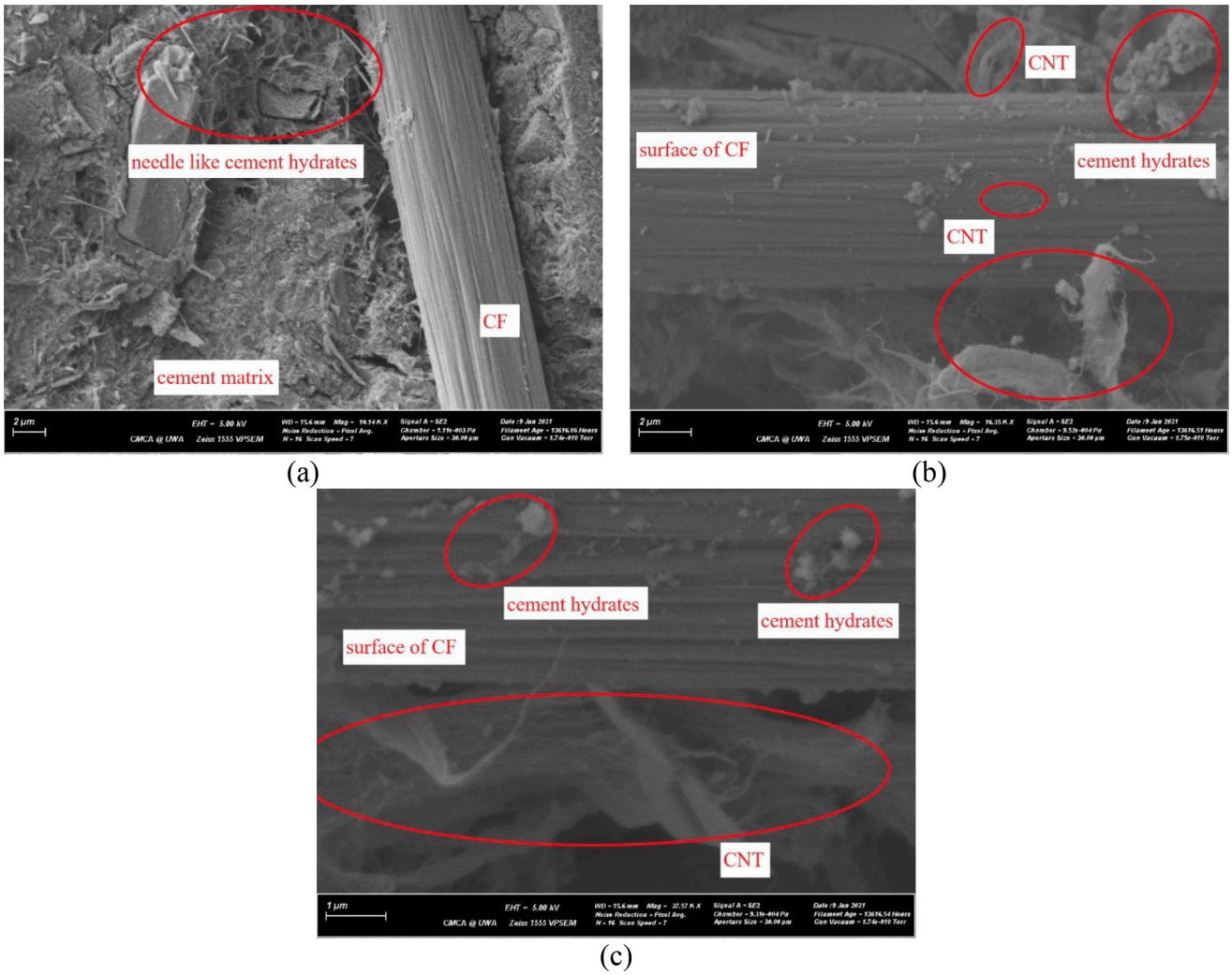


Fig. 18. The SEM images of cementitious composites containing hybrid MA, CF and CNT.

cementitious composites at a larger magnification, indicating that needle-like cement hydrates, a general form of C-S-H gel, can be observed. The presented figure proved that when CNT is at 0.05 wt%, it can adequately disperse within the matrix. The individual CNT can act as the reinforcement, and the addition of CNT can contribute to the formation of C-S-H gel. CNTs may fill the pores between C-S-H products,

which can provide a reinforcing effect and densify the matrix, and consequently, enhanced the mechanical properties of the composites [36,60]. However, when CNT is adding at a larger dosage, it is less likely for CNT to be uniformly distributed within the matrix. As shown in Fig. 15a, CNT agglomerate site could be observed. The CNTs were presented in bundle form instead of being embedded into the cement matrix. According to the enlarged images presented in Fig. 15b and c, the CNT demonstrate a loose bonding strength with the surrounding matrix, where noticeable gap and voids could be observed. The gaps and voids may block the conductive path and affect the electrical resistivity and piezoresistivity of the composites. Besides, the CNT bundles can act as defects within the matrix and affect the mechanical properties of the cementitious composites.

Fig. 16 shows the SEM images of cementitious composites containing hybrid CNT (0.05 wt%) and CF at 0.3 wt% (Fig. 16a) and 0.5 wt% (Fig. 16b), respectively. individual CNT can be observed embedded within the cement matrix, indicating that with the presence of CF, 0.05 wt% of CNT can also be properly dispersed. Besides, the matrix presents a dense form, and needle-like cement hydrates can be observed. This indicates that CNT can accelerate the formation of the C-S-H gel by creating more nucleation sites [55,61]. Fig. 17 demonstrates the differences of CF surface with and without the addition of CNT. No obvious cement hydrates could be observed adhering on the surface of CF, as shown in Fig. 17a. This phenomenon can be explained by the hydrophobic characteristic of the CF surface, which also indicate the week bonding between the CF and surrounding matrix [62]. Therefore, composites containing CF alone present poor repeatability of the piezoresistive response as the CF may be deboned during the loading process and not recover to its original position after the load is removed. However, it can be seen from Fig. 17b that hydration products could adhere to the surface of CF with the addition of CNT. It is possible that CNT attached to the CF surface can bond into the surrounding matrix and enhance the bonding strength of CF. Besides, the CNT can improve the chemical reaction between the surface functional groups and the cement hydrates, which also contribute to the adhesion between CF and the matrix [49].

Fig. 18 present the SEM images of cementitious composites containing hybrid MA, CF and CNT. Needle-like cement hydrates can be observed within the matrix, as shown in Fig. 18(a), which is able to fill the gap and reduce the matrix porosity. Cement hydrates can be found adhering on the surface of CF, indicating a good bonding between CF and the surrounding matrix, which explained the improved repeatability and stability of the piezoresistive response. Although slight CNT agglomerate can be observed according to the SEM images (Fig. 18b and c), individual CNT can be observed embedded within the matrix and attaching on the surface of CF. The CNT can bridge the CF and the surrounding matrix, which contribute to the bonding between CF and matrix and enhance the continuity of the electrically conductive network, consequently leading to improved compressive strength piezoresistive performance.

4. Conclusions

This study aimed to evaluate the combined effect of MA, CF and CNT on the mechanical, electrical and piezoresistive properties of cementitious composites. The following conclusions can be made:

MA can achieve a 5% increment in compressive and a 25% increment flexural strength with a value of 37.3 and 5.7 MPa, respectively, at a concentration of 100 wt%. However, the hybridisation of CF and MA cannot enhance the mechanical properties due to less numbers of CF incorporated and reduced workability.

CNTs may fill the pores between C-S-H products, providing a reinforcing effect and densifying the matrix. Compressive strength at 38.4 MPa and flexural strength at 6.5 MPa can be achieved, with a 7% and 42% increase, respectively, when incorporating 0.1 wt%. of CNT. However, agglomeration can be observed when CNT is incorporating at a large concentration.

100 wt% of MA, 0.3 wt% of CF and 0.05 wt% of CNT is the best functional filler combination in terms of the compressive strength, which achieved a 17% of improvement, proving that multiple reinforcing effects can be achieved when combining different types of functional fillers, which a single filler cannot achieve.

MA and CNT cannot develop a continuous conductive network when adding into the matrix by themselves. In addition, the CNT agglomeration resulted in large signal noise when CNT was added at 0.5 and 1 wt %. However, MA and CNT can contribute to the conductive network formed by CF.

The hybrid MA/CF and CF /CNT exhibit a synergistic effect and lead to lower resistivity. The maximum FCR at 55% can be achieved with less CF incorporation (0.5 wt%) with the combination of MA (100 wt%). Composites containing CF at 0.5 wt% and CNT at 0.05 wt% achieved the lowest resistivity with a value of 250 $\Omega \cdot$ cm, which is less than one-third of the resistivity achieved by 0.7 wt% of CF.

100 wt% of MA, 0.3 wt% of CF and 0.05 wt% is the best functional filler combination which can be beneficial for improving the piezoresistive performance of cementitious composites. The piezoresistive response achieved a maximum FCR of 44.7% and demonstrates enhanced linearity, repeatability, signal to noise ratio and stability.

CRediT authorship contribution statement

Lining Wang: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – original draft. **Farhad Aslani:** Funding acquisition, Supervision, Project administration, Resources, Conceptualization, Methodology, Data curation, Investigation, Visualization, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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