

Synergistic effects of CNT and CB inclusion on the piezoresistive sensing behaviors of cementitious composites blended with fly ash

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Abstract. The present study investigated the synergistic effects of carbon nanotube (CNT) and carbon black (CB) inclusions on the piezoresistive sensing behaviors of cementitious composites. Four different CNT and CB combinations were considered to form different conductive networks in the binder material composed of Portland cement and fly ash. The cement was substituted with fly ash at levels of 0 or 50% by the mass of binder. The specimens were cured up to 100 days to observe the variations of the electrical characteristics with hydration progress, and the piezoresistive sensing behaviors of the specimens were measured under cyclic loading tests. The fabricated specimens were additionally evaluated with flowability, resistivity and cyclic loading tests, and morphological analysis. The scanning electron microscopy and energy disperse X-ray spectroscopy test results indicated that CNT and CB inclusion induced synergistic formations of electrically conductive networks, which led to an improvement of piezoresistive sensing behaviors. Moreover, the incorporation of fly ash having Fe³⁺ components decreased the electrical resistivity, improving both the linearity of fractional changes in the electrical resistivity and reproducibility expressed as R² under cyclic loading conditions.

Keywords: Carbon black (CB); Carbon nanotube (CNT); cementitious composites; fly ash; piezoresistive sensing behaviors

1. Introduction

Cementitious materials are the most frequently utilized construction materials in the world (Bilotti *et al.* 2013, Han *et al.* 2009); however, they are brittle and hence vulnerable to cracks, which may decrease the durability of matrix (Seo *et al.* 2020, Bilotti *et al.* 2013, Han *et al.* 2009, Hannan *et al.* 2018, Krishansamy and Arumulla 2018, Nasr *et al.* 2018). These problems have necessitated the development of effective crack monitoring methods on cementitious materials (Ubertini *et al.* 2014, Gomis *et al.* 2015, Jang *et al.* 2018, 2021). The addition of electrically conductive fillers, such as steel fiber, graphite, carbon fiber (CF), carbon nanotube (CNT), and carbon black (CB) allows the cementitious composites to improve their electrical conductivity, thereby achieving sensing capabilities (Xie *et al.* 2012, Han *et al.* 2016, Nam *et al.* 2016, Jang *et al.* 2021, Yoon *et al.* 2020a, Tohidi *et al.* 2018). Based on the newly implemented characteristics, electrically conductive cementitious composites have been proposed to effectively

monitor cracks that occur in various construction systems (Xie *et al.* 2012, Han *et al.* 2016, Nam *et al.* 2016).

A conductive pathway in cementitious composites could be changed when external loadings are applied to composites or cracks occur in the composites (Xie *et al.* 2012, Han *et al.* 2016, Nam *et al.* 2016). These principles enabled the conductive composites to be used as cement-based piezoresistive stress sensors (Kim *et al.* 2014b, Monteiro *et al.* 2017). Nam *et al.* (2016) fabricated electrically conductive cementitious composites incorporating Multi-walled CNT (MWCNT), reporting that the composites exhibited an electrical resistance change of 6.5% under vehicle loadings. Kim *et al.* (2014) reported the effects of CNT concentration on electrical and piezoresistive sensing behaviors, and the cementitious composites with 0.5 wt.% of CNT under 10 MPa of compressive stress exhibited approximately 25% in electrical resistance change. Monteiro *et al.* (2017) investigated the effect of CB addition on piezoresistive sensing behaviors of cementitious composites, and the CB-incorporated composites exhibited 1.72×10^{-3} MPa⁻¹ of sensitivity.

Recently, many researchers have examined cementitious composites containing different types of electrically conductive fillers to improve their mechanical, electrical, and piezoresistive sensing behaviors (Al-Dahawi *et al.* 2016, Kim *et al.* 2018b, Han *et al.* 2015). Al-Dahawi *et al.* (2016) reported that mechanical and electrical properties of cementitious composites incorporating conductive fillers

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with nano-sized (CNT and GNP) and micro-sized (CF) materials, indicating that the incorporation of different types of electrically conductive fillers can improve the mechanical and electrical properties of cementitious composites (Al-Dahawi *et al.* 2016). Kim *et al.* (2018) investigated the electrical characteristics of cementitious composites incorporating different replacement levels of CNT and CF. It was found that the well-formed conductive pathways composed of multiscale conductive fillers improved the effective electrical characteristics of the composites (Kim *et al.* 2018b). In addition, Han *et al.* (2015) investigated the synergistic effect of CNT and CB on the piezoresistive sensing. It was shown that the composites containing CNT and CB exhibited sensitivity of 2.69% MPa^{-1} , while that of the composites containing CNT solely was 0.4% MPa^{-1} (Han *et al.* 2015).

On the other hand, for several decades, supplementary cementitious materials (SCMs) (e.g., fly ash, blast furnace slag, silica fume, calcined clays, and natural pozzolans) have been utilized as a partial replacement for Portland cement due to concerns pertaining to the CO_2 emissions (Provis *et al.* 2015, Juenger and Siddique 2015, Ghafari *et al.* 2016, Wu *et al.* 2017, Seo *et al.* 2020). It is well known that the addition of SCMs in the cementitious composites improves the mechanical properties of the cementitious composites by the activation of pozzolanic and latent-hydraulic reactions (Ghafari *et al.* 2016, Juenger and Siddique 2015, Wu *et al.* 2017). Some researchers have reported the effect of SCMs on electrical characteristics of cementitious composites, concluding that an addition of SCMs can change the electrical resistivity (Zornoza *et al.* 2010, Nam and Lee 2016, Kim *et al.* 2014a).

Kim *et al.* (2014) used silica fume in CNT-incorporated cementitious composites, and incorporating of silica fume enhanced mechanical and electrical properties due to the improvement of CNT dispersion. Zornoza *et al.* (2010) fabricated the cementitious composites incorporating with three different amounts of fly ash (5.6%, 15.9%, and 24.3%) and reported the effectiveness of fly ash incorporation on the EMI shielding capabilities of the composites. Moreover, Nam and Lee (2016) fabricated CNT-incorporated cementitious composites containing fly ash, and observed the enhanced electrical and EMI shielding characteristics of the composites. It was reported that the dissolved Fe^{3+} from the fly ash could improve the electrical and EMI shielding characteristics of the composites (Nam and Lee 2016).

The incorporation of CNT and CB has been presumed to improve the electrical characteristics of cement-based composites, nevertheless, fewer efforts were given to exploring their synergistic and piezoresistive sensing behaviors in cementitious composites blended with fly ash (Chung 2012, Kim *et al.* 2019b). In this regard, herein, CNT and CB were simultaneously incorporated to form the electrically conductive networks in the cementitious composites, and fly ash was utilized to replace the cement. The specimens were cured up to 100 days to observe the variations of the electrical characteristics with hydration progress, and the piezoresistive sensing behaviors of the specimens were evaluated in terms of the cyclic loading

tests. In addition, the scanning electron microscopy (SEM) observation and energy disperse X-ray spectroscopy (EDS) analysis were conducted to elucidate the mechanisms on the electrical and piezoresistive sensing test results.

2. Experimental details

Type I Portland cement (PC; SAMPYO CEMENT Co., Ltd) and class C fly ash (MAXCON Materials Co., Ltd), each conforming to ASTM C 150 and ASTM C 618, respectively, were used as binder materials. Silica fume manufactured from Elkem Inc., (EMS-970) was utilized to enhance the CNT dispersions in the specimens (Kim *et al.* 2016). The chemical compositions of the cement, fly ash, and silica fume are listed in Table 1. The silica fume was mainly composed of SiO_2 with a specific gravity and average diameter of 2.1 and 200 nm, respectively (Nam and Lee 2016). The MWCNT (Jeio Co. Ltd., Korea) with an average diameter of 10 nm and length of approximately 100-200 μm was used as an electrically conductive filler. Furthermore, CB (ACE C & Tech Co. Ltd., Korea) with an approximate diameter of 30 nm was added as an electrically conductive filler. A polycarboxylate-type superplasticizer (Dongnam Co., Ltd., FLOWMIX 3000L) was used to reduce water contents and improve CNT dispersions in the specimens (Kim *et al.* 2016, 2018a, b, c). In the present study, silica fume and superplasticizer were used to improve the dispersion of CNT particles in the cementitious composites due to the ball-bearing effect of silica fume and steric repulsion effect caused by the superplasticizer, respectively.

The specimens were classified into two groups according to the utilized fly ash content. Fly ash was used to replace the cement by 0 or 50% by binder mass, and silica fume, water, and polycarboxylate-typed superplasticizer were added in the specimens by 10%, 35%, and 1% by binder mass, respectively. Four different specimens were prepared for each group according to contents of electrically conductive fillers. CNT and CB contents added to the specimens were fixed at 0.4%, or 0.8% by binder mass, respectively, considering the percolation threshold region of cementitious composites with CNT (Nam *et al.* 2016). Hence, a total of eight different set of specimens were prepared, and the details of mix proportions are summarized in Table 2.

The CNT and CB-incorporated cementitious composites blended with fly ash were fabricated using $50 \times 50 \times 50 \text{ mm}^3$ cubic molds conforming to ASTM C 109. The fabrication process of specimens was as follows: Binder materials (cement and fly ash), silica fume, and electrically conductive fillers (CNT and CB) were incorporated into a standard Hobart mixer, and were dry mixed for 5 min. Then, solutions (water and superplasticizer) were poured into the dried mixtures, and were mixed for 5 min, and the mixtures were poured into cubic molds. Here, copper-typed electrodes were used to measure the electrical resistances of the specimens. The corresponding length, width, and thickness of the used copper electrodes were 70 mm, 30 mm, and 0.3 mm respectively. The silver paste was painted

Table 1 Chemical compositions of cement, fly ash, and silica fume used in the present study (wt.%)

Chemical composition	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	Cr ₂ O ₃	P ₂ O ₅
Cement	-	5.9	21.0	2.1	-	62.5	3.2	-	-
Fly ash	3.01	12.94	37.14	4.84	0.76	28.68	3.97	-	0.659
Silica fume	-	4.00	90.70	-	2.00	1.30	1.09	1.30	-

Table 2 Mix proportions of the specimens (wt.%)

Specimens	Cement	Fly ash	Binder	CNT	CB	SF	Water	SP
N4				0.4	0			
N4B4	100	0	100	0.4	0.4	10	35	1
N4B8				0.4	0.8			
N8				0.8	0			
FN4				0.4	0			
FN4B4	50	50	100	0.4	0.4	10	35	1
FN4B8				0.4	0.8			
FN8				0.8	0			

on both electrodes to minimize the contact resistance between the electrodes and specimens, and the electrodes were inserted to the specimens with 30 mm distance. The specimens were wrapped and placed in an oven at a temperature of 25°C for initial 24 h. After 24 h of initial curing, the specimens were demolded, and then cured at oven with identical conditions for 100 days to observe the reaction fly ash in the specimens (De Weerd *et al.* 2011, Zeng *et al.* 2012, Hanjitsuwan *et al.* 2011).

The dispersion of electrically conductive fillers in the cementitious composites was affected by the fluidity of mixtures (Kim *et al.* 2014a, 2016, Nam *et al.* 2016). A flowability test of fresh-state mixtures was, therefore, conducted in accordance with the specification provided in the ASTM C 1437 (Kim *et al.* 2014a, b). Three diameters of fresh mixtures on table flow test apparatus were measured, and were averaged to calculate a representative value. In addition, the electrical DC resistances of specimens were measured using a digital multi-meter. The specimens were cured up to 100 days to observe variations of electrical characteristics caused by dissolution of fly ash (Park *et al.* 2019).

The piezoresistive sensing behaviors of specimens were observed under cyclic loadings. The compressive load of 25 kN at a rate of 0.02 mm/s was applied to the specimens for 5 cycles, while the electrical DC resistance was measured using the two connected probes. The fractional change of electrical resistivity (FCR) was calculated as (Yoon *et al.* 2020a, Jang *et al.* 2020a, b, c, d, 2021a, b, Khalid *et al.* 2021)

$$FCR (\%) = \frac{\rho - \rho_0}{\rho_0} \times 100 \quad (1)$$

where ρ and ρ_0 denote the calculated electrical resistivity at each compressive load and initial electrical resistivity of the specimens, respectively (Nam *et al.* 2016, Park *et al.* 2019). The piezoresistive sensing behaviors of the specimens including the maximum FCR, reproducibility expressed as

R^2 , and linearity of FCR were then examined.

Microstructural morphologies of the specimens were examined through a field emission SEM-EDS (FE-SEM, Hitachi S4800). It is noted that the specimens for SEM and EDS analyses were prepared according to RILEM recommendations (Snellings *et al.* 2018). The specimens were crushed and immersed in isopropanol for 15 min. Afterwards, they were vacuum filtered and rinsed with both isopropanol and diethyl ether. The specimens were then stored in a low vacuum desiccator before the analysis (Bae *et al.* 2020).

3. Electrical characteristics of the composites

The flowability values of CNT- and CB-incorporated cementitious composites blended with fly ash are shown in Fig. 1. The flowability values decreased as the contents of electrically conductive fillers increased, which can be deduced from the water absorbed by the conductive fillers. Previous studies reported that CNT with hollow porous tube shapes can absorb water (Jin *et al.* 2016, Kim *et al.* 2018a, b), and CB with large specific area surfaces can also absorb water which led to low flowability values of fresh-

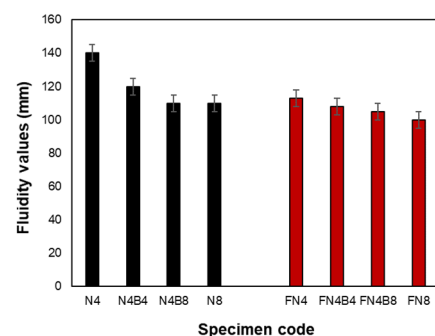


Fig. 1 Fluidity values of the cementitious composites

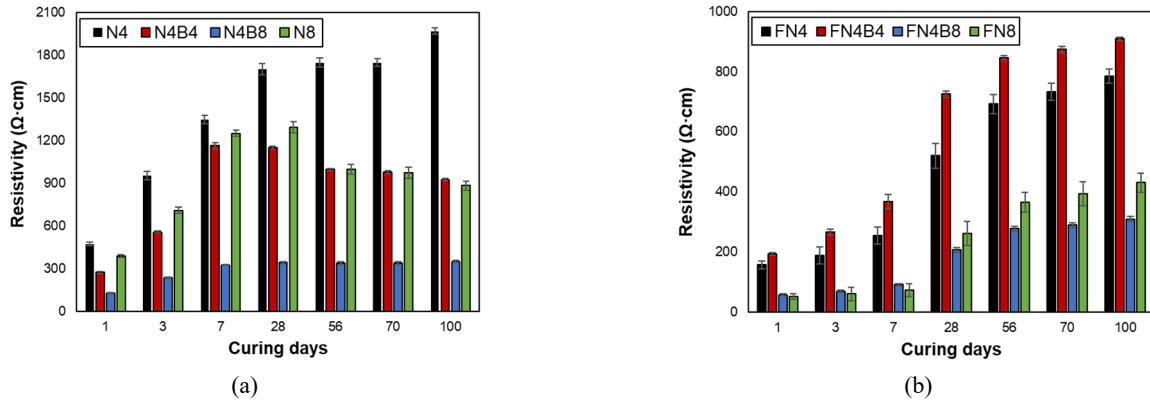


Fig. 2 Electrical resistivity of the cementitious composites blended with (a) 0%; and (b) 50% of fly ash/binder ratios

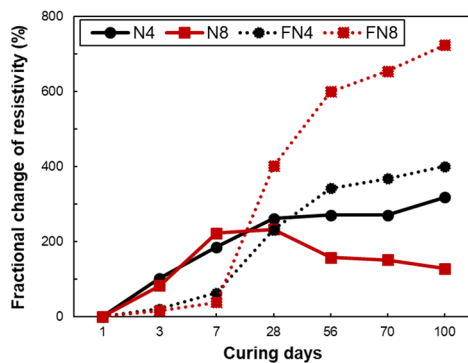


Fig. 3 Fractional changes of electrical resistivity during curing days

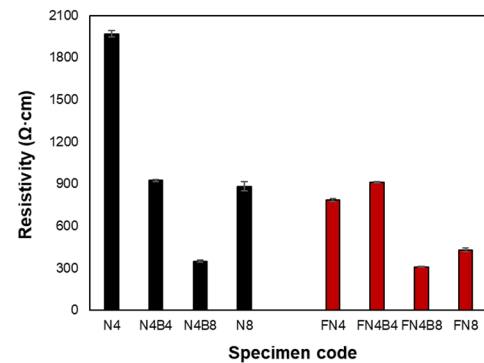


Fig. 4 Electrical resistivity of the cementitious composites at 100 days curing

state mixtures (Dai *et al.* 2010). The incorporation of the conductive fillers reduced the overall flowability of the specimens, however, the reduction ratio was not large as the filler content increased. The electrical resistivity values of the specimens measured up to 100 days of curing is shown in Fig. 2, and the fractional changes of resistivity of N4, N8, FN4, and FN8 specimens are calculated using the initial values of resistivity in each specimen (i.e., 470, 390, 157, and 52 $\Omega\cdot\text{cm}$) as illustrated in Fig. 3. The resistivity values increased as curing proceeded owing to the progress of hydration. Specifically, the specimens with fly ash showed the stable electrical resistivity of specimens after 70 days due to the effects associated with the reaction of fly ash as shown in Fig. 3 (Kim *et al.* 2016, Nam and Lee 2016). It can be derived from the degree of hydration with time as incorporated of the fly ash. Xiao and Li (2008) investigated the effect of fly ash incorporation on the hydration of cementitious composites, indicating the stable electrical resistivity is observed slower compared to the specimens without fly ash. The electrical resistivity of specimens cured at 100 day was shown in Fig. 4, revealing that the electrical resistivity decreased as contents of electrically conductive fillers increased. Especially, it was noted that the electrical resistivity of N4B8 and FN4B8 specimens was lower than that of N8 and FN8 specimens. This phenomenon can be deduced from the formation of CNT- and CB-based electrically conductive networks. The incorporation of spherical CB with a large specific area,

which could be located between CNT particles, led to the well- formed electrically conductive networks (Han *et al.* 2015). In Fig. 4, it was observed that the electrical resistivity of specimens blended with fly ash was lower than that of the specimens without fly ash. It is believed that the results are related to the flowability properties of specimens in Fig. 1. The fluidity values of N4 and FN4 specimens were 140 and 115 \pm 5 mm, respectively. Kim *et al.* (2014a, b) reported that the fluidity values of the cement paste can affect the dispersion of electrically conductive fillers. Specifically, it is noted that the decreasing of fluidity values can possibly cause the decreasing of the electrical resistivity of cementitious composites (Kim *et al.* 2014a, b). Here, the fluidity value of FN4 (i.e., 120 \pm 10 mm) is regarded as favorable fluidity value to disperse CNT into the composites, thereby affected a decrease in electrical resistivity (Kim *et al.* 2016, 2017, Nam *et al.* 2016). Moreover, a reduction in electrical resistivity of the specimens was observed in the specimens blended with fly ash, and it can be deduced by the Fe^{3+} ions present in the fly ash (See Fig. 4). Nam and Lee (2016) reported that amounts of Fe^{3+} increased as the fly ash to binder ratio increased, and it decreased the electrical resistivity of cementitious composites. The present test results exhibited a similar aspect to what was reported in the earlier studies, and the fly ash incorporation showed the potential to decrease the electrical resistivity of the cementitious composites (Hanjitsuwan *et al.* 2011, 2014, Nam and Lee 2016).

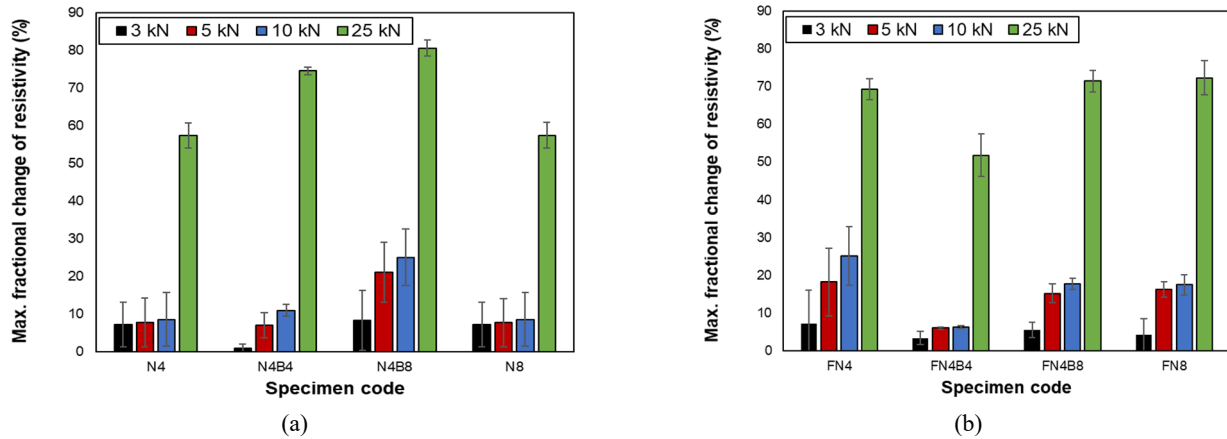


Fig. 5 Maximum fractional change in electrical resistivity under cyclic loadings

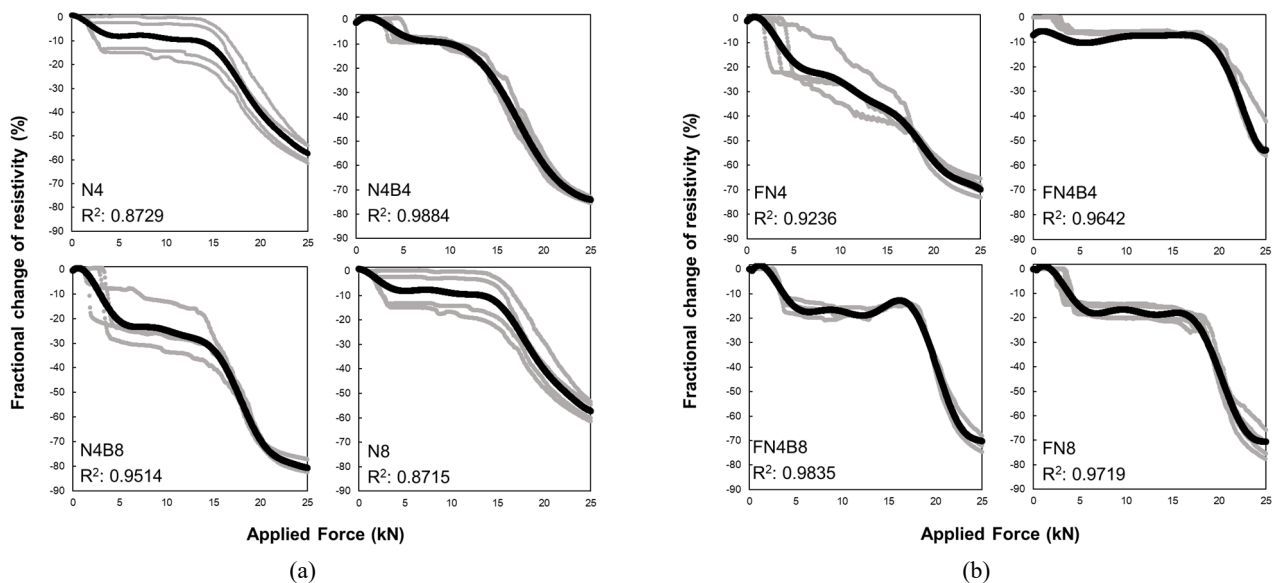


Fig. 6 Relationship between the fractional change in electrical resistivity and applied forces: (a) 0%; and (b) 50% of fly ash/binder ratios

4. Synergistic effects of CNT and CB inclusions on piezoresistive sensing behaviors

The synergistic effect of CNT and CB on piezoresistive sensing behaviors of the specimens were investigated under the cyclic loadings. The relationship between FCR and applied forces, and maximum FCR at different applied forces (3 kN, 5 kN, 10 kN, and 25 kN) were calculated as shown in Fig. 5. As seen in Fig. 5, the effect of CNT content on the FCR of specimens was mimic, which was believed to be due to the percolation thresholds of the cementitious composites incorporating with CNT (Nam *et al.* 2016, Han *et al.* 2015, Monteiro *et al.* 2017). The 0.8 wt.% of CNT content was higher than the percolation threshold ranges (Nam *et al.* 2016), and therefore, it may help to form the dense electrical conductivity networks which lead to a low FCR under the cyclic loadings (Kim *et al.* 2016, 2019a). Meanwhile, the decreased standard deviation of results as CB content increased were shown in Fig. 5. The addition of CB reduced the redistribution of CNT-based electrically

conductive networks under the cyclic loadings, thereby reducing the standard deviation of test results. It was also found that the addition of CB improved the reproducibility (R^2 values), since incorporation of CB may reduce disturbances of CNT-based conductive networks under cyclic loadings as shown in Fig. 6.

This phenomenon can be known that distances between CNT particles can be changed when they are loaded, which causes breakdowns of CNT-based electrical conductivity networks (Han *et al.* 2015). Therefore, it can be concluded that the addition of CB improved the piezoresistive sensing behaviors of cementitious composites incorporating CNT.

Furthermore, it can be observed that incorporation of fly ash which had Fe^{3+} components improved the linearity of FCR at different loadings, and improved the reproducibility expressed as R^2 values. It was likely because the diameter of fly ash was larger than that of electrically conductive fillers, which reduced the redistribution of electrically conductive fillers when loadings were applied. In this regard, experimental results in the present study confirmed

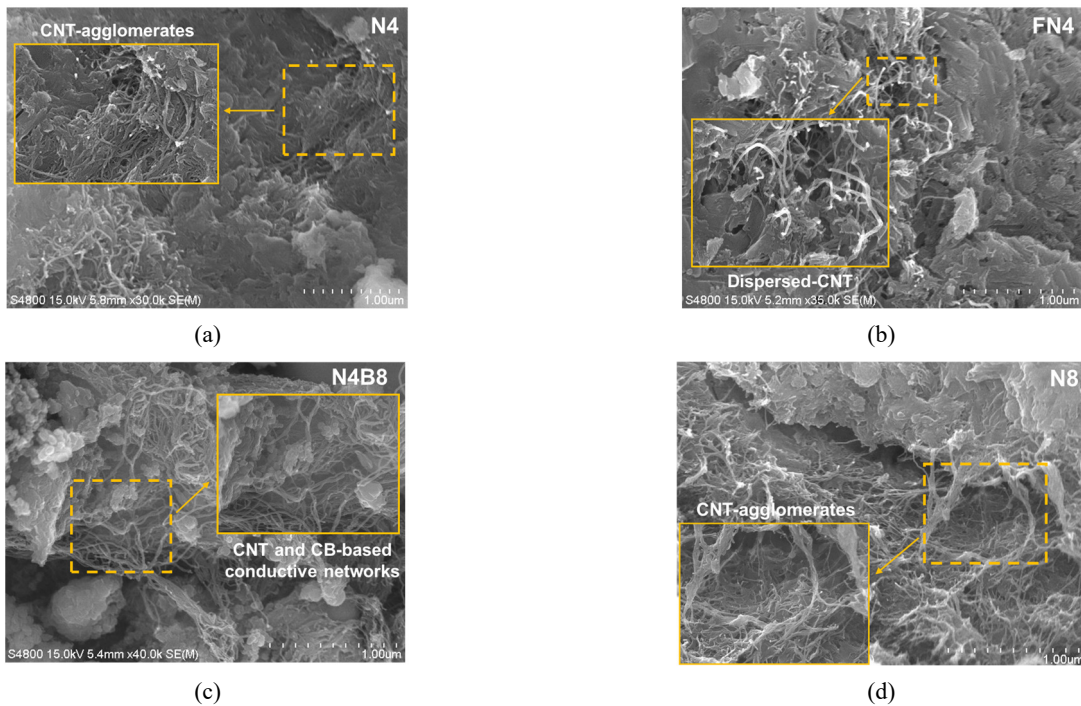


Fig. 7 SEM images of (a) N4; (b) FN4; (c) N8; and (d) N4B8 specimens

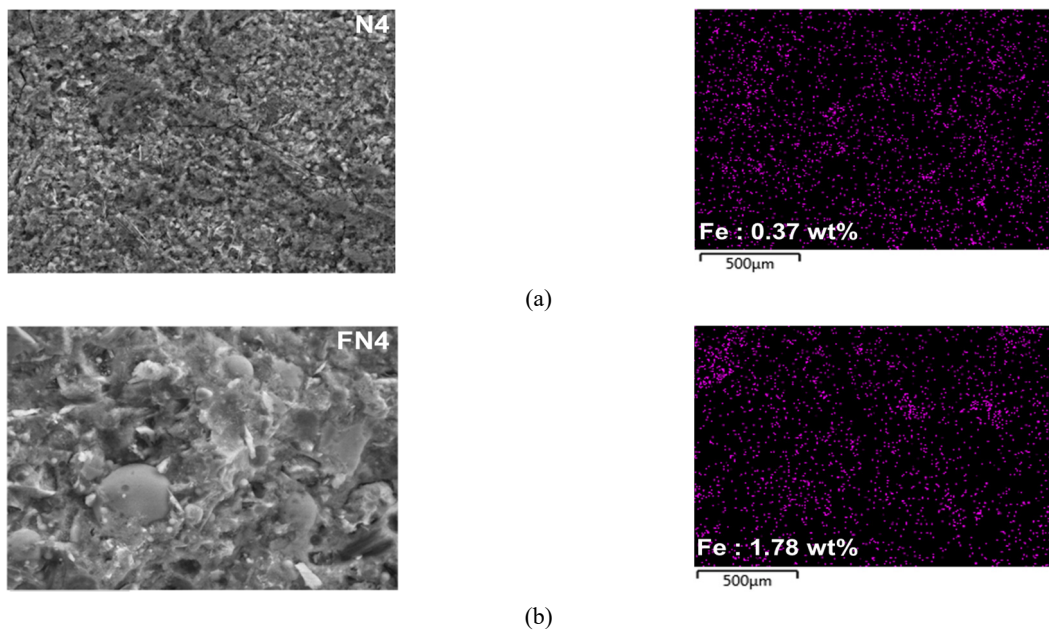


Fig. 8 EDS images of the (a) N4; and (b) FN4 specimens with Fe atomic signals

that incorporations of CB and fly ash-embedded cement have the potential to improve the piezoresistive sensing expressed as R^2 values. Microstructural images of CNT-CB-incorporated cementitious composites blended with fly ash were taken by SEM observation and EDS analysis to discuss with schematic illustration and the obtained experimental results. It was found that the specimens with high flowability values (≈ 140 mm) exhibited CNT-agglomerates; however, the dispersed CNT particles were observed in the specimens with favorable flowability values (≈ 115 mm), as shown in Figs. 7(a) and (b). The synergistic

effects of CNT and CB on piezoresistive sensing behaviors could be observed in Fig. 7(c). In Fig. 7(c), the CB with spherical shape was located between CNT particles, which supplemented the formations of electrically conductive networks, thereby reducing re-distributions of CNT particles under cyclic loadings. In addition, the N8 specimen with CNT content exceeded the percolation threshold region (≈ 0.8 wt.%) formed dense electrically conductive networks, causing CNT-agglomerates (See, Fig. 7(d)).

Meanwhile, the EDS analysis was conducted to

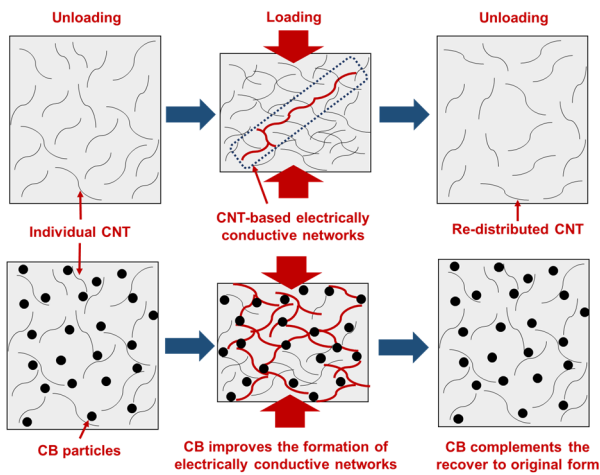


Fig. 9 Schematic illustration of electromechanical sensing principle of the cementitious composites

investigate the elemental composition of the specimens. Among the elemental compositions, the Fe^{3+} components which can improve the electrical characteristics as incorporated in the cementitious composites were noted considering the results in previous studies (Nam and Lee 2016, Hanjitsuwan *et al.* 2011, 2014). Nam and Lee (2016) reported that Fe^{3+} components increased as the increasing of fly ash in the cementitious composites, showing good agreements with the present results in Fig. 8. There are various factor including fluidity value, chemical composition, and types of dispersant which affect the electrical characteristics of CNT-incorporated cementitious composites. Apart from the fluidity value, the chemical composition i.e., Fe^{3+} in fly ash is possibly attributed to decrease the electrical resistivity of the specimens.

However, the effects of fly ash incorporation on the electrical characteristics of the cementitious composites will be investigated systematically in any forthcoming studies. However, the effects of fly ash incorporation on the electrical characteristics of the cementitious composites will be investigated systematically in any forthcoming studies.

A schematic illustration of CNT- and CB- based electrically conductive networks were depicted in Fig. 9. It addressed the addition of CB improved the formation of electrically conductive networks, and complemented the recovery of electrically conductive networks to original form during the cyclic loadings, led to the high reproducibility as investigated in Fig. 6. Consequently, the results of the cyclic loading test, SEM observation, EDS analysis, and the schematic illustration exhibited the applicability of using CB and fly ash to enhance the electrical and piezoresistive sensing behaviors of CNT-incorporated cementitious composites, and they are high compatibility with the present experimental results.

5. Conclusions

The present study showed the synergistic effects of CNT and CB inclusions on the electrical and piezoresistive sensing behaviors of cementitious composites blended with

fly ash. The piezoresistive sensing behaviors of the specimens were examined in terms of the cyclic loadings conditions, and various analyses including flowability, FE-SEM, and EDS were conducted to identify the effects of the multiscale fillers and Fe^{3+} components. The main findings obtained in this study are as follow:

- The electrical resistivity values of the specimens increased as the curing time increased, and the electrical resistivity values became stable after 70 days of curing due to the additional reaction of fly ash.
- The electrical resistivity of specimens reduced as the entire conductive filler increased. Specifically, the incorporation of CNT and CB with different shapes led to the formation of synergistic electrically conductive networks, reducing the electrical resistivity of specimens.
- The inclusion of CB decreased standard deviations of electrical resistivity values, and improved the reproducibility during cyclic loadings. This is possibly attributed to the incorporated CB reduced the disturbances of CNT-based electrically conductive networks during curing process and cyclic loading.
- It was observed that Fe^{3+} components in fly ash decreased electrical resistivity of specimens, and they led to an improvement on the linearity of FCR and the piezoresistive sensing reproducibility under cyclic loadings.

Further studies will be carried out to observe the long-term sensing performances of the fabricated composites and the practical application such as crack sensing as the composites are installed in cement-based structures.

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