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# MULTI-FUNCTIONAL CEMENTITIOUS COMPOSITES WITH SENSING AND HEALING CAPABILITIES

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For smart concretes to be developed, it must be gathering high mechanical and durability properties, in addition to satisfying special characteristics such as self-monitoring of damage. This study outlines attempts to develop advanced Engineered Cementitious Composites (ECC) with combined self-sensing and self-healing capabilities. The aim is to maintain or improve the high mechanical and ductility properties of ECC, while enhancing the self-monitoring and self-healing capabilities. To assure the self-sensing functionality, carbon-based materials with different volumes were incorporated in ECC formulations. The self-healing rates of control and piezoresistive ECC's were assessed by pre-cracking specimens up to 60% of their original flexure deformations and left those samples to heal under moist curing. The mechanical performances and ductility were evaluated based on compressive and flexural strengths, and mid-span beam deflection capacity measurements. The self-healing/self-sensing efficiency was tested by assessing the electrical resistivity (ER) variations of cylindrical specimens. Mechanical results of carbon-based ECC mixtures showed better or comparable performances than the corresponding control ECC. This study also reveals that the type of carbon-based materials and moisture state of specimens considerably influence the self-sensing/self-healing ability of ECC mixtures.

*Keywords:* Smart concrete, Carbon fiber, Nano-tubes, Ductility, Pre-cracking.

## 1 INTRODUCTION

In recent years, there is growing efforts toward the development of smart multi-functional engineering materials. For reinforced concretes, multi-functionality means that the material has to provide additional non-structural properties without using any embedded or attached devices (Galao *et al.* 2014). Among the conductive materials that were incorporated successfully in cementitious composites, the use of carbon-based materials, such us carbon fibers (CF), carbon nanotubes (CNT) and carbon black (CB), showed not only high self-sensing ability; however also, improved mechanical and durability properties (Al-Dahawi *et al.* 2016a). Regarding the structural serviceability, concretes reinforced with carbon materials were capable to vibration sensing, and shrinkage and fatigue monitoring (Nešpor *et al.* 2016). In addition, for damage sensing, studies reported also strain-sensing under compression and self-sensing of damage initiation, especially when using carbon nanofibers (CNF) or CNT (Galao *et al.* 2014).

Along with their ability to resist large tensile and shear forces due to their multiple micro-cracking behavior, high ductility and tight crack width, Engineered Cementitious

Composites (ECC) has been also reported as an ideal self-healing material in a variety of environmental conditions (Siad *et al.* 2015). However, in view of self-sensing ability, regular ECCs (without conductive materials) showed negligible sensing properties. The incorporation of carbon-based materials into ECC formulations presented a promising self-sensing material (Al-Dahawi *et al.* 2016b), with enhanced mechanical and durability properties (Li *et al.* 2013). Among the limited studies completed on health monitoring of ECCs, CB was used successfully for strain and damage sensing (Li *et al.* 2013). No work on the combined effect of self-healing and self-sensing has so far been carried out. This study, investigate the possibility of combining self-sensing (of micro-cracking) and self-healing abilities through the incorporation of different amount of CF or CNT in ECC formulations. In addition, mechanical, durability and piezorestive behaviors were also assessed in order to assure the multifunctional behaviors of ECCs.

## 2 EXPERIMENTAL PROGRAM

### 2.1 Materials

Portland cement (PC) conforming to ASTM C150 (2012a), Class-F fly ash (FA) complying with ASTM C618 (2012b), Silica sand (SS) with a maximum aggregate size of 400 µm, polyvinyl alcohol (PVA) fibers, and high range water reducing admixture (HRWRA) were used in the production of all ECC mixtures. Two conductive carbon-based materials characterized with high mechanical and electrical properties were used during the study, namely carbon fibers (CF) and carbon nanotubes (CNT). Physico-mechanical properties of CF and CNT are presented in Table 1 and the SEM micrographs are shown in Figure 1.

Table 1. Properties of CF and CNT.

| Material | CF | CNT |
|---|---|---|
| Resistivity (ohm-cm) | 0.00155 | > 0.01 |
| Density | 1.81 | 2.1 |
| Length (mm) | 6 | 0.01-0.030 |
| Carbon Content (%) | 95 | > 90 |
| Fiber Diameter (µm) | 7.2 | 0.01-0.03 |
| Tensile Strength | 4137 | - |
| Tensile Modulus | 242 | - |

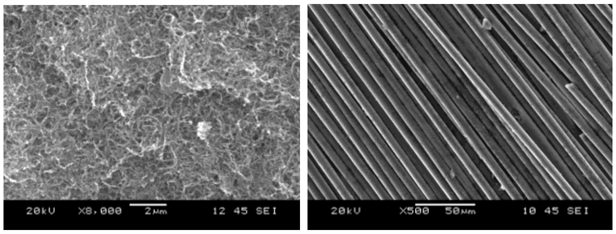

Figure 1. SEM micrographs of CF and CNT used.

### 2.2 Mixture Proportions, Specimen Preparation and Compressive Strength Results

The amount of CF and CNT used in ECCs were 1% and 0.5% by volume of the total mixture, and 0.50% and 0.25% by mass of the cementitious materials, respectively, as presented in Table 2. 360×75×50 mm prisms were used to assess modulus of ruptures (MOR) and mid-span beam deflection capacities under four-point bending load. For the self-sensing/self-healing evaluation, Ø100×50 mm cylinders were produced to monitor the change in electrical resistivity (ER). Based on compressive strength results, the incorporation of CF or CNT into ECC mixtures increased the compressive strength at different ages, though differences seem decreased at advanced curing time, especially between ECCF0.5, ECC1 and ECC1.2. When comparing results of ECC-based conductive materials, ECCN0.5 exhibited the greatest values at all testing times with differences ranged between 4.9 MPa at 28 days and 0.9MPa at 28+90 days, compared to ECCF1. In addition to the possible filling effect of nano-carbon particles by bridging nano-sized and micro-sized pores even within C-S-H and C-H hydrates gel (Manzur *et al.* 2014), the presence of CNT may also have enhanced the stiffness of C–S–H gel (Shah *et al.* 2009) affecting positively the results.

Table 2. Mixture proportions and compressive strength results.

| Proportions | PC | FA | W | SS<br>(kg/m$^3$) | PVA | HRWRA | CF | CNT<br>(%) | Compressive Strength (MPa)<br>7d | 28d | 120d |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ECC1.2 | 540 | 650 | 322 | 428 | 19.5 | 6.7 | 0 | 0 | 40.2 | 58.8 | 73.6 |
| ECCF0.5 | 548 | 493 | 325 | 434 | 19.5 | 7.5 | 0.5 | 0 | 46.1 | 59.5 | 73.8 |
| ECCF1 | 557 | 334 | 331 | 441 | 19.5 | 9.0 | 1.0 | 0 | 48.8 | 60.8 | 76.0 |
| ECCNT0.25 | 567 | 170 | 337 | 449 | 19.5 | 11.2 | 0 | 0.25 | 48.2 | 62.6 | 76.8 |
| ECCNT0.5 | 577 | 0 | 343 | 457 | 19.5 | 12.5 | 0 | 0.50 | 52.2 | 65.7 | 77.0 |

## 2.3 Self-Healing and Self-Sensing Assessment Techniques

To assess the self-healing/self-sensing rates, several prisms and cylinders were deliberately preloaded up to 60% of their ultimate flexural deformations and splitting tensile deformations, respectively. Ten specimens from each mixture were used to calculate the average results. Self-sensing/self-healing ability of pre-cracked specimens was based on electrical resistivity measurements recorded by means of concrete electrical resistivity meter. The effect of moisture state of specimens was also verified by measuring ER at constant conditions in saturated, partially-saturated and dried states. The ER results were determined as follows by Eq. (1):

$$ER = Z\cos(\theta) \times \ A/L \qquad (1)$$

ER (Ω.cm): electrical resistivity, Z (Ω): impedance measured by the device, A (cm$^2$): cross-sectional area of the specimen, L (m): length of the specimen and θ stand for phase angle (º).

Figure 2. Electrical resistivity testing of ECC cylindrical specimens.

## 2.4 Flexure Performance

### 2.4.1 *Crack characterization*

Due to the important relationship between cracking developed and self-healing efficiency, the initial crack width and number (at 28-day preloading) were measured (Table 3). Typical crack pattern at 28 days of aging is shown in Figure 3. Compared to ECC1.2, unlike the increase in crack widths and decrease in number of cracks with CF content, the presence of CNT reduced the crack widths and increased the number of cracks with CNT content. These prove that the micro-cracking behavior is largely influenced by the type of conductive materials incorporated in ECC.

Table 3. Crack characteristics of preloaded specimens.

| Mix ID | Residual crack width (CW) (μm)<br>Average CW | Maximum CW | Number of cracks |
|---|---|---|---|
| ECC1.2 | ~50 | 70 | 11-13 |
| ECCF0.5 | ~60 | 80 | 7-8 |
| ECCF1 | ~60 | 80 | 5-7 |
| ECCN0.25 | ~40 | 50 | 12-14 |
| ECCN0.5 | ~30 | 50 | 14-16 |

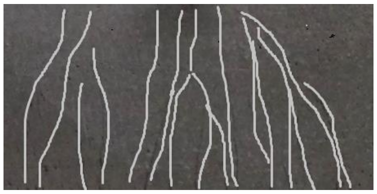

Figure 3. Typical crack pattern in ECCN0.5.

### 2.4.2 *Modulus of rupture (MOR) and mid span beam deflection capacities*

Figures 4 and 5 present ultimate flexural strengths and mid-span beam deflection capacities respectively, of preloaded/reloaded ECC specimens tested at after pre-cracking and wetting curing. From Figures 4 and 5, compared to ECC1.2, flexure strengths of sound specimens at 28-day increased with CF and CNT addition, and mid-span beam deflections decreased considerably with CF levels; however, equivalent or improved deflections were registered when using CNT. When considering the self-healing rate, the presence of CF or CNT was associated with good and faster self-healing rate for both MOR and deflection capacity results. If considering the crosslinking effect of CNT particles with hydration products explained above, the incorporation of CNTs into ECCs was likely associated with improved hardened paste skeleton, which also led to resist micro-cracks formation and better self-healing performances. For CF-ECCs, a higher fiber bridging capacity and an increased fiber-to-matrix bond interface is expected when adding CFs into ECCs, which can suggest a pullout effect of fibers when applying certain flexure strength (Siddique *et al.* 2014). However, after pre-cracking, CFs provided additional cores for the precipitation of healing products resulting in better recovery than control ECC.

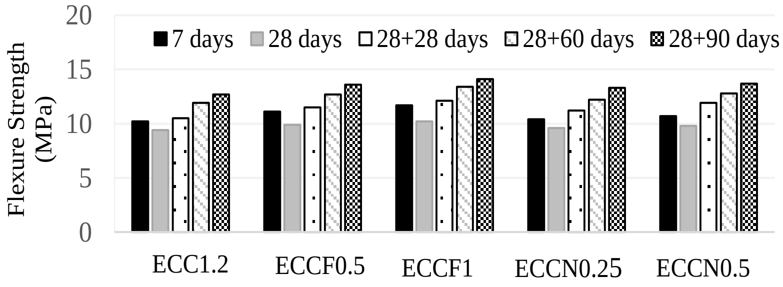

Figure 4. Modulus of rupture of Re-loaded ECC specimens.

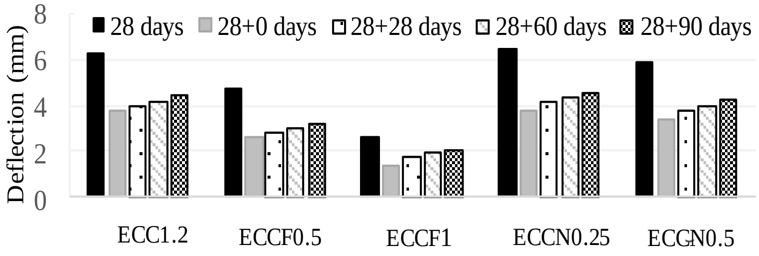

Figure 5. Deflection capacities of re-loaded ECC specimens.

### 2.4.3 *Electrical resistivity*

Changes in ER results of sound and pre-loaded ECC specimens are shown in Table 4. The comparison of ER of different ECCs revealed remarkable decrements as CF and CNT content increased, which can confirm the effectiveness of CF and CNT in increasing ECC conductivity and sensing, though the incorporation of CF contents was accompanied with much lower ER results resulting in better self-sensing ability than the use of CNTs. However, the comparison of results of sound and preloaded specimens at the age of 28 days revealed that the incorporation of CF caused ECCs to be not self-sensing to pre-cracking, even when the moisture state of specimens was changed from saturated to dried state. In contrary, CNT-based ECCs exhibited self-sensing to damage; however, only in partially-saturated and dried states. The sharp reductions in ER when using CF in ECC is in line with recent literature and was attributed to the extended conductive path along the ECC specimens through the presence of fiber to fiber contact (Al-Dahawi *et al*. 2016a), which may also have created conductive bridges inside the micro-cracks and resulted consequently in the absence of self-sensing to pre-cracking when using CF. After applying the initial preloading at 28 days, ER decrements were around 18% for ECC1.2 and between 8.5% and 10% for CNT-ECCs and that for both partially saturated and dried states. These differences were probably due to the decreased crack width with CNT content. Although, the number of cracks increased when incorporation CNT, crack width was reported in literature as more important to the ER results than crack numbers (Siad *et al*. 2015). If considering the self-healing rate, all ECCs showed recovery in ER at all moisture states. However, because the absence of self-sensing of pre-cracking for CF-ECCs, only self-healing rates of CNT-ECCs in partially saturated and dried states can be considered for combined self-sensing/self-healing ability. Compared to sound specimens, the ER of ECC1.2, ECC0.25 and ECC0.5 at 28+90 days, were -9.4%, -3.9% and +1% in partially saturated state; and they were -9.2%, -2.7% and -0.2% in dried state. In addition to the high self-sensing ability of CNT-ECCs, the ER results confirmed also the improved self-healing rate with CNT content. The close relationship between the self-healing rate and tight crack widths can explain the improved recovery in case of CNT- ECCs.

Table 4. ER measurements of ECC specimens incorporating carbon-based conductive materials.

| Moisture State | Mix ID | | Curing time (days) and ER (Ω m) | | | | |
|---|---|---|---|---|---|---|---|
| | | | 28+0 | 28+14 | 28+30 | 28+60 | 28+90 |
| Saturated state | ECC1.2 | Sound | 135.9 | 193.7 | 248.3 | 429.4 | 446.4 |
| | | Preloaded | 111.6 | 166.7 | 217.4 | 386.3 | 403.8 |
| | ECCF0.5 | Sound | 25.6 | 33.8 | 54.4 | 66.5 | 70.8 |
| | | Preloaded | 28.4 | 33.8 | 51.9 | 66.9 | 79.5 |
| | ECCF1 | Sound | 7.9 | 23.3 | 18.8 | 17.2 | 20.4 |
| | | Preloaded | 7.4 | 20.8 | 17.5 | 14.6 | 20.8 |
| | ECCN0.25 | Sound | 103.9 | 179.9 | 265.8 | 326.9 | 360 |
| | | Preloaded | 108 | 177.8 | 262.4 | 328.6 | 369.1 |
| | ECCN0.5 | Sound | 90.9 | 147.3 | 208.3 | 268.4 | 307.4 |
| | | Preloaded | 93.8 | 150.2 | 212.8 | 271.2 | 327.5 |
| Partially-Saturated state | ECC1.2 | Sound | 164.4 | 215.4 | 257.8 | 445.4 | 463.5 |
| | | Preloaded | 135.4 | 185.3 | 229.4 | 400.5 | 419.8 |
| | ECCF0.5 | Sound | 28.6 | 41.5 | 60.1 | 75 | 76.4 |
| | | Preloaded | 32.9 | 40.9 | 58 | 73.6 | 73.5 |
| | ECCF1 | Sound | 8.9 | 21.5 | 20.5 | 21.8 | 22.3 |
| | | Preloaded | 9.4 | 22.1 | 22.3 | 22.8 | 23.4 |
| | ECCN0.25 | Sound | 115.6 | 207.5 | 338.6 | 338.3 | 365.7 |
| | | Preloaded | 104.8 | 192 | 321.9 | 326.9 | 351.2 |
| | ECCN0.5 | Sound | 103.1 | 219.1 | 297.4 | 307.1 | 352.5 |
| | | Preloaded | 92.8 | 205.1 | 285.6 | 307.5 | 356.2 |
| Dried state | ECC1.2 | Sound | 200.5 | 248.4 | 495.1 | 629.6 | 688.4 |
| | | Preloaded | 165.1 | 210.3 | 439.9 | 565.4 | 625.4 |
| | ECCF0.5 | Sound | 32.7 | 46.9 | 86.5 | 111.2 | 108.1 |
| | | Preloaded | 36.1 | 50.8 | 90.7 | 109.2 | 108 |
| | ECCF1 | Sound | 13.3 | 28.6 | 25.2 | 22.8 | 28.8 |
| | | Preloaded | 13.9 | 29.1 | 26.4 | 22.2 | 27.6 |
| | ECCN0.25 | Sound | 140.1 | 230.1 | 352.9 | 411.3 | 448.9 |
| | | Preloaded | 127.2 | 214.1 | 338.7 | 400 | 437.4 |
| | ECCN0.5 | Sound | 130 | 217.5 | 325.5 | 382 | 422.6 |
| | | Preloaded | 118.9 | 204.2 | 314.2 | 380.6 | 421.8 |

## 3 CONCLUSIONS

The following conclusions can be drawn from the results of this study:

- The use of CF improved the compressive and flexure strengths of sound ECC specimens, and significantly reduced their ductility. Although, the self-healing of CF-based ECCs was evident from the MOR and deflections results, and the ER of CF-ECCs enhanced with curing time, the absence of self-sensing of initial pre-cracking in ER results confirmed that the combined self-sensing/self-healing cannot be achieved by using CF.
- The incorporation of CNT into ECCs enhanced the compressive and flexure strength of sound specimens and showed better or equivalent ductility than the control ECC. Also, the self-healing rates of mechanical properties showed improvement with CNT content.
- The moisture state of specimens influenced considerably the ER and the self-sensing of pre-cracked ECCs. Unlike CF-ECCs, the self-sensing ability of initial pre-cracking was evident for CNT-ECCs tested at partially saturated and dried states, showing an improved self-sensing/self-healing ability when using CNT in ECCs.

This study provides a preliminary test results for multi-functional ECC with self-sensing and healing capabilities. However, for a complete understanding of behavior of multi-functional material, it will be necessary to conduct further research on this subject.

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