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Medium-term self-healing evaluation of Engineered Cementitious Composites with varying amounts of fly ash and exposure durations

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- The medium-term healing allowed ECCs to effectively regain strength and stiffness.
- High-fly-ash ECCs continued moderate healing beyond the age of 270 days.
- The major components of the medium-term precipitation were CaCO3 and C-S-H.
- The recovered strain capacity of ECCs increased along with the amount of fly ash.

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ABSTRACT

Extensive studies have shown that young Engineering Cementitious Composites (ECCs) have the potential to achieve effective self-healing. The present study investigates the medium-term self-healing performance of cracks in ECCs that are relevant in the medium and long-term stages of the material service life. For this purpose, the prepared ECC specimens are pre-cracked at an age of 180 days. The major experimental variables are the weight fraction of fly ash in ECCs (a fly ash to cement ratio of 1.2, 1.6, or 2.0) and the healing duration (7, 28, or 90 days). The medium-term self-healing performance is quantified using a resonant frequency test followed by a uniaxial tensile test. In addition, scanning electron microscopy and energy dispersive X-ray analyses are employed to observe the micro-structure of the healed crack and identify the medium-term healing product, respectively. The results suggest that as long as water is present in the environment, ECCs have moderate medium-term self-healing ability, and can partially recover their tensile mechanical properties. In particular, effective medium-term self-healing performance can be achieved within 90 days of conditioning for ECCs with a pre-strain of less than 1%. © 2016 Elsevier Ltd. All rights reserved.

1. Introduction

High performance fiber reinforced concrete (HPFRC) is characterized by tensile strain-hardening behavior accompanied by closely spaced hairline cracks [1–5]. Depending on the properties of the matrix and fibers used, the ultimate tensile strain of HPFRC materials ranges between 0.3% and 7%, which is significantly larger than the 0.01%–0.02% failure strain of normal concrete. When HPFRC is under compression, the short discontinuous fibers, similar to stirrups in RC members, can provide a confining effect to increase the compressive ductility and residual strength. Many researchers have explored applications of HPFRC to improve concrete structures from various perspectives, such as simplification of steel detailing [6,7], strengthening of earthquake-resistance

* Corresponding author. E-mail address: cchung@mail.ncku.edu.tw (C.-C. Hung). behavior [7–10], and enhancement of sustainability [11–13]. Engineered Cementitious Composites (ECCs) are a unique class of HPFRC materials. They have an extraordinary tensile strain capacity, which is commonly reported to be more than 2%. In addition, ECCs have an intrinsic tight crack-width control property, which limits the crack width to be less than 60 μ m prior to failure [1,2].

In recent years increasing attention has been drawn to the potential of ECCs to achieve effective self-healing. Similar to normal concrete materials, the self-healing process in ECCs is generally due to a combination of complex physical and chemical mechanisms [14], such as the formation of calcium carbonate or calcium hydroxide crystals, swelling of C–S–H gels, further hydration of unreacted cementitious materials, and cracks filled by impurities within water or loose concrete particles. ECCs have better self-healing efficiency than conventional concrete materials, and this is mainly because the remarkable crack width control ability of such materials requires fewer newly grown healing crystals





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to fill the cracks [15]. In addition, ECCs have a higher percentage of cementitious components and a lower water-binder (w/b) ratio than traditional concrete materials, which allows more unhydrated cementitious materials to react in the self-healing process.

Li and Yang [15] and Yang et al. [16–18] studied the self-healing behavior of ECCs under various environmental conditions, and concluded that the presence of water was a critical factor affecting the self-healing performance of these materials. It was also indicated in [15] that a critical criterion for self-healing to occur in concrete materials was to have a crack width less than 150 µm. In particular, more satisfactory self-healing performance could be achieved when the crack width was less than 50 μ m. Yang et al. [16] found that early-age (3 days) ECCs could show a robust self-healing ability for recovering material stiffness when their pre-strain was less than 0.3%. When the pre-strain was increased from 00.3% to 3%, the recovery in stiffness decreased significantly from 100% to merely 10%. Furthermore, Yang et al. [16] found that the major healing precipitation in the cracks with a width of less than 20 µm was C-S-H gels while that in the cracks with a width of greater than 50 µm was a mixture of C-S-H gels and calcite particles. Özbay et al. [19] and Zhang et al. [20] evaluated the self-healing performance of ECCs via splitting tensile tests, rapid chloride permeability tests (RCPTs), and water absorption tests. It was shown by Zhang et al. [20] that the use of a higher volume of fly ash in ECCs (e.g. a fly ash/cement ratio of 4.0) made the matrix more porous. In addition, it leaded to a smaller crack width, thus enhancing the self-healing ability of ECCs. It was also indicated in [20] that the major self-healing precipitation in the ECCs with a high fly ash/ cement ratio was C-S-H and calcium carbonate. Şahmaran et al. [21] assessed the self-healing performance of ECCs incorporating different supplementary cementitious materials via RCPTs. It was found that the type of the reaction products, as a result of different supplementary cementitious materials, had significant effects on the self-healing capability of ECCs.

Huang et al. [22] showed that the healing process in young cement pastes slowed down markedly after 300 h. More recently. Huang et al. [23] indicated that during the self-healing process. the water in the crack migrated into concrete through the crack surfaces, facilitating additional hydration of unhydrated cement. As a result, the layer of the bulk paste adjacent to crack surfaces became denser. While the densified microstructure adjacent to the crack restrained the diffusion of reaction products from the bulk paste into cracks, it also decreased the ingress of aggressive agents into the bulk concrete matrix. Moreover, Hilloulin et al. [24] showed that satisfactory recovery in bending strength and stiffness could be obtained within 2 weeks for young cement paste with a crack width of less than 10 μ m. They also suggested that the gradual strength regains were due to the development of portlandite and C-S-H whereas the relatively fast stiffness regains were a result of crack bridging by ettringite and CA(S)H phases as well as small quantities of C-S-H and portlandite.

It is worth noting that most existing studies focus on the selfhealing behavior of relatively young ECCs that are pre-damaged at an age of 28 days or less. There is thus still a significant gap in knowledge about the lifetime self-healing performance of ECCs. In particular, many cracking occurrences are relevant in the medium stage of concrete service life, due to, for example, seasonal temperature variations, freeze/thaw cycles, external restraints, creep, and accidental overloads. The present study investigates the medium-term self-healing performance of ECCs, and the major experimental variables are the weight fraction of fly ash and the healing duration. Multiple approaches are employed to qualify and quantify the medium-term self-healing performance, namely, direct tensile tests, resonant frequency tests, scanning electron microscopy (SEM), and energy dispersive X-ray (EDX) analyses.

2. Specimen preparations

The details of the ECC specimens, including the materials, mixture design, curing procedure, and self-healing conditioning, are summarized below.

2.1. Materials

Table 1 presents the material components and their relative weight proportions in the prepared ECC specimens. The w/b ratio of all mixtures is 0.32. Mixtures H12, H16, and H20 have fly ash to cement ratios of 1.2, 1.6, and 2.0, respectively. H12 is the typical ECC mixture that has been widely studied in the literature. The chemical composition of the fly ash is given in Table 2. The silica sand used in this work has a maximum particle size of 0.2 mm. All mixtures have a 2% volumetric fraction of polyvinyl alcohol (PVA) fibers. The length and diameter of the PVA fibers are 12 mm and 39 μ m, respectively. The fibers have a density of 1300 kg/m³, an elastic modulus of 41 GPa, a tensile strength of 1600 MPa, and a maximum elongation ratio of 6%.

2.2. Mixing, casting, and curing

All powders are pre-mixed in a Hobart mixer. Half of the total fluid containing water and superplasticizer is first added to the mixer and sufficiently mixed. The PVA fibers along with the remaining fluid are then added to the mixture and mixed until there is a uniform distribution of materials. The fresh mix is cast into molds and covered with plastic sheets. The specimens are demolded after 24 h and water cured at room temperature $(23 \pm 2 \,^{\circ}\text{C})$ for 28 days. They are then left to air cure in the laboratory environment with $60 \pm 5\%$ RH and $23 \pm 2 \,^{\circ}\text{C}$ until the age of 180 days. The ECC specimens have dimensions of 240 mm × 60 mm × 15 mm.

2.3. Pre-cracked specimens and exposure conditions

Fig. 1 shows the experimental procedure for evaluation of the medium-term self-healing performance of ECCs. At the age of

 Table 1

 Mixed proportions of the specimens (proportion by weight).

| Mixture | Type I Portland cement | Class F fly ash | Silica sand | Water | Polycarboxylate- based superplasticizer | PVA fibers (volume fraction) |
|---------|------------------------------|--------------------|----------------|-------|---|------------------------------------|
| H12 | 1 | 1.2 | 0.8 | 0.68 | 0.025 | 2% |
| H16 | 1 | 1.6 | 0.8 | 0.81 | 0.025 | 2% |
| H20 | 1 | 2.0 | 0.8 | 0.94 | 0.025 | 2% |

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| Chemica | l compositions | of | fly | ash. |
|---------|----------------|----|-----|------|
|---------|----------------|----|-----|------|

| Compound (%) | Class F | FA |
|---------------------------------|---------|-------|
| CaO | <5 | 4.37 |
| SiO ₂ | | 64.69 |
| Al ₂ O ₃ | | 19.03 |
| Fe ₂ O ₃ | | 8.34 |
| P ₂ O ₅ | | 0.01 |
| K ₂ O | | 2.01 |
| TiO ₂ | | 0.86 |
| MgO | <5 | 2.31 |
| Na ₂ O | | 1.22 |
| SO ₃ | <5 | 0.12 |
| NH ₄ ⁺ | | 0.07 |
| $SiO_2 + Fe_2O_3 + Al_2O_3$ (%) | >70 | 92.06 |
| water content | <3 | 0.07 |
| loss of ignition | <12 | 2.56 |

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