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INFLUENCE OF SUPERABSORBENT POLYMER PARTICLES ON THE SELF-HEALING BEHAVIOR OF ENGINEERED CEMENTITIOUS COMPOSITES

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Abstract: Self-healing behavior of engineered cementitious composites (ECC) with superabsorbent polymers (SAPs) was investigated in this paper. Four point bending tests were used to precrack ECC specimens at the age of 7 and 28 days, respectively. The precracked samples were then cured in 99%RH curing and 99%RH/room air cycle curing to promote the occurrence of self-healing behavior and in room air curing for control. The addition of SAPs help tighten the crack width, resulting in enhanced flexural stiffness for the 99%RH cured and 99%RH/room air cycle cured samples. Furthermore, the effect of SAPs also promotes a larger degree recovery of deformation capacity, despite of much lower the initial deformation capacity for ECC2. The self-healed samples were observed by environment scanning electron microscope (ESEM) to reveal the products of self-healing behavior.

1 INTRODUCTION

Self-healing of concrete has been observed for many years. For civil infrastructures, for example bridge decks and pavements, where water and CO_2 are available, concrete can produce the chemical products to heal itself damage. Self-healing is mainly attributed to the formation of calcium carbonate, which is a result of calcium ion in concrete and CO_2 dissolved in water [1,2]. Continuous hydration of unhydrated cementitious materials is another intrinsic self-healing mechanism [3]. Due to the limited of self-healing products, the crack width of the cementitious materials has been found to be essential for intrinsic self-healing to occur [4,5]. The crack width required to promote self-healing roughly below 100 μm, especially lower than 50 μm for self-healing of cementitious materials [4,6]. The control of crack width is therefore one important criterion for attaining intrinsic self-healing in concrete.

Engineered Cementitious Composites (ECC) is a special type of High Performance Fiber Reinforced Cementitious Composites (HPFRCC), which micromechanically designed to achieve high damage tolerance under normal service conditions [7]. It is a new material, with high ductility under uniaxial tensile loading, and improved durability due to multiple micro-cracking and tight crack width. A typical uniaxial tensile stress-strain curve is shown in Fig.1. The crack width development is also shown in this figure, which presents that crack width increase up to 60 μm at 1% strain. Between 1% and 4% strain, the crack width stabilizes and tends to remain constant at 60 μm, with the number of cracks increase [9].

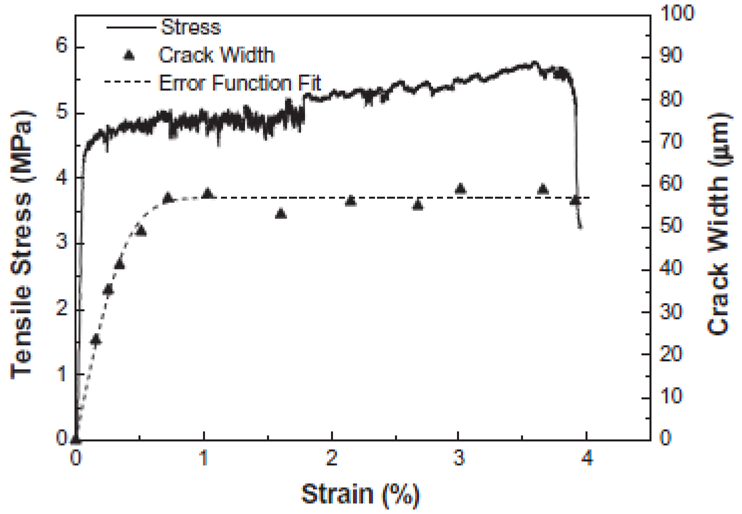


Figure 1: Typical uniaxial tensile stress-strain-crack width curve of ECC [8] showing high tensile ductility and tight crack width.

Recently, various alternative materials were used in research of self-healing concrete. The self-healing potential of cement-based materials with calcium sulfoaluminate based expansive agents used as a cement replacement material were investigated by Hosada et al.[10], Kishi et al.[11] and Sisomphon and Copuroglu [12]. Jaroenratanapiron and Sahamitmongkol [13] evaluated the self-healing performance of mortars with fly ash, silica fume and a crystalline admixture. Kim and Schlangen [14] analyzed the feasibility of Super Absorbent Polymers (SAPs) to stimulate self-healing in ECC.

In an effort to enhance the self-healing potential of cement-based materials, ECC mixtures incorporating SAPs were evaluated. The influence of curing condition and precracking time on the self-healing behavior of ECC with SAPs was investigated. Three different curing conditions were employed in this investigation, including 95%RH/room air cycle curing (CR1), 95%RH curing (CR2) and room air curing (CR3). The precracking time varied from 7 days to 28 days in order to reveal the effect of timing of damage on the self-healing behavior of ECC with SAPs. In the following sections, the test program will be introduced in details, including material preparation and four-point bending test. Furthermore, test results on mechanical behaviors, such as deformation capacity, flexural strength, stiffness and crack pattern will be presented and discussed. Finally, the self-healing process and self-healing products will examined by environment scanning electron microscope (ESEM).

2 OUTLINE OF THE RESEARCH AND EXPERIMENTAL SET UP

2.1 Outline of the research

In order to study the effect of SAPs on the self-healing behavior of ECC, four-point bending test (FPBT) was adopted. The overall program for FPBT was shown in Fig. 2. In total two different mixtures were prepared: one control mixture without SAPs, another mixture containing SAPs for 4%. The next step was the preparation of the specimens. The coupons were evenly cut into 4 species and then ground in order to attain a smooth surface. Afterwards, the specimens were divided into 2 main categories: a) the specimens cured for 7 days and b) those cured for 28 days after casting under standard curing conditions. Seven days after casting some of the specimens were tested until final failure while others were preloaded up to vertical deformation of 3 mm respectively using FPBT. Some of the preloaded samples were then cured in three curing conditions (CR1, CR2 and CR3)) for 10 cycles. After the curing period, the CR1 cured samples were put under ESEM in order to ascertain the extent of self-healing and self-healing products. Finally, the CR1, CR2 and CR3 cured specimens were then tested up to final failure by FPBT. The same procedure was followed also for the samples that cured for 28 days after casting.

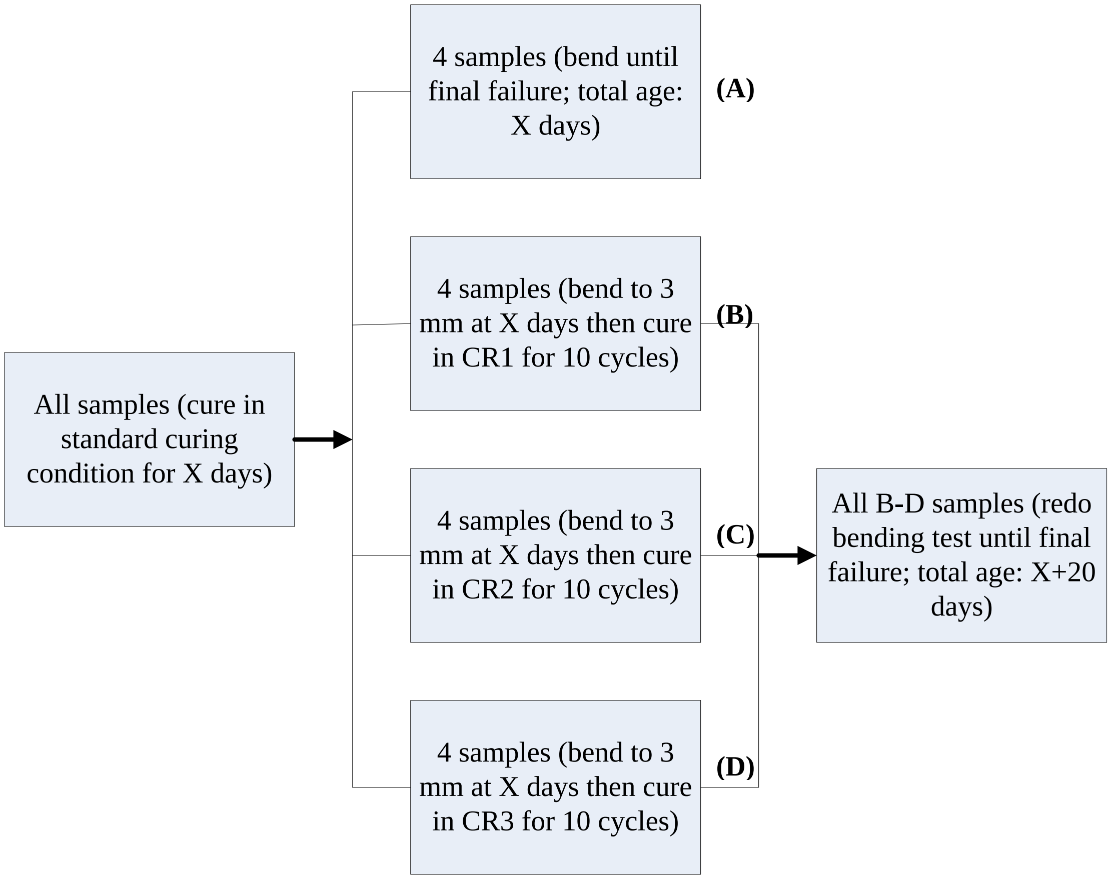


Figure 2: Bending test program of self-healing ECC (X: pre-cracking time, varies from 7 to 28 days).

2.2 Material design and preparation of the specimens

The mixture proportions of ECCs used in this paper are presented in Table 1. The ECC1 is used as a reference in the ECC mix design. SAPs are mixed in a proportion of 4% by weight of cement. The dimensions of the fibers that were used for mixtures are given in Table 2.

Table 1: Mix proportion of ECCs with SAPs (mix by weight (fiber by volume))

Mixture ID	Cement	FA	Sand	Water	SAP	Superplasticizer	Fiber	W/B
ECC1	1.0	1.5	0.9	0.61	0	0.02	2%	0.25
ECC2	1.0	1.5	0.9	0.61	0.04	0.02	2%	0.25

Table 2: Properties of PVA fiber

Diameter (μm)	Length (mm)	Tensile strength (MPa)	Modulus (GPa)	Density (g/cm^3)
39	12	1620	42.8	1.2

The solid materials, including cement, fly ash, sand and SAPs, were first mixed with a mortar mixer for 2 min, followed by the addition of water and Superplasticizer. Mixing continued at low speed for 5 min and then at high speed for 2 min. After fibers were added, the material was mixed at high speed for another 5 min. The fresh ECC was cast into molds and then demolded after 24 hours curing. The specimens were then cured under standard curing condition (20 ℃, 95% RH) until testing. The bending specimens have dimensions of 400x70x16 mm. Especially, for the samples that had to be examined under the ESEM a particular preparation was followed. After pre-loading at 7 days, bending specimens which containing crack width of 30 μm were cut into 5x5x5 mm cubic samples. After start imaging, the cubic samples were continued curing under CR1 until tested time.

Three different curing regimes were used to simulate different RH environment exposures: a) CR1 (95%RH/room air cycle): Pre-cracked ECC specimens were stored in a 95%RH curing condition at 20 ℃ for 24 h, and then cured in room air at 20±1 ℃, 50±5% for 24h; b) CR2 (95%RH): Pre-cracked ECC specimens were stored in a 95%RH curing condition at 20 ℃ for 48 h; c) CR3 (room air): Pre-cracked ECC specimens were exposed to room air at 20±1 ℃, 50±5% for 48 h.

After curing, the coupon specimens were used in FPBT. The full span of FPBT was 300 mm with the middle span of 100 mm. The FPBT was conducted under displacement control of 1 mm/min. Typically it takes about 20-30 min before the specimens exhaust its deflection capacity and fail.

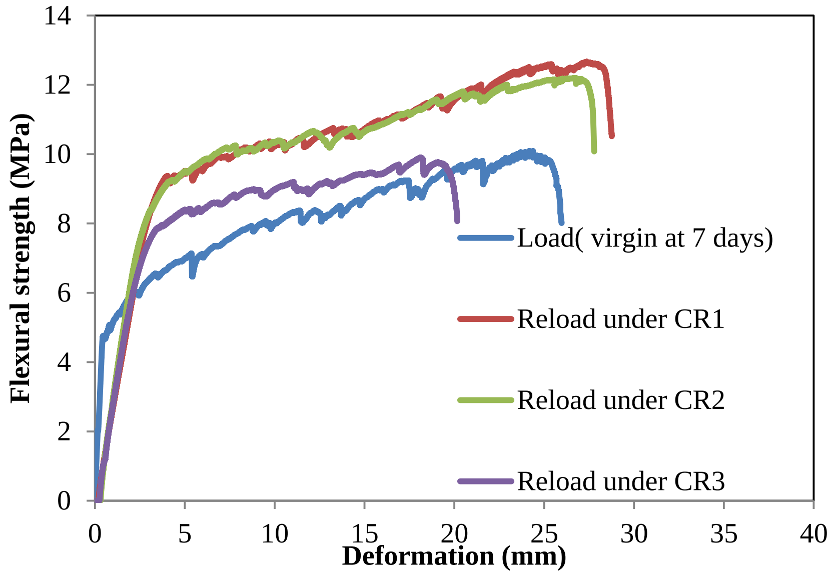
3 RESULTS AND DISCUSSION

3.1 Four point bending test

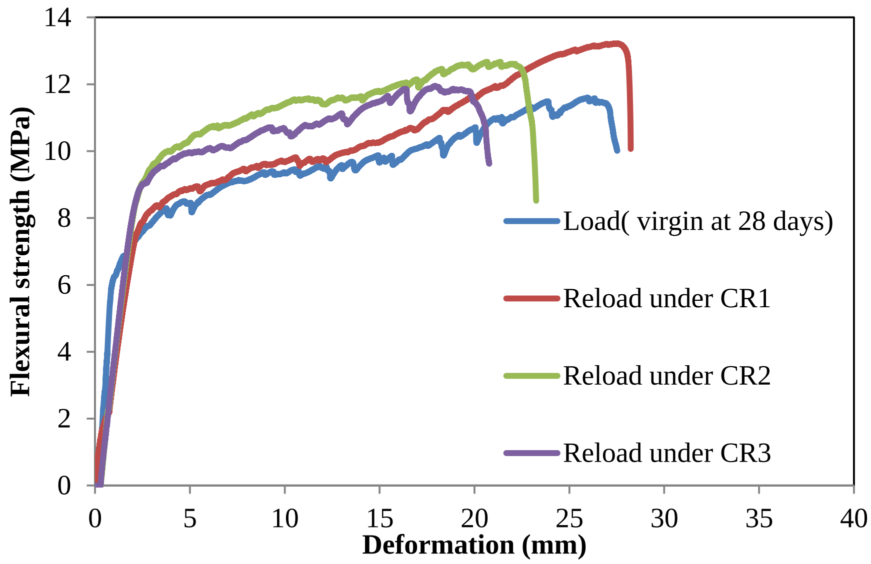
It includes two categories for each mixture: one was tested at 7 days after casting, others tested after 10 cycles curing. Each category contains four basic schemes regarding the curing method as follows: 1) the virgin specimens were tested until final failure; 2) the preloaded and then cured in CR1 for 10 cycles; 3) the preloaded and then cured in CR2 for 10 cycles; 3) the preloaded and then cured in CR3 for 10 cycles. The flexural strength-deformation curves of ECC2 are shown in Fig.3.

From test results, it can be seen that the flexural strength of 28 days specimens under four curing schemes (virgin, CR1 cured, CR2 cured and CR3 cured) is higher than that of the 7 days specimens, which is what was expected considering the fact that the maturity of specimens was higher for 28 days case, and the materials has fully developed its ultimate load capacity after curing for 10 cycles. And the deformation of ECC2 cured under all conditions is higher than ECC1, which may be explained that SAPs act as artificial flaws to trigger initiative crack easily occurred in matrix, which can help ECC specimens to realize pseudo strain-hardening performance [15]. From Fig.3 (a) and (b), it can be observed that the deformability of ECC2 is relatively well after incorporating SAPs. The recovery ratio of deformability after pre-cracking is larger compared with that of the

ECC1.



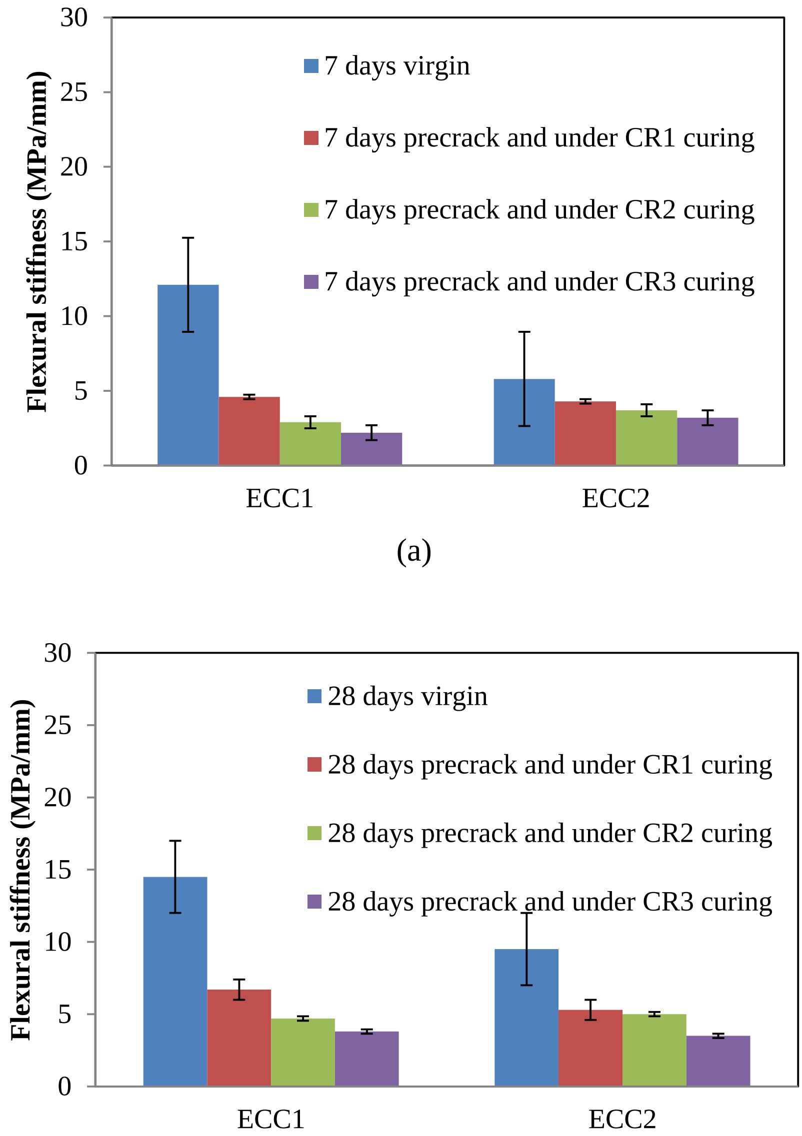
(a)



(b)

Figure 3: Comparison of typical flexural behavior of ECC2.

The stiffness is defined as the slope of the initial linear part of flexural strength-deformation curves. As shown in Fig.4, the stiffness of the CR1 cured specimens is higher than that of others. It can be explained that the fiber bridging capacity could be enhanced due to more additional bonding between healing products and fiber surface since the presence of SAPs and the healing products are more intense for specimens cured in CR1 than that in CR2 and CR3. On the contrary, the stiffness of the CR1 cured specimens has a lower value comparing with that of the virgin ones both in 7 and 28 days. This is expected, because calcium hydrates and/or calcium carbonate, which are usually the main components of healing products, are weaker than hydrated calcium silicate (CSH) produced from cement hydration.



(a)

(b)

Figure 4: Comparison of flexural stiffness of different mixtures at different precracking time (a) 7 days and (b) 28 days**.**

3.2 Crack width

It is seen that specimens of all ECCs exhibit multiple micro-cracking behavior under FPBT, as is shown in Fig.5. The average crack width for ECC is shown in Fig.6. It can be seen clearly that the crack width is much smaller for ECC2 compared with ECC1, which may be explained by the effect of SAPs on residual crack width of ECC specimens. Tight crack width in ECC specimens before rupture is beneficial to the self-healing behavior, especially for the recovery of flexural stiffness.

Figure 5: Multiple cracking along the length of the ECC specimen**.**

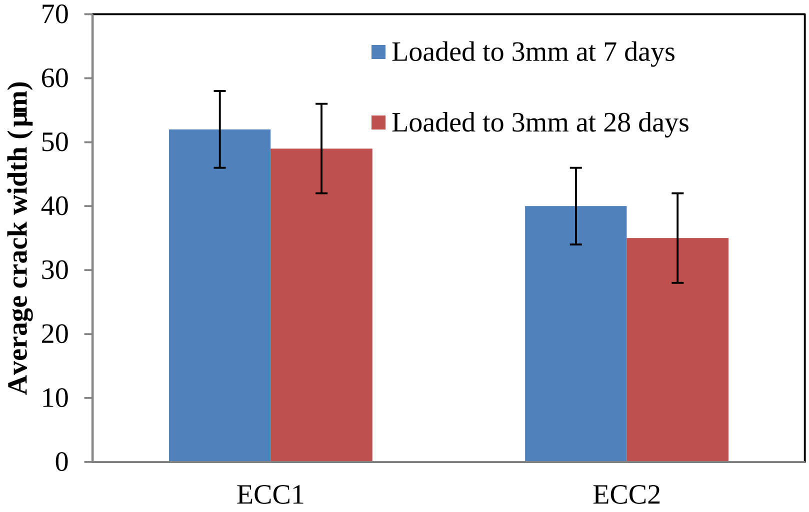
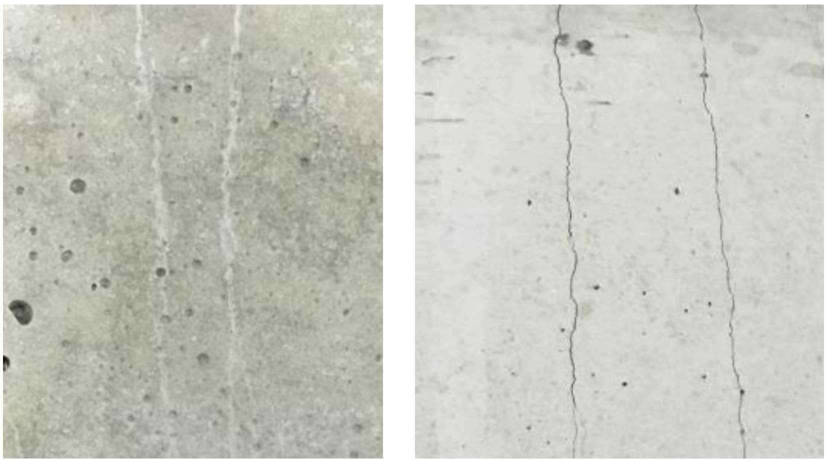


Figure 6: Comparison of average crack width**.**

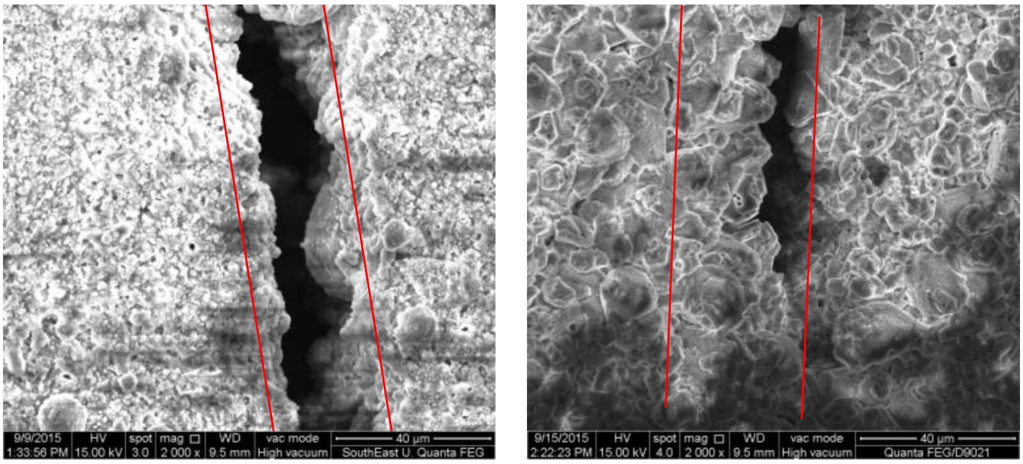
3.3 Self-healing behavior

Fig.7 shows that self-healing behavior occurred in cracks of ECC specimens under different curing conditions. It can be clearly seen from Fig.7 (a) that abundant white residue crystals present along the crack lines after CR1 cycles under naked eyes. All CR1 cured specimens showed the similar white residues inside the crack, while there are no signs of self-healing for specimens under CR3 curing as shown in Fig.7 (b). Therefore, the curing regime after pre-cracking is critical to the self-healing behavior for ECC specimens.



(a) (b)

Figure7: (a) Healed specimen after CR1 curing; (b) Unhealed specimen after CR3 curing.



(a) (b)

Figure 8: (a) CR3 cured specimen shows no sign of healing; (b) CR1 cured specimen shows healing products fill the crack.

Fig.8 (b) shows a typical pattern of a partially healed crack. It can be observed from this picture that the self-healing products grow along the crack lines from left to right. The products can be recognized by their morphology. For example, the crystals that are sticking out of the crack are met in various sizes and they are most probably calcium carbonate crystals generated by the carbonation of calcium hydroxide. This phenomenon appears in the presence of CO2 in air, the water from SAPs manages to take moisture out of a humid environment and the calcium met coming either from cement or limestone. In addition, the self-healing behavior of ECC incorporating SAPs is faster than that of without SAPs. It can be seen from Fig.9 (b) that healing products of ECC2 completely fill the crack for 3 CR1 cycles, while that of ECC1 partially fill the crack for same cycles. The moisture uptake by SAPs seems to be enough to promote self-healing.

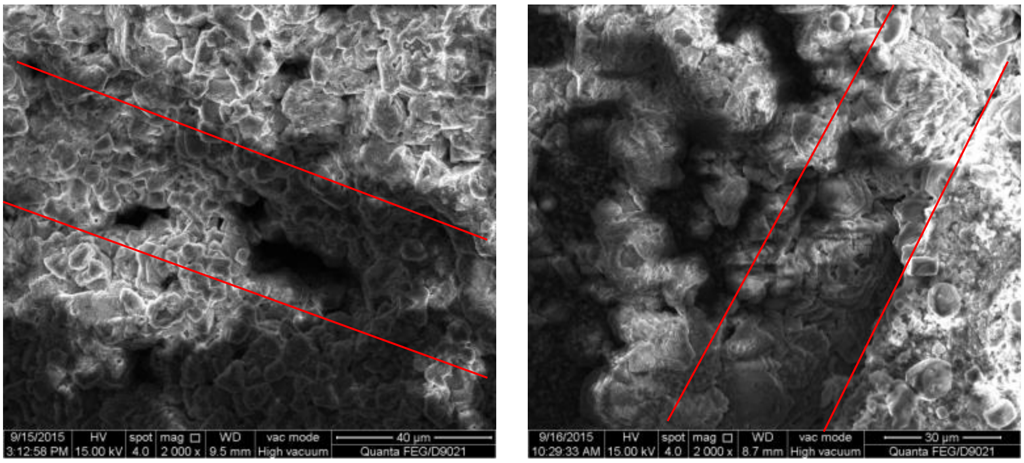
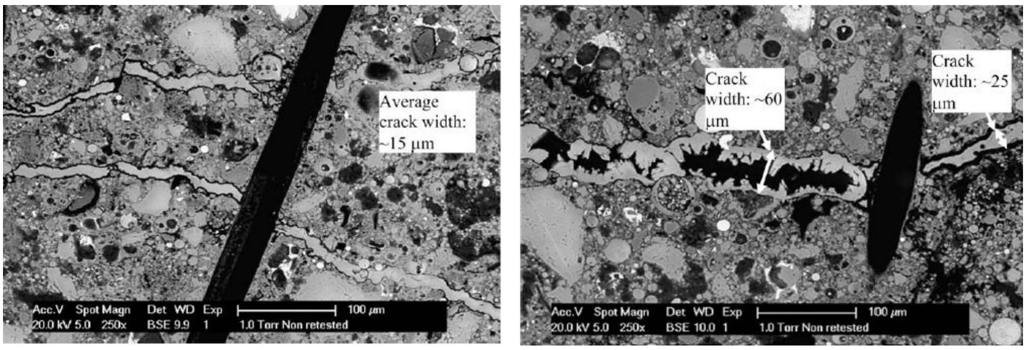


Figure 9: (a) Partial crack healing (ECC1); (b) Saturated crack healing (ECC2).

The effect of crack width on self-healing behavior was shown in Fig.10 [5]. It can be observed that cracks can be fully healed when crack width is 15 μm and 25 μm, while it only partially healed when crack width is 60 μm. Similarly, Fig.9 shows that partially healed for ECC1, while it was completely healed for ECC2 with 30 μm crack. It demonstrated that the crack width is critical factor to the extent of self-healing behavior. Hence , self-healing is much easier to occur in ECC2 than ECC1 due to the presence of SAPs which not only help ECC attain a tighter crack width but also can take water out of high moisture environment for it. The addition of SAPs in ECC2 is beneficial to self-healing behavior. It demonstrates the feasibility of mixing SAPs can enhance the capacity of self-healing.



(a) (b)

Figure 10: (a) Full crack healing after water curing; (b) Partial crack healing after water curing.

CONCLUSIONS

Self-healing behavior of pre-cracked ECC with SAPs and local waste material (Fly ash) is investigated in this paper. Four point bending test is used to pre-loading ECC specimens deformation up to 3 mm with subsequent curing in CR1, CR2 and CR3 for 10 cycles. The majority of specimens cured in CR1 showed greatly recovery of mechanical properties comparing with that cured in

CR3. The observations using ESEM further confirmed the result from bending test. The following conclusions can be drawn from this study:

(1) Self-healing behavior is observed when the pre-cracked specimens are cured in CR1 and CR2. Flexural test results show specimens cured in CR1 and CR2 have a certain extent of recovery of the mechanical properties. On the other hand, specimens cured in CR3 revealed no such effect. Furthermore, in many cases the performance after CR3 curing even get worse, denoting that cracks exposed in air won't make self-healing behavior due to the lack of water from low humid environment which is necessary to self-healing.

(2) Mixing SAPs into ECC can lower the crack width further, which is beneficial to self-healing; consequently, enhance the recovery of flexural stiffness. What's more, SAPs can contribute to the internal healing of a crack by absorbing fluids from the surroundings.

(3) The age of the pre-loading influences ECC's mechanical recovery. The recovery level of pre-loading at 7 days case is higher than that at 28 days case. It may be explained to the fact that at 7 days after casting, there remains abundant unhydrated cementitious material to initiate continuous hydration during the period of self-healing. On the other hand, the extent of hydration for 28 days case is so higher that there is little active substance left.

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