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Autogenous healing of engineered cementitious composites at early age

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ABSTRACT

Autogenous healing of early ages (3 days) ECC damaged by tensile preloading was investigated after exposure to different conditioning regimes: water/air cycles, water/high temperature air cycles, 90%RH/air cycles, and submersion in water. Resonant frequency measurements and uniaxial tensile tests were used to assess the rate and extent of self-healing. The test results show that ECC, tailored for high tensile ductility up to several percent and with self-controlled crack width below 60 μm, experiences autogenous healing under environmental exposures in the presence of water. However, the recovery for these early age specimens is not as efficient as the recovery for more mature specimen, for the same amount of pre-damage and exposure to the same environment. Even so, the self-healing for these early age specimens demonstrates high robustness when the preloading strain is limited to 0.3%. This conclusion is supported by the evidence of resonant frequency and stiffness recovery of the healed ECC materials.

1. Introduction

High strength concrete with high cement content and a low water/cement ratio has been developed with the availability of new generations of high-range water reducing admixtures in recent years. High strength concrete is expected to improve the structural performance and durability of reinforced concrete structures due to a dense microstructure that lowers water permeability and limits access of corrosives to steel reinforcement [1]. However, several investigations [2,3] revealed that high strength concrete structures experience early age cracking due to the increase of autogenous shrinkage and concrete brittleness. Early age cracks can have negative impacts on durability and the service life of reinforced concrete infrastructure. This has heightened concerns about early age cracking in high strength concrete in recent years and remains a technical challenge to the practice and research communities.

Several different strategies have been employed to control early age cracking of high strength concrete [4]. For structural engineers, efforts have been placed on detailing the rebar reinforcement, and increasing the reinforcing ratio and the arrangement of transverse or confinement reinforcement. For materials engineers various kinds of fibers, fiber mesh, shrinkage reducing agents, and curing admixtures have been adopted for crack control. These approaches are effective in certain situations but their efficacy and consistency remain to be proven in others. Therefore, it is highly desirable to develop concrete materials that automatically regain lost performance in mechanical and permeability properties after early age cracking. Such self-healing concrete should reduce repair needs and extend structural service life.

Self-healing of cracked concrete is an often-studied phenomenon. Two strategies for the promotion of self-healing have proven promising. One approach focuses on the embedment of capsules that contain self-healing compounds within the concrete material [5,6], while the other relies on a continuous dispersion of self-healing compounds (i.e., free calcium ions or unhydrated cement particles) intrinsic to the concrete matrix. The latter, often referred to as autogenous healing, has various advantages over encapsulation self-healing [7], including economics, which is extremely important for the highly cost-sensitive construction industry. Autogenous healing is the research focus of the present study.

Previous researchers have studied the necessary conditions for autogenous healing in concrete materials. These studies have resulted in identifying three general criteria critical to robust self-healing: the presence of specific chemical species [8–14], exposure to various environmental conditions [12,14–20], and small crack width [8,9,15,21–23].

Autogenous healing can occur in a variety of environmental conditions ranging from underwater immersion to cyclic wet–dry exposures. These conditions are practical for many infrastructure types, particularly for transportation infrastructure. Hence this criterion for robust self-healing can be easily satisfied.

The second criteria — the need for adequate concentrations of certain critical chemical species, is also readily satisfied, at least for concrete mixes with high cement content, due to the presence of a large amount of unhydrated or partially hydrated cement grains. As well, the presence of CO_2 in air and NaCl in seawater and deicing salt are elements that support self-healing.

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The third criterion, the control of crack width to below 150 μm and preferably below 50 μm, represents the most formidable challenge in the design and implementation of self-healing concrete materials [7,8,15,18–21,23–25]. While artificial control of crack width to less than 150 μm is feasible in the laboratory using feedback control load-frames, reliable control of the crack width in concrete structures in the field in a consistent manner has not been attained. This explains why reliable autogenous healing in concrete structures is not typically realized in actual reinforced concrete structures.

Engineered Cementitious Composite (ECC) is a unique type of high performance fiber-reinforced cementitious composite, featuring high tensile ductility with moderate fiber volume fraction (2 vol.% or less). The tensile strain capacity of ECC is 2–5%, several hundred times that of normal concrete. The compressive strength of ECC ranges from 50 to 80 MPa depending on mix composition, putting ECC in the class of high strength concrete materials, but without the associated brittleness. Of special relevance to the present study is the capability of ECC materials to deform to high tensile strains while maintaining very tight crack widths, as shown in Fig. 1. Crack width in this ECC stabilizes at roughly 60 μm when the tensile strain exceeds about 1%, and does not increase further until fracture localization, when the strain capacity is exceeded. ECC with self-controlled crack width as low as 20 μm have been developed [26]. Prior to exhausting the tensile strain capacity, increased deformation is accommodated by an increasing number of microcracks, although the average microcrack width remains essentially constant. This is in contrast to the continuous widening of a single crack in an ordinary concrete specimen (with or without fiber) loaded in uniaxial tension in the tension-softening stage.

The tight crack width of ECC is an inherent material property similar to the compressive strength or the elastic modulus, as it is independent of loading, structure size and geometry, and steel reinforcement ratio. The tight crack width of ECC is a feature of the deliberately tailored fiber–matrix interaction within the material [27]. Because the tight crack width is an inherent property this behavior is the same for specimens in the laboratory or for full-scale structures in the field, even at early ages. This has been confirmed in bridge deck patch repair using ECC [28], where crack widths in ECC monitored from early age to several years remain below 50 μm. Based on the observations mentioned, damaged ECC materials with tight crack widths may be expected to readily undergo autogenous healing.

The hypothesis regarding the self-healing of damaged early age (3 days) ECC mentioned is examined in detail in this paper. A companion study conducted by the authors confirmed self-healing in ECC at a mature age of 6 months when exposed to wetting and drying cycles [29]. The objective of the research reported in the present paper is to experimentally investigate the rate and extent of self-healing under a broader set of environmental exposure conditions that simulate those commonly encountered by civil infrastructure, and focuses in particular on early ages. The effect of different pre-damage levels on the extent of self-healing is also evaluated. Finally the differences and similarities in healing behavior of early age ECC and that of more mature ECC are analyzed.

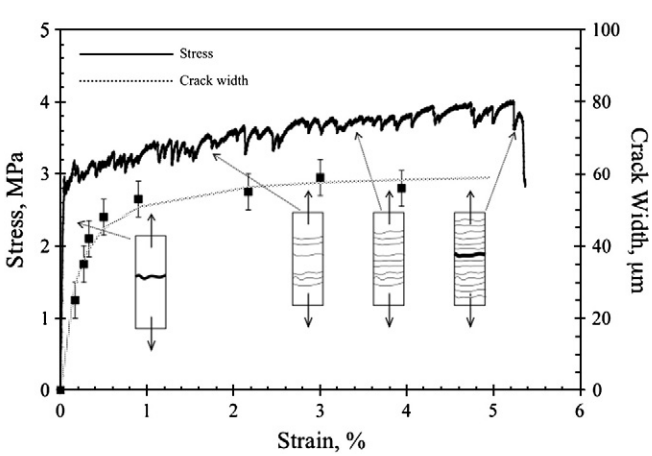

Fig. 1. Typical tensile stress–strain–crack width curve of ECC.

Table 1
Mix proportion of ECC.

Material	Cement	Aggregate	Fly ash	Water	HRWR	Fiber
Unit weight, kg/m^3	578	462	694	319	17	26

2. Experimental investigation

2.1. Mix proportion and specimen preparation

The mix proportions of the ECC material are given in Table 1. The ECC material utilized for these studies has a tensile strain capacity of 3% and steady state crack width of about 60 μm. To prepare the ECC material, Type I ordinary Portland cement, fine silica sand with 110 μm average grain size, Class F normal fly ash from Rockdale, Texas, 2 vol.% polyvinyl alcohol (PVA) fibers (Kuralon-II REC-15), and a polycarboxylate-based high-range water reducer (ADVA® Cast 530) were used. The fly ash conforms to ASTM C618 requirements. The PVA fibers are 12 mm long with an average diameter of 39 μm. The nominal tensile strength and density of the PVA fiber are 1600 MPa and 1300 kg/m^3, respectively. Due to the strongly hydrophilic nature of PVA fiber, the fiber is coated with an oiling agent by the manufacturer (1.2% by weight) in order to reduce the fiber/matrix interfacial chemical bond strength. This decision was made through ECC micromechanics material design theory and has been experimentally demonstrated to be effective for maximizing tensile ductility in ECC in previous investigations [30,31].

A force-based Hobart mixer with 20 L capacity was used in preparing a single batch of ECC material. The fresh mixture was then cast into coupon molds measuring 300 × 76 × 12.5 mm. The fresh ECC specimens were covered with plastic sheets and demolded after 24 h. After demolding, specimens were air-cured at 50 ± 5%RH and 20 ± 1 °C for 2 days.

Direct uniaxial tensile tests were then conducted on the 3rd day to pre-damage ECC specimens at specific tensile deformation, i.e., 0.3%, 0.5%, 1%, 2%, and 3%. The smallest of these imposed damaging strains, 0.3%, is 30 times larger than the failure strain of normal concrete materials (0.01%).

When the tensile strain reached the predetermined values, the tensile load was released and the specimens were removed from the tensile test machine to prepare for cyclic wet–dry exposure. Upon unloading crack widths were measured using an optical microscope. Due to the high tensile ductility of ECC, these specimens remain in the strain-hardening stage, displaying microcrack damage but no localized large cracks. Table 2 shows the number of microcracks and their corresponding average and maximum crack widths over a gauge length of 100 mm. It can be seen that that while the crack number increases significantly with increasing imposed strain, the average crack width increases only modesty. The maximum crack width, however, does increase markedly with increasing imposed strain.

Table 2
Crack width and number of pre-cracked ECCs.

Tensile strain,%	0.3	0.5	1	2	3
Number of cracks	5	7	16	24	41
Average crack width, μm	27	34	39	45	37
Maximum crack width, μm	50	50	60	70	95

2.2. Environmental exposure

Five different conditioning regimes were used to simulate different environmental exposures and to investigate their influence on the autogenous healing of ECC at early ages.

1. CR1 (water/air cycle) subjected ECC specimens to submersion in water at 20 °C for 24 h and drying in laboratory air at 20 ± 1 °C, 50 ± 5%RH for 24 h. Water was replaced after every cycle. This conditioning regime was used to simulate cyclic outdoor environments, such as that experienced during alternating rainy and cloudy days.
2. CR2 (water/hot air cycle) consisted of submersion in water at 20 °C for 24 h, oven drying at 55 °C for 22 h, and cooling in laboratory air at 20 ± 1 °C, 50 ± 5%RH for 2 h. Water was replaced after every cycle. This regime was used to simulate outdoor environments such as rainy days followed by sunshine and high temperatures in summer.
3. CR3 (90%RH/air cycle) consisted of specimen stored in a 90 ± 1%RH curing cabinet at 20 °C for 24 h, and then in laboratory air at 50 ± 5%RH and 20 ± 1 °C for 24 h. This regime was used to simulate high humidity outdoor environments without submerrsion in water.
4. CR4 (water) consisted of continuous submersion in water at 20 °C until the predetermined testing ages. This regime was used to simulate ECC in underwater structures.
5. CR5 (air) considered direct exposure to laboratory air at 20 ± 1 °C, 50 ± 5%RH until the predetermined testing ages. This conditioning was used as the reference regime.

2.3. Evaluation methods for autogenous healing

Resonant frequency (RF) measurement based on ASTM C215 (Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequency of Concrete Specimens) was used in the transverse mode to monitor the rate and extent of autogenous healing of pre-damaged ECC specimens after different environmental exposures. The RF measurement technique is typically used to quantify concrete damage after freezing and thawing cycles. In this study, however, this technique is used to quantify the recovery of bulk mechanical properties. After pre-damaging, the specimen experiences a drastic drop in RF magnitude. Recovery of RF after exposure to the various conditioning regimes is considered a measure of recovery. The RF measurements serve as a rapid and nondestructive testing technique.

In addition to RF studies, uniaxial tensile tests were conducted to determine the tensile properties of the pre-damaged ECC specimens after exposure to the conditioning regimes. Although more tedious to carry out than the RF measurements, as well as destructive, uniaxial tensile reloading gives a more direct assessment of weakening caused by crack damage and the subsequent recovery caused by self-healing. An MTS servo-hydraulic system was used in displacement control mode to conduct the tensile tests at a loading rate of 0.0025 mm/s. Two external linear variable displacement transducers were attached to the specimen to measure the specimen deformation, from which the tensile strain was determined (by dividing the deformation by the gage length of 100 mm). This is the same loading method used to create the pre-damage in the specimens.

3. Experimental results and discussion

3.1. Recovery of resonant frequency by self-healing

The autogenous healing of ECC as measured by the change of RF is reported in Fig. 2 as a function of the number of conditioning cycles. The RF values on the y-axis indicate the drop of RF from about 800 Hz, the RF corresponding to the virgin specimen without damage. When recovery does occur, the RF increases with the number of cycles, initially at a rapid rate, but eventually (and typically after approximately 4–5 cycles) levels off. The shaded bands in these plots indicate the RF range of virgin ECC specimens that had undergone the same conditioning regime. Within these bands, a slight increase in RF is observed with the number of cycles due to continued hydration of the bulk material. Ideally, if full recovery occurs, the RF should approach this band (about 800 Hz) after exposure. In general, specimens subjected to a higher initial tensile strain exhibit lower initial frequencies due to a larger number of cracks and ultimately lower recovery values after environmental exposure.

Noticeable autogenous healing after even a single cycle of environmental conditions involving water was observed. The development of additional autogenous healing slows greatly after 4–5 cycles. After ten CR1, CR2, and CR4 cycles, the RF of pre-damaged ECC specimens attained 62–90%, 70–90%, and 84–96% of virgin ECC specimens, respectively. Although CR3 subjected ECC specimens to a high humidity environment, RF recovery was limited; after ten CR3 cycles, the RF of pre-damaged ECC specimens was only 44–76% of virgin ECC specimens. As expected, the resonant frequency of pre-damaged ECC specimens did not show any distinct change with exposure to CR5 cycles.

Based on the observations mentioned, it was concluded that the presence of water is the most critical environmental factor in engaging ECC healing. Continued hydration of unhydrated cement particles is faster in water and water also promotes the dissolution of calcium hydroxide from the concrete matrix near the crack surface, which is necessary for the formation of calcium carbonate healing products. More importantly, for the time duration of these experiments, very little carbonation can take place in air because only carbon dioxide dissolved in the surface films of water is available [32]. Carbon dioxide in gaseous form reacts with calcium hydroxide only in sufficiently high relative humidity and over long time periods [33].

3.2. Recovery of tensile properties through self-healing

Although resonant frequency testing serves as a means of rapid assessment, it only provides a qualitative indication of the level of recovery. In order to analyze the magnitude of recovery of tensile properties, uniaxial tensile testing on healed ECC specimens subjected to the five environmental exposure regimes in Section 2.2 were conducted. As with the RF test results, specimens exposed to the CR3 and CR5 cycles did not show significant healing. The RF test results also indicated that the rising RF curves (under CR1, CR2 and CR4) almost always level off within ten cycles. For this reason, the tensile reloadings to examine rehealing were conducted after exposure to ten CR1, CR2, or CR4 cycles.

Fig. 3 shows the representative tensile stress–strain curves of the reloading test, along with those of preloading. For the reloading stress–strain curve, the permanent residual strain left from the preloading stage is not accounted for. This means that the actual strain experienced by a specimen is higher than that indicated in the reloading stress–strain curves. The tensile strain capacity of ECC is governed by a delicate balance between mortar matrix toughness and fiber bridging characteristics [30,34]. The different conditioning regimes may affect this balance, resulting in the variations in the composite strain capacity shown in Fig. 3.

For ECC materials that are designed to be damage-tolerant, retaining tensile strength and ductility after damage with or without self-healing is not surprising. This is confirmed by the equal or even higher tensile strength reached in the reloading stress–strain curves shown in Fig. 3. The most revealing effect of self-healing is in the specimen stiffness. Without healing, the stiffness of the crack-damaged ECC can be substantially lower, by as much as a factor of ten or more compared with the virgin specimen [29]. The reloading stiffness in this case is the stiffness of the relatively soft bridging

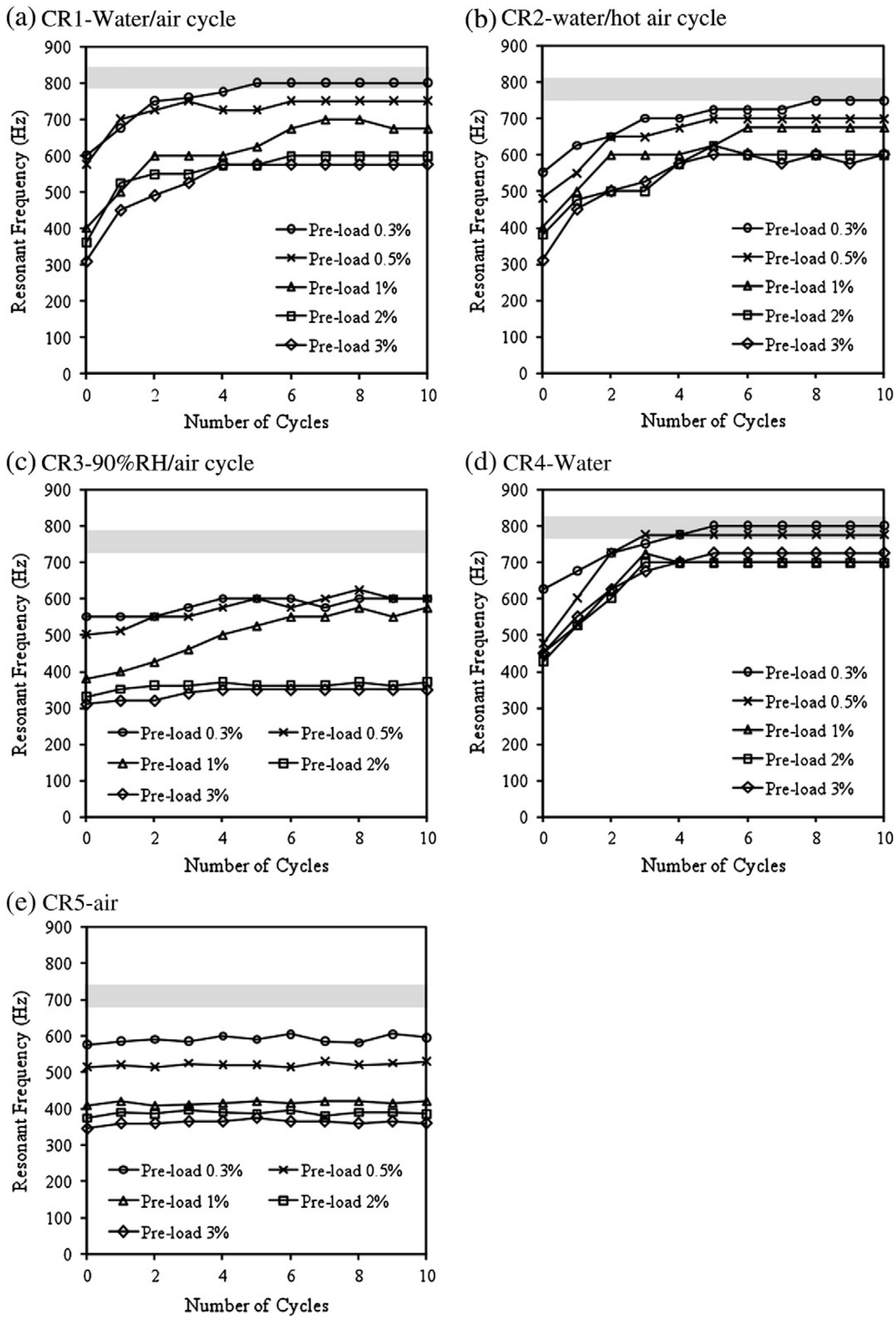

Fig. 2. Autogenous healing of early age ECC as measured by the change of RF after each cycle of (a) CR1 (water/air), (b) CR2 (water/hot air), (c) CR3 (90%RH/air cycle), (d) CR4 (water immersion) and (e) CR 5 (laboratory air). The shaded area represents the RF of virgin ECC specimens that have undergone the same conditioning regimes.

fibers. As may be expected, the stiffness reduction increases with the number of microcracks associated with a particular preloading strain level. Recovery of material stiffness on reloading provides a measure of the quality of self-healing in ECC.

In general, the reloading curves show a tensile strain capacity of 2% along with a higher tensile strength when compared with the preloading curve. This observation does not necessarily imply rehealing. Rather, this is most likely the effect of continued hydration processes at early ages [35] superimposed on the damage-tolerant characteristics of ECC. In particular, the elevated temperature of 55 °C used in CR2 accelerated the hydration of unreacted cement and fly ash, leading to the increase of fiber/matrix interfacial bonding and the highest tensile strength upon self-healing and reloading.

Fig. 4 shows the stiffness of healed ECC specimens as a percentage of that of the virgin specimens. Stiffness was defined as the secant of the initial rising branch of the stress–strain curve, in which the first point is

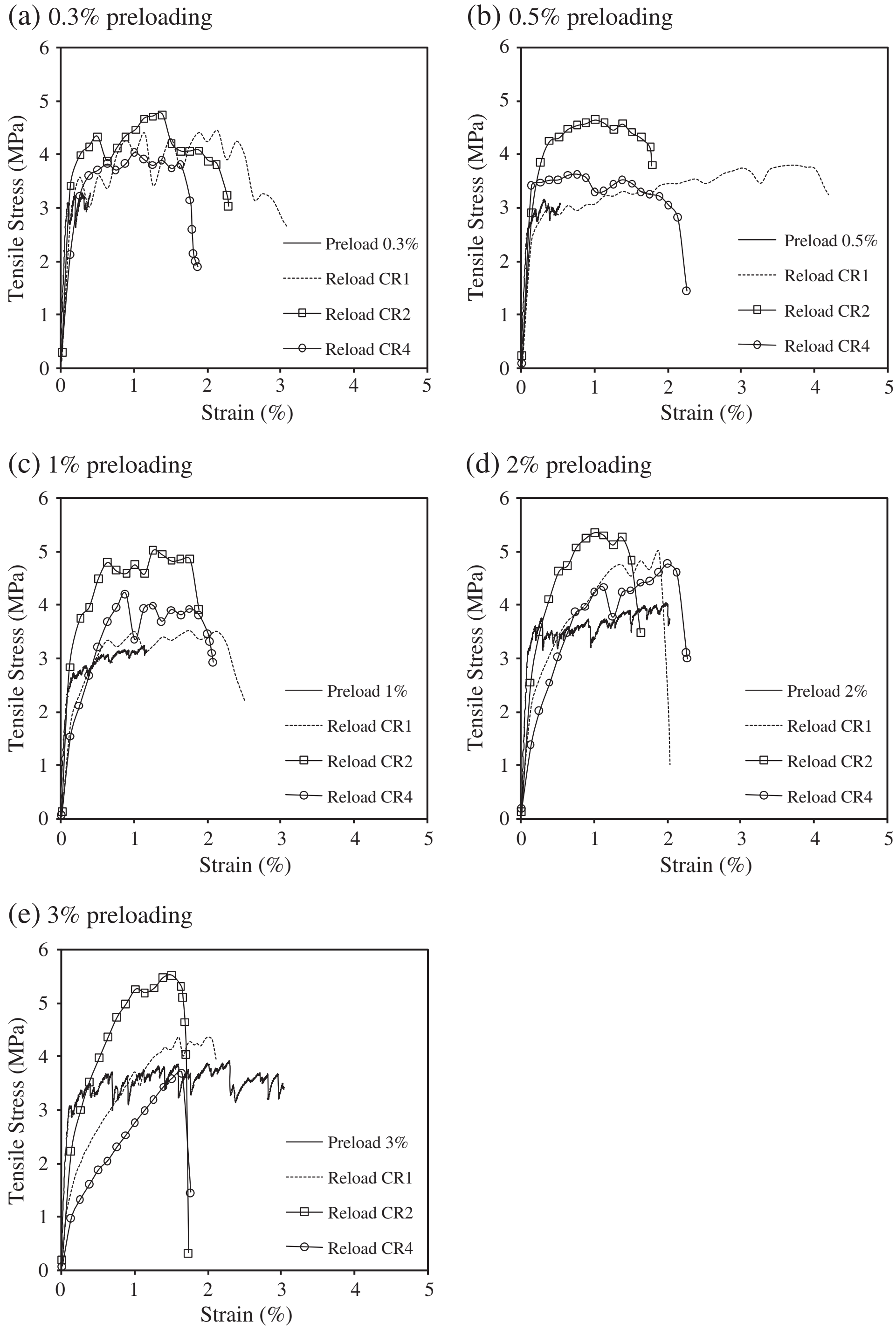

Fig. 3. Typical preloading and reloading tensile stress–strain curve of ECC with predetermined tensile deformation of (a) 0.3%, (b) 0.5%, (c) 1%, (d) 0.5%, and (e) 2%, after different environmental conditioning regimes.

chosen at 0.5 MPa and the second point at 2.5 MPa. The general trend shows that specimens with lower pre-damage levels tend to have higher recovered stiffness. Indeed, specimens with 0.3% pre-damage demonstrated complete rehealing under CR1 and CR2, and 80% rehealing under CR4. Even for specimens with 0.5% pre-straining, stiffness improve to over 60% when exposed to CR1 and CR2. At higher imposed strains during pre-damaging, the averaged crack width and particularly the maximum crack width increases (Table 2). This combined with a larger number of cracks contributes to reduced stiffness recovery.

Specimens subjected to ten CR2 cycles achieved the highest recovered stiffness (with the exception of specimens preloaded to 2%). This can be attributed to the effect of elevated temperature used in CR2 that accelerated hydration at early ages combined with the self-healing effect.

Samples exposed to CR4 cycles show a lower recovered stiffness (with the exception of specimens preloaded to 2%). This may be explained by the high internal moisture and the action of excessive water inside the healed ECC specimen continuously immersed in water.

The tensile tests were conducted immediately after ten conditioning cycles. In the case of CR4, the internal relative humidity was close to 100%. It has been reported that hardened cement paste with a large internal surface area exhibits intense fluid–matrix interactions

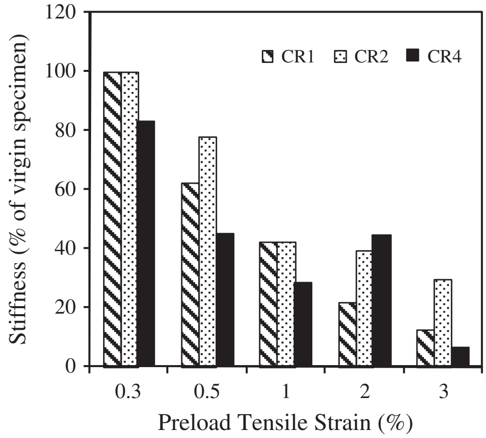

Fig. 4. Recovered stiffness of ECC specimens with five pre-damaging levels, and after exposure to three different exposure regimes.

[36], including moisture induced molecular adsorption forces along pore walls, capillary pressures in capillary pores, and swelling pressure due to the presence of interlayer hydrate water in nanopores. The induced forces are known to be extremely sensitive to fluid saturation level. Calcium–silicate–hydrate, the main binding phase in hardened cement, is composed of laminar sheets with interlayer absorbed water. The microscopic swelling pressure increases with increasing degree of water saturation. The repulsive forces push the laminar sheets apart and the cohesive forces among the hydrated products of cement decrease. The water molecules will decrease the surface energy of the hydrated products of cement and the van der Waals bonding among the hydrated products of cement [37]. Therefore, a higher degree of water saturation is likely responsible for the reduced stiffness of submerged ECC specimens.

Fig. 5a shows the crack damage on an ECC specimen after being subjected to 2% tensile preloading. False color microscopy was used to enhance the contrast. Fig. 5b displays an image of the same specimen after ten CR4 cycles. The distinctive white residue (healing product) is abundant within the cracks and near the crack faces on the specimen surface. As mentioned before, the average crack width for this specimen was about 45 μm.

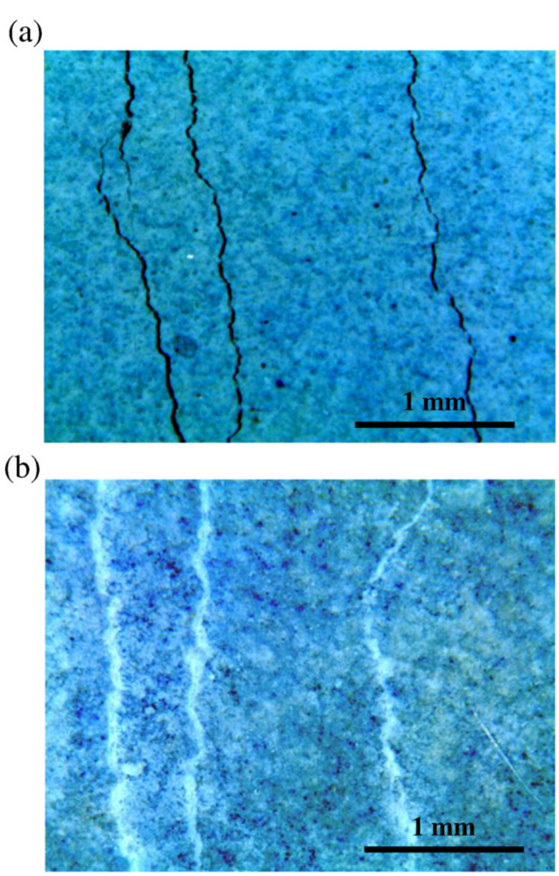

Fig. 5. False color micrograph of crack-damaged ECC specimen (a) before rehealing, and (b) after healing in CR4 (water).

3.3. Comparison of self-healing at early age with that at mature age

Prior to conducting this study, it was anticipated that better self-healing should result from early age specimens given the larger amount of unhydrated cement particles. Comparison of the self-healing characteristics of the early age (3 days) specimens with those of more mature specimens at 90-day age or older, however, indicates that higher stiffness recovery magnitude is attained in mature specimens than in young specimens.

Fig. 6 shows the percentage stiffness recovery for the 3-day age specimens as well as those for 6-month old specimens, after pre-damage to different levels, and then allowed to undergo rehealing with identical (ten) cycles of wetting and drying (CR1). As was pointed out earlier, the recovery decreases with increasing damage level for the young specimens, dropping from 100% at 0.3% pre-damage to about 10% recovery at 3% pre-damage. For the more mature specimens, remarkably, the recovery was maintained at approximately 80% for all four levels of pre-damage (0.5%, 1.0%, 2%, 3%) imposed [29].

ESEM examination of the self-heal products formed inside the pre-damage cracks for specimens of 3-day and 90-day ages did not reveal clear differences [38]. Regardless of age, a common trend observed was that smaller cracks (less than 20 μm width before healing took place) tend to be more completely filled with CSH gels after 10 cycles of CR1, while the larger cracks (50 μm or larger) tend to be partially filled with a mixture of CSH gels and calcite particles after the same exposure condition resulting in incomplete healing.

The observed stronger ability of stiffness recovery in mature specimens appears to have derived from the different crack patterns generated in the young and mature specimens under identical pre-damage magnitude. In general, the younger specimens tend to develop a smaller number of cracks of a larger averaged width compared to mature specimens, for the same imposed (pre-damage) strain. Fig. 7 shows the averaged crack width of the 3-day old specimens compared to that of 90-day old specimens, at four pre-damage levels. (Based on previous studies (e.g. [39]), the crack

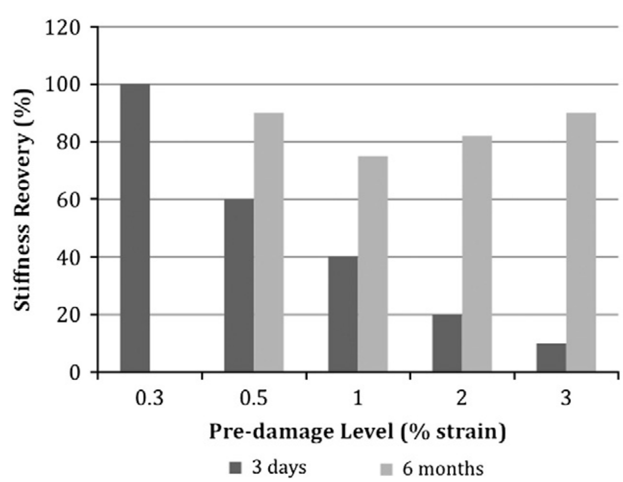

Fig. 6. Stiffness recovery on rehealing for 3-day and 6-month age specimens, subjected to various pre-damage levels.

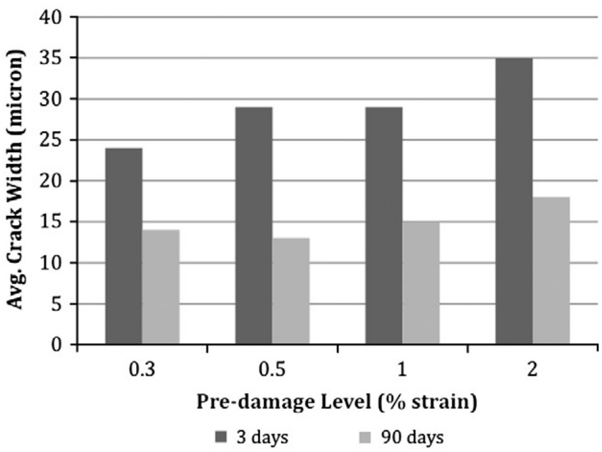

Fig. 7. Averaged crack width of 3-day and 90-day age ECC specimens subjected to various pre-damage levels.

pattern and strain capacity of ECC does not change appreciably beyond 90-day age.) It is clear that the mature ECC accommodates the imposed damage strain by developing a larger number of cracks, but maintains a tight crack width close to 15 μm on average. In contrast, the 3-day old ECC shows increasing number of cracks with increasing imposed damage strain accompanied by increasing crack width, rising from 24 μm to 35 μm on average as the imposed pre-damage strain increased from 0.3% to 2%. The larger crack width in the younger specimens reflect the less well developed bond between the reinforcing PVA fiber and the mortar matrix material, which in turn governs the stiffness of the bridging fiber and therefore the crack opening magnitude. It is likely that the relatively less complete rehealing associated with the larger crack width in the younger specimen is responsible for the lesser amount of stiffness recovery observed, when compared with the more mature specimens.

4. Conclusions

This paper presents the findings of an investigation into the autogenous healing of ECC subjected to different environmental exposure regimes, after deliberate damage by preloading in direct tension at 3 days of age. The inherent tight crack width of ECC, less than 60 μm, leads ECC to display robust self-healing properties at early ages with appropriate environmental exposure. Self-healed ECC shows substantial RF recovery as well as recovery of uniaxial tensile stiffness. This study reveals that mechanical property recovery due to autogenous healing (not just transport properties as has been reported in the literature, e.g. [29]) is achievable in cementitious materials with early age cracking, in commonly encountered exposure environments. Specifically,

- Specimen contact with external water, continuously or intermittently, is the most important environmental factor to engage autogenous healing of ECC at an early age. The RF data suggest a rapid rate of autogenous healing for the first few cycles of water exposure, but a substantially lower rate after 4–5 cycles and reaching a plateau beyond 10 cycles.
- In general, early age ECC with lower initial damage levels (e.g. at 0.3 or 0.5% preloading strain) with fewer microcracks experienced higher levels of recovery in RF and stiffness. However, even specimens with up to 3% pre-straining experienced rehealing, albeit incompletely. In these specimens, the averaged crack widths were maintained at below 50 μm.
- When exposed to external water, self-healing of crack-damaged ECC showed distinct stiffness and RF enhancements, resulting in true mechanical recovery. In some cases (e.g. preloading at 0.3% in CR1 and CR2), the stiffness and RF recoveries of the crack-damaged ECC were complete (i.e. 100%).
- Rehealing in terms of stiffness recovery was found to be lesser in 3-day early age specimens than in more mature specimens of 90 days or older age. This is likely a result of the ability of more mature specimens to maintain extremely tight crack width (about 15 μm) even at higher pre-damage level (e.g. up to 2% strain), whereas the early age specimens reveal increasing crack width as the pre-damage level imposed rises.

The self-healing of microcracks in ECC is expected to overcome the problem of early age cracking in high performance concrete materials for infrastructures exposed to water, e.g. transportation infrastructure such as roadways and bridges or in water-retaining structures. Thus self-healing ECC should maintain stiffness, strength and ductility even when subjected to damaging loads at an early age, especially when the damage level is limited to below 0.3%. Field studies should be conducted to confirm this expectation in actual structures exposed to natural environments.

Acknowledgments

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