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The effects of environmental conditioning and self-healing on the transport and mechanical property of engineered cementitious composites at early ages

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ABSTRACT

Autogenous recovery of mechanical properties and transport properties of ECC at early-ages under different environmental conditioning is reported in this paper. The mechanical properties studied include dynamic modulus, tensile stiffness, strength and ductility. The transport properties studied include water permeability and chloride diffusivity. The chemical make-up and physical properties, self-controlled tight crack width and high tensile ductility in particular, of ECC makes self-healing prevails in a variety of environmental conditions, include water permeation and submersion, environmental temperature, wetting and drying cycles. It is demonstrated by microstructure observation that self-healing of pre-damaged composite takes place automatically at cracked locations without external intervention. The establishment of self-healing in ECC can improve the long term ductility and durability of civil engineering structures after cracking.

KEYWORDS

ECC;
Environmental Conditioning;
Self-healing;
Permeability;
Microstructure.

INTRODUCTION

During the last decade, significant efforts have been made to develop engineered cementitious composites (ECC) which exhibits tough, strain-hardening behavior under tension in spite of low fiber volume fraction[1,2]. ECC is also an ultra-ductile fiber reinforced cement based composite which has metal-like features when loaded in tension. The uniaxial stress-strain curve shows a yield point followed by strain-hardening up to several percent of strain, resulting in a material ductility of at least two orders of magnitude higher than normal concrete or standard fiber reinforced concrete[3]. ECC provides crack width to below 100μm even when deformed to several percent tensile strain (Figure 1). Fiber breakage is prevented and pullout from the matrix is enabled instead, leading to tensile strain capacity in excess of 6% for PVA-ECC containing 2% by volume Poly Vinyl Alcohol (PVA) fiber which is a unique implementation by Li[4].

The superiority of ECC has been brought about by the micromechanics approach and the development in fiber, matrix, interface and composites processing technology. Micromechanics relates macroscopic proper-

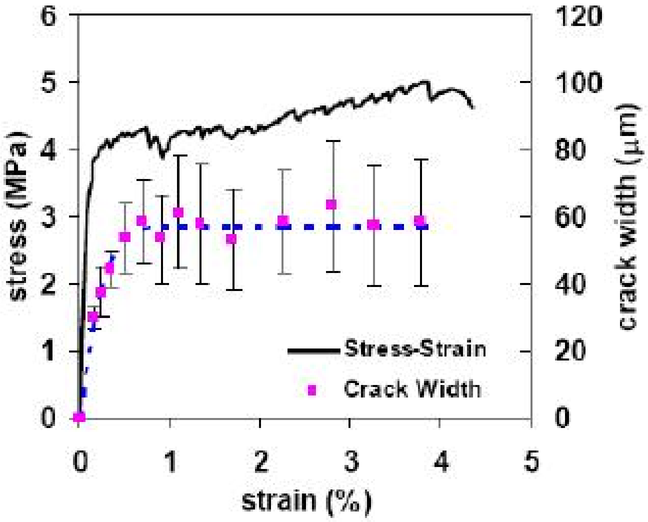


Figure 1 : Typical tensile stress-strain-crack width relationship of PVA-ECC

ties to the microstructure of a composite, and forms the backbone of material design theory[5]. Especially, it allows systematic microstructure tailoring of ECC as well as material optimization which can lead to extreme composite ductility.

Various methods have been attempted to cope with cracking of high strength cementitious composite at early ages[6]. A number of investigations[7,8] revealed that high cement content and low water-cement ratio distinctly raise the cracking potential at early age due to the increase of chemical and autogenous shrinkage and brittleness of high strength concrete. Those early age cracks provide pathways for the penetration of aggressive ions, and therefore greatly reduce the time to initiation of corrosion of steel reinforcement and shorten the service life of infrastructure as a consequence. For structural engineers, efforts have been placed on detailing the rebar reinforcement, including increase of reinforcing ratio and the arrangement of transversal or confinement reinforcement. For materials engineers, various kinds of fibers, fiber mesh, shrinkage reducing agent, polymers, crack sealer, and curing admixture have been applied for crack control. These approaches are effective in certain situations but their efficency and consistency remain to be doubted in other situations. Therefore, the development of cementitious composite materials which can automatically regain this loss of performance after early age cracking is highly desirable. The compressive strength of ECC ranges from 50 to 80MPa depending on mix composition, putting ECC into the class of high strength concrete materials, but without the associated brittleness[9,10,11].

In this study, autogenous healing of cracked ECC (cracks were generated by pre-loading) at early ages was experimentally investigated and verified. Focus has been placed on early age self-healing behavior under various environmental exposures, including drying action of wind and sun and wetting by rain runoff or snowmelt. Cyclic wetting and drying at controlled temperatures was used as an accelerated test method to simulate outdoor environmental conditions. Experimental investigations on the extent and rate of self-healing for ECC material pre-loaded to various strain levels to deliberately induce various levels of damage, along with the mechanical properties of ECC after self-healing, are presented.

ENVIRONMENTAL CONDITIONING AND SELF-HEALING

The robustness of self-healing should be examined under a variety of environmental exposures typically experienced by concrete infrastructure systems. In the investigation of self-healing of ECC, various environmental conditioning regimes have been adopted. These include cyclic wetting and drying, conditioning temperature, and immersion in water or chloride solution. Specifics of the environmental conditionings (EC) are summarized below:

- EC1 (water/air cycle) subjected pre-cracked ECC specimens to submersion in water at 20°C for 24 hours and drying in laboratory air at 21±1°C, 50±5% RH for 24 hours, during which no temperature effects are considered. This condition is used to simulate cyclic outdoor environments such as rainy days and unclouded days.
- EC2 (water/hot air cycle) consisted of submersion of pre-cracked ECC specimens in water at 20ºC for 24 hours, oven drying at 55°C for 22 hours, and cooling in laboratory air at 21±1°C, 50±5% RH for 2 hours. This condition is used to simulate cyclic outdoor environments such as rainy days followed by sunshine and high temperatures in summer.
- EC3 (90%RH/air cycle) consisted of specimen storage in 90±1%RH curing cabin at 20°C for 24

hours, and cooling in laboratory air at 21±1°C, 50±5% RH for 24 hours. This condition is used to simulate high humidity outdoor environments but with no exterior water available.

- EC4 (water submersion) consisted of submersion in water at 20°C till the predetermined testing ages. This condition is used to simulate ECC in some underwater structures.
- EC5 (air) consisted of direct exposure to laboratory air at 21±1°C, 50±5%RH till the predetermined testing ages. This condition is used as the coupon condition.
- EC6 (water permeation) consisted of continuous permeation through cracked ECC specimen in water at 20°C till the predetermined testing ages. This condition is used to simulate infrastructure in continuous contact with water with a hydraulic gradient, such as water tank, pipelines, and irrigation channels.
- EC7 (chloride solution submersion) considered direct exposure of pre-cracked ECC specimens to a solution with high chloride content. This condition is used to simulate the exposure to deicing salt in transportation infrastructure or parking structures, or in concrete containers of solutions with high salt content.

In order to develop a comprehensive understanding of self-healing in ECC, four methods have been used in examining its self-healing behavior. The dynamic modulus measurements provide a quick means to assess the presence of self-healing. The uniaxial tension test is used to determine self-healing of mechanical properties. Water permeability is used to examine the recovery of transport property through permeation. Surface chemical analysis, i.e. Energy Dispersive X-ray Spectroscopy (EDX), and Environmental Scanning Electron Microscopy (ESEM) are used to analyze the chemical composition and morphology of self-healing product. Together, they show unequivocally the presence of self-healing in ECC in both the transport sense and in the mechanical sense.

EXPERIMENTAL

The mix proportion of ECC material follows TABLE 1. To prepare the material, a force-based Hobart mixer with 20L capacity was used in preparing a single batch of ECC material. The fresh mixture was then cast into coupon molds measuring 300mm by 76mm by 12.5mm. The fresh ECC specimens were covered with plastic sheets and demolded after 24 hours. After demolding, specimens were air cured at 50±5% RH, 21±1°C for 2 days. Uniaxial tensile tests were then conducted (Figure.2) on the 3rd day to preload/pre-crack ECC specimens at specified tensile deformation, i.e. 0.3%, 0.5%, 1%, 2%, and 3%. When the tensile strain reached the predetermined value, the tensile load was released, and the specimens were removed from the tensile test machine to prepare for predetermined environmental conditioning, i.e. EC1, EC2, EC3, EC4, and EC5. TABLE 2 shows the average number of cracks and their corresponding maximum crack widths over a gauge length of 100mm. All crack width measurements are conducted in the unloaded state.

Figure 2 : Uniaxial tensile test setup

TABLE 1 : Mix proportions of ECC

Materials	Cement	Aggregate	Fly ash	Water	HRWR	Fiber
Unit weight (kg/m3)	578	462	694	319	17	26

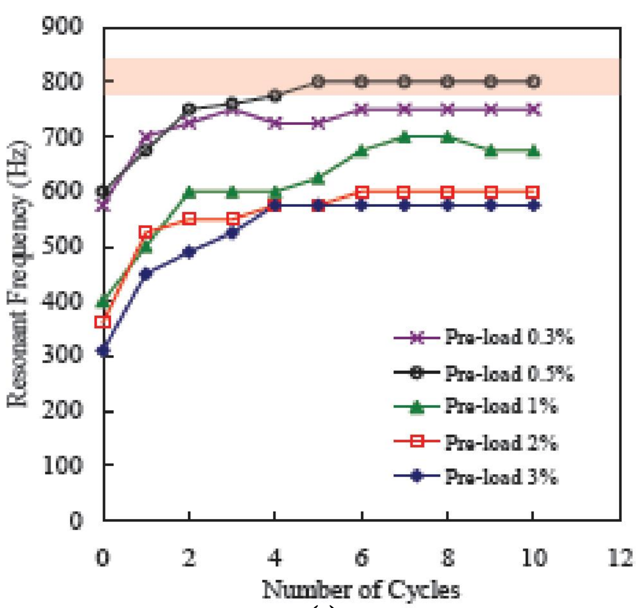
TABLE 2 : Crack characteristics of pre-load ECC

Tensile strain (%)	3	2	1	0.5	0.3
Number of cracks	41	24	16	7	5
Maximum crack width (μm)	95	70	60	50	50

Recovery of dynamic modulus

The material dynamic modulus measurement based on ASTM C215 (Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequency of Concrete Specimens) appears to be a particularly promising technique to monitor the extent and rate of autogenous healing. This test method (ASTM C215), which relies on changes in resonant frequency, has proven to be a good gauge of material degradation due to freeze thaw damage and is specifically referenced within ASTM C666 for freeze thaw evaluation. Rather than quantifying damage; however, it has been adapted to measure the extent and rate of self-healing in cracked concrete, when healing is seen as a reduction in material damage.

Test results from the resonant frequency tests are given in Figure 3 and 4. Each data point is a mean value of two specimens. Figure 3 plots the resonant frequency versus the number of cyclic conditioning. It demonstrates the rate/process of autogenous healing of cracked ECC specimens under different environmental exposures. The shaded area indicates the range of resonant frequencies of virgin (no preloading induced damage) ECC specimens which had undergone the same environmental conditioning.



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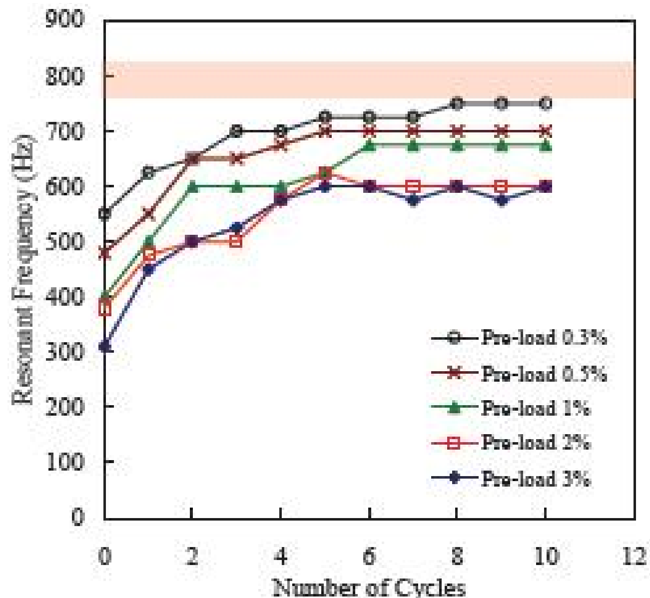

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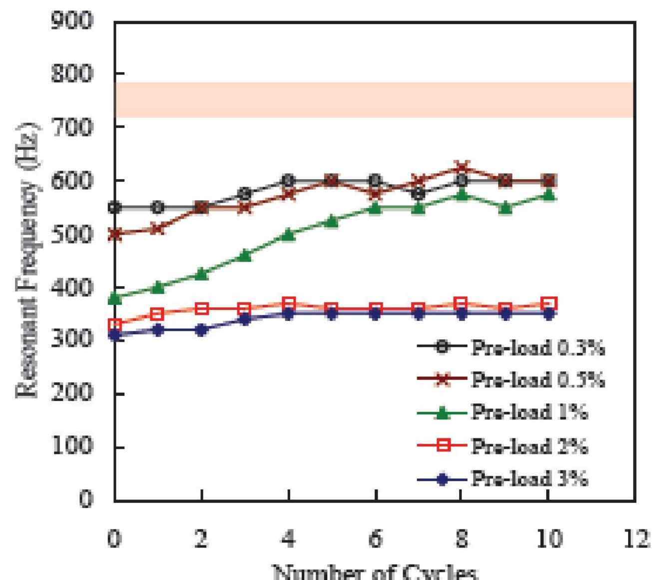

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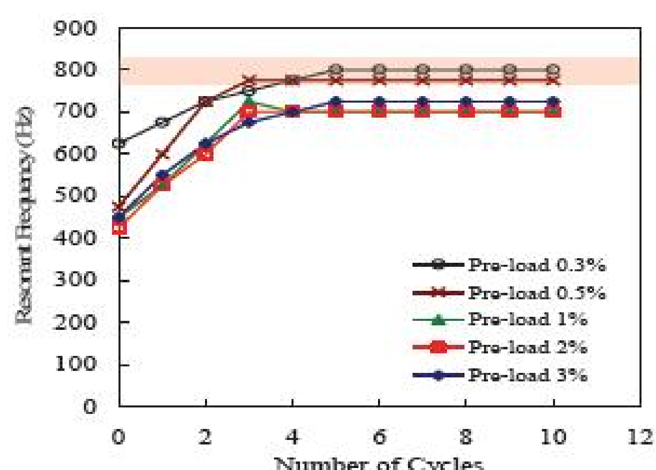

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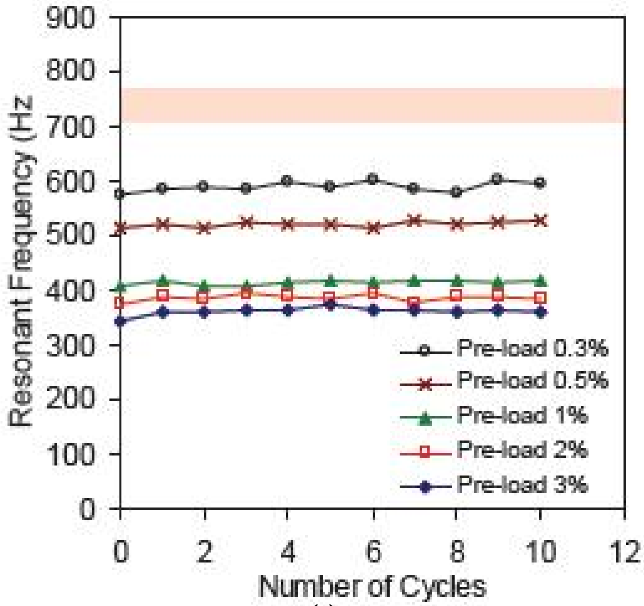


(e)

Figure 3 : ECC autogenous healing rate/process (i.e. resonant frequency vs number of cyclic environmental conditioning) at different environmental exposures at early ages (a) EC1-water/air cycle, (b) EC2-water/hot air cycle, (c) EC3 - 90%RH/air cycle, (d) EC4-water (e) EC5-air

Among the five environmental conditionings, EC1 (water/air cycle), EC2 (water/hot air cycle), and EC4 (water) significantly promote the autogenous healing of cracked ECCs at early ages, and roughly 4 to 5 conditioning cycles are necessary to engage noticeable autogenous healing of cracked ECC material. Unlike self-helaing at later age in which the presence of both water and carbon dioxide are necessary to form calcium carbonate, early age self-healing occurs even when the specimen is submersed in water (EC4). This may be attributed to continuous hydration of damaged ECC to form C-S-H gel at early ages when plenty of water is provided under EC4. In general, specimens subjected to higher preloaded tensile strain exhibit lower initial frequency due to a higher degree of damage, and ultimately lower recovery values after environmental exposure. However, the resonant frequency can almost recover to the RF value of virgin ECC specimens in EC4 (water) even for specimens with large predetermined tensile deformation. Interestingly, although EC3 (90%RH/air cycle) subjected ECC specimens to a high humidity environment, RF recovery was limited. The resonant frequency of cracked ECC specimens exposed to air (EC5) did not show any distinct change with environmental exposures.

By calculating the ratio of the resonant frequency after ten wet-dry cycles to the resonant frequencies of virgin ECC specimens which had undergone the same conditioning regimes, the extent of self-healing can be deduced as shown in Figure 4. After conditioning regime 1 (EC1), the normalized RF regained to 62~96% of initial values for the various preloading level, in contrast to the 39~77% the preload specimens without self-healing. For the EC2 (water/hot air cycle) specimens, the resonant frequencies after pre-loading were 43~73% of the initial value, and after self-healing had stabilized at 76~93% of initial. Regarding the recovery of RF, the effect of EC1 (water/air cycle) is similar to that of EC2 (water/hot air cycle). For EC4 (water) specimens, the resonant frequencies after pre-loading were 62~83% of the initial value, and after self-healing attained 95~100% of initial, in which the RF of most cracked ECC specimens approached those of virgin specimens. Almost no self-healing occurred in cracked ECC specimens when exposed to air (EC5). For EC3 (90% RH/air cycle) specimens, the resonant frequencies after pre-loading were 40~73% of the initial value, and after self-healing had recovered to 42~78% of initial. Again, limited self-healing was evident in high humidity condition.

Based on the above observations it can be concluded that the presence of water is the most critical factor in engaging autogenous healing of ECC at early ages (e.g. EC1, EC2, and EC4). Further hydration of unhydrated cement particle is faster in water and, also, water encourages the dissolving and leaching of calcium hydroxide from the concrete matrix near the crack surface, a precondition for the formation of self-healing products. More importantly, very little carbonation can take place in air because only carbon dioxide dissolved in the surface films of water is available. Carbon dioxide in gaseous form does not react with calcium hydroxide. It can be noted that submersion in water (EC4) results in weight change of ECC specimens at early age which can increase the resonant frequency. The higher resonant frequency resulted from water absorption within concrete was attributed to entry of interlayer water into Tober-morite gel and to much more cracks full of water. In this investigation, the rank of different conditioning regime in their effectiveness in recovery of resonant frequency of ECC at early ages is EC4

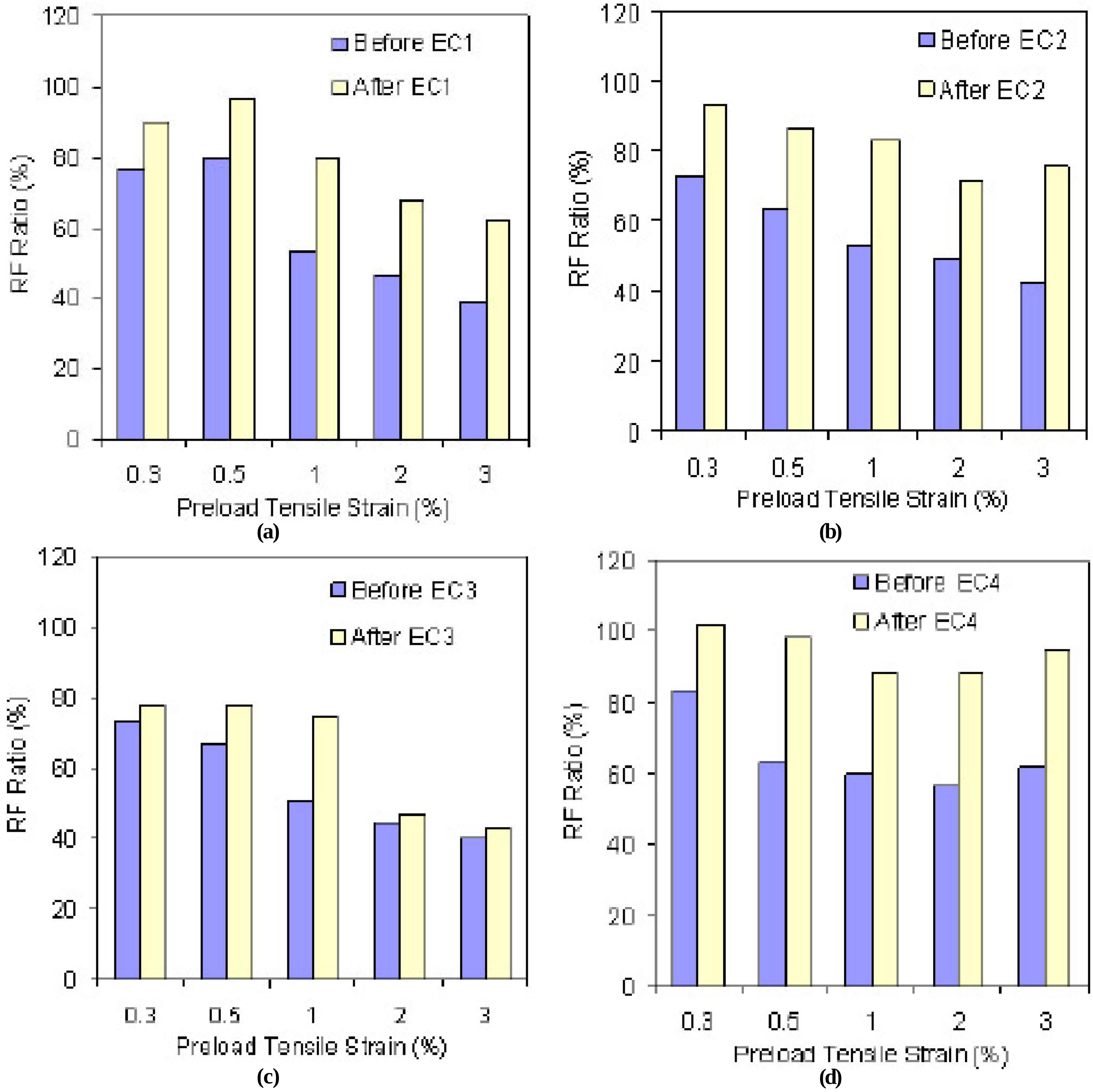
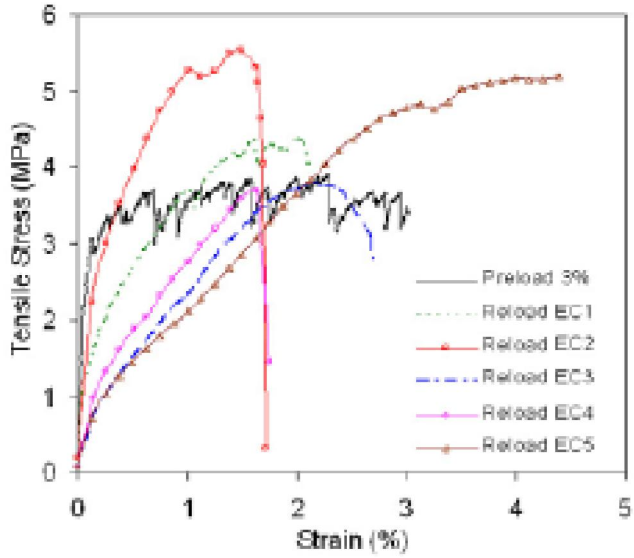


Figure 4 : Extent of ECC self-healing (i.e. resonant frequency ratio) under different environmental exposures (a)EC 1-water/air cycle, (b) EC 2-water/hot air cycle, (c) EC 3-90%RH/air cycle, (d) EC 4-water

(water)>EC1 (water/air cycle)~EC2 (water/hot air cycle)>EC3 (90%RH/air cycle)>EC5 (air). However, due to the water absorption side-effect described above, the effectiveness of self-healing under EC 4 need to be investigated further in order to assess the true self-healing in mechanical properties, stiffness in particular.

Recovery of tensile properties

Although resonant frequency test serves as a means of rapid assessment, it only provides an indication of the level of recovery after self-healing. In order to analyze the recovery magnitude of tensile stiffness, ductility and strength, uniaxial tensile test was conducted. Figure 5 shows the representative reloading tensile stress-strain curves of preloaded ECC specimens after cyclic conditioning (10 cycles) along with the tensile stress-strain curve of pre-loading. In the stress-strain curve of the reloading stage, the permanent residual strain introduced in the preloading stage is not accounted for.



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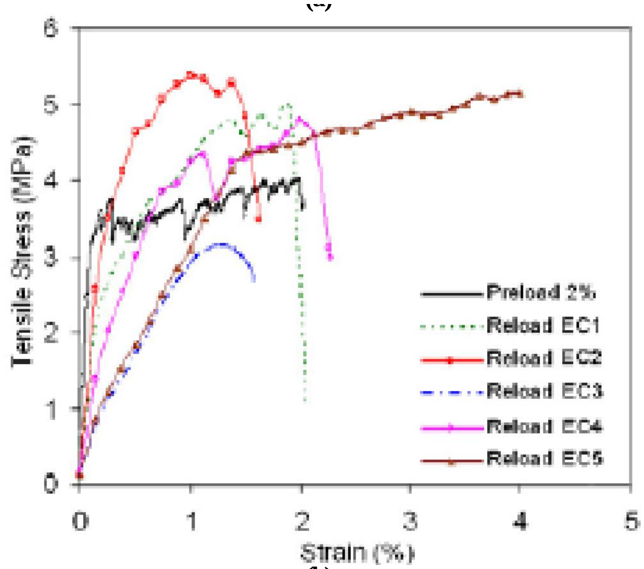

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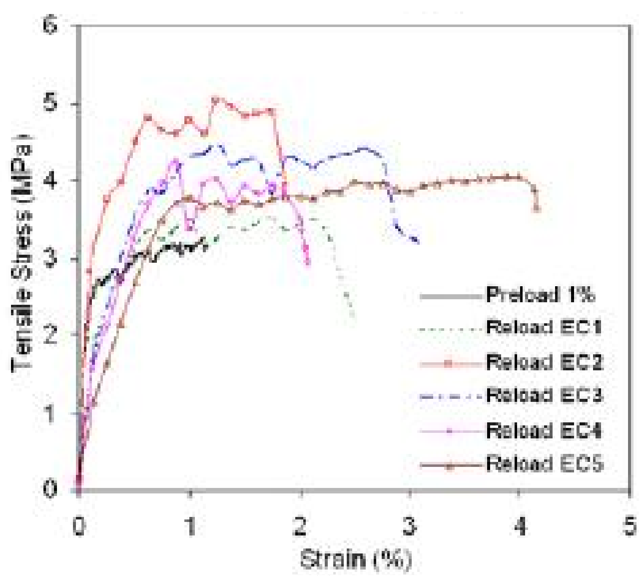

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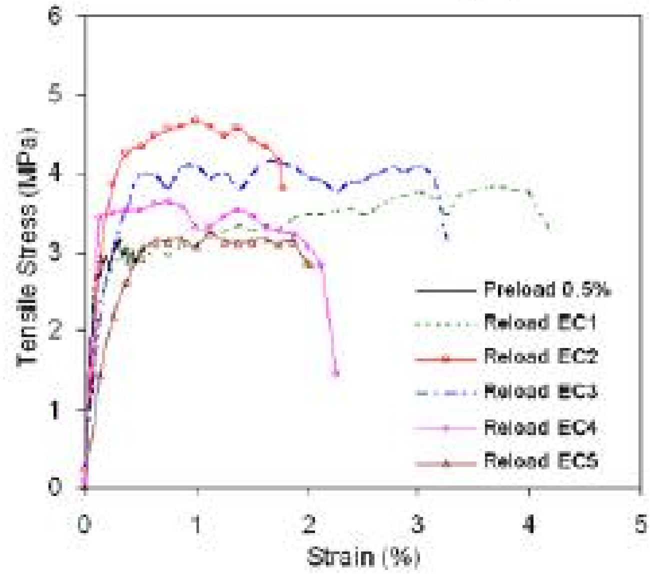

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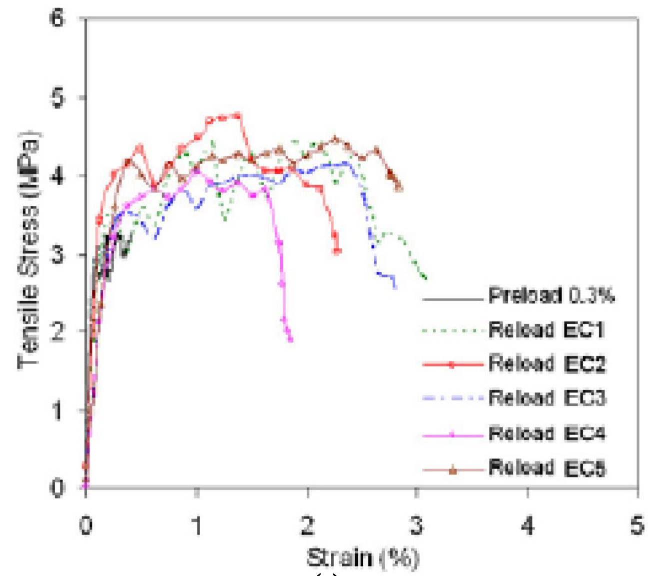


(c)

Figure 5 : Typical preloading and reloading tensile stress-strain curve of ECC with pre-determined tensile deformation of (a) 3% preloading, (b) 2% preloading, (c) 1% preloading, (d) 0.5% preloading, (e) 0.3% preloading

Stiffness

As can be seen from Figure 5, the slope of the initial raising branch of the tensile stress-strain curve, i.e. stiffness, is different between the preloading and reloading curves. The slope of the preloading curve represents the stiffness of virgin ECC material and the slope of the reloading curve of EC5 (air) may be considered as the stiffness of cracked ECC without any healing. It is clear that the stiffness of the unhealed specimen was drastically reduced due to the presence of the micro crack damage generated during preloading. However, as can be expected, the amount of stiffness reduction diminishes as the preloading (strain) magnitude decreases. After rehealing (under EC 1 to EC 4), the material regained a certain degree of its original stiffness depending on different environmental exposures. In some cases, the stiffness can almost fully recover.

Figure 6 displays the stiffness ratio as a function of preloading strain and conditioning regimes. Stiffness ratio is defined as the ratio of the stiffness of the reloading curve to the stiffness of the preloading curve. As can be seen, low initial damage level, i.e. low pre-determined tensile deformation, has high stiffness ratio under all five environmental conditionings. For example, the stiffness ratio at preloading strain of 3% ranges from 5% to 30%, while the stiffness ratio at preloading strain of 0.3% ranges from 30% to 100%. In general, EC2 (water/hot air cycle) exhibits the highest stiffness ratio of ECC specimen and EC 5 (air) shows the lowest stiffness ratio. The stiffness of ECC material with low preloaded strain can recover completely under EC1 (water/air cycle) and EC2 (water/hot air cycle). At high preloaded stains, however, EC2 (water/hot air cycle) shows a better recovery than EC1 (water/air cycle). This might be attributed to the enhanced hydration due to exposure to higher temperature in EC2.

Interestingly, unlike the result observed from the resonant frequency measurements, EC 4 (water) does not show the most significant stiffness recovery among

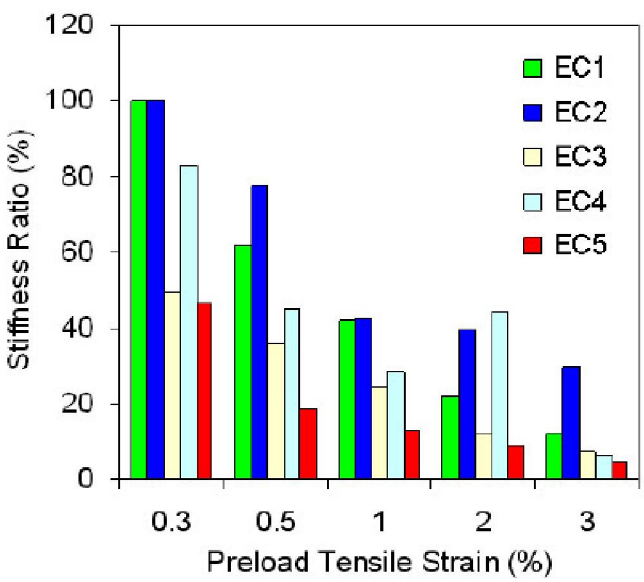


Figure 6 : ECC stiffness ratio under different environmental exposures, for specimens with different pre-loading levels

the five environmental conditions. This can be explained by the high internal moisture and actions of excessive water inside the self-healed ECC specimen continously immersed in water (EC4). It should be pointed out that self-healed ECC specimens in EC4 (water) were reloaded immediately after a long time exposure to water and the internal relative humidity is close to 100%. It has been reported that harden cement paste with a huge internal surface area exhibits intense fluid-matrix interactions. Moisture induced micro-forces, include molecular adsorption forces along pore walls, capillary pressures in capillary pores, and interlayer fluid pressures (swelling pressure) due to the presence of interlayer hydrate water in nanopores, arise as a result of repulsive forces between water molecules and keeping the pore walls at a certain distance. The induced forces are known to be extremely sensitive to fluid saturation level. CSH (Calcium-Silicate-Hydrate) as main binder ingredients in cement is composed of laminar sheets with interlayer absorbed water. The microscopic swelling pressure increases with increasing degree of saturation of water. 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. Therefore, higher degree of saturation of water obviously results in a reduction of the stiffness of wet ECC specimen. It was concluded that resonant frequency measurements although providing a rapid test method can not precisely assess the quality of self-healing in some cases. Based on the uniaxial tensile test results which gives the true qualification of self-healing, the rank in the stiffness ratio of cracked ECC at early ages is EC2 (water/hot air cycle)>EC1 (water/air cycle)>EC4 (water)>EC3 (90%RH/air cycle)>EC5 (air).

Tensile strain capacity

Figure 7 shows the result of tensile strain capacity of pre-cracked ECC subjected to different environmental exposures. After 10 cycles of conditioning subsequent to different levels of induced damage, all specimens retain tensile strain capacity above 1.5%, a ductility distinctly higher than normal concrete.

Among the five environmental conditionings, EC5

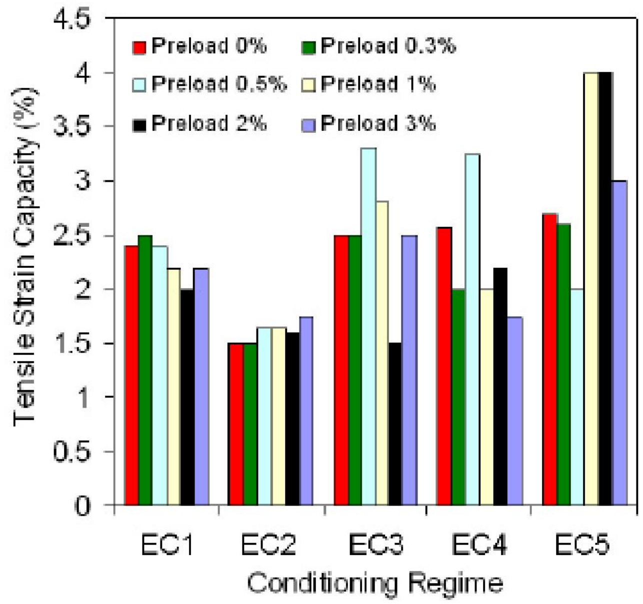


Figure 7 : ECC tensile strain capacity of reloading under different environmental exposures

(air) results in the highest tensile strain capacity and EC2 (water/hot air cycle) results in the lowest one. This is a direct reflection of the effect of hydration degree of the mortar matrix on ECC tensile strain capacity due to different environmental conditionings at early age. Recall that specimens subjected to EC2 are submersed in water and then dried in air at 55°C. With this temperature increase, the moisture in the specimens will migrate out and may result in a process similar to steam curing. Therefore, hydration of unreacted cement and fly ash will be accelerated, leading to an increased strength of ECC matrix and fiber/matrix interfacial bonding (due to a strong hydrophilic nature of PVA fiber), especially at early ages. The increase of matrix cracking strength prevents crack initiating and propagation from defect sites and excessively strong interfacial bonding increases the tendency of fiber rupture. Both mechanisms cause a negative impact on the development of multiple microcracking, and therefore lower tensile ductility observed as a consequence.

Tensile strength

The ultimate tensile strength, which is governed by the fiber bridging strength, of all ECC specimens at reloading are larger than the preloading tensile strength and this is most likely the effect of continued hydration process at early ages, i.e. increased fiber bridging strength with further hydration. In particular, the ultimate strength after self-healing for EC2 (water/hot air cycle) is much higher than that of the pre-loaded specimens (Figure 5) which again implies an accelerated hydration process leading to a stronger fiber/matrix interfacial bonding when ECC specimens are subjected to EC2 (water/hot air cycle).

Recovery of permeability

The ECC specimens were preloaded to the predetermined tensile strain 1.5%, 2% and 3% respectively. Prior to the permeability testing, ECC specimens were kept in water for 14 days to ensure complete water saturation. Permeability testing was conducted on both uncracked and cracked ECC samples after immersion in water. For the lower permeability materials a falling head test setup was used and shown schematically in Figure 8.

The edges of the coupon specimen were sealed with epoxy to facilitate unidirectional flow through the cross section. Due to the length of time associated with this type of testing, crack width permeability measurements were performed in the unloaded state.

Formulations for calculating the water permeability from the falling head test are summarized in eqns (1).

$$\mathbf{k = \frac{aT}{At} ln(\frac{h_0}{h_f})} \qquad \textbf{(1)}$$

Where:

k=water permeability coefficient (m/sec)
a=cross sectional area of stand pipe (m2)=$2.84\times10^{-5}m^2$
T =specimen thickness in direction of flow (m)=0.012m
A=cross sectional area of specimen exposed to flow (m2)=$8.93\times10^{-3}m^2$
t=time duration of the test (sec)=varies
h=constant water head in constant head test (m)=varies
h_o=initial water head in falling head test (m)=varies
$h_{f=}$final water head in falling water test (m)=varies

Falling head tests were conducted continuously over a period of three weeks until a steady state value of permeability had been reached. The reported permeability values are final steady state value exhibited.

Figure 9 shows the rate of permeation through the ECC specimens dropped drastically from the initial values until asymptotically reaching the recorded value, even though the crack widths during permeability test-

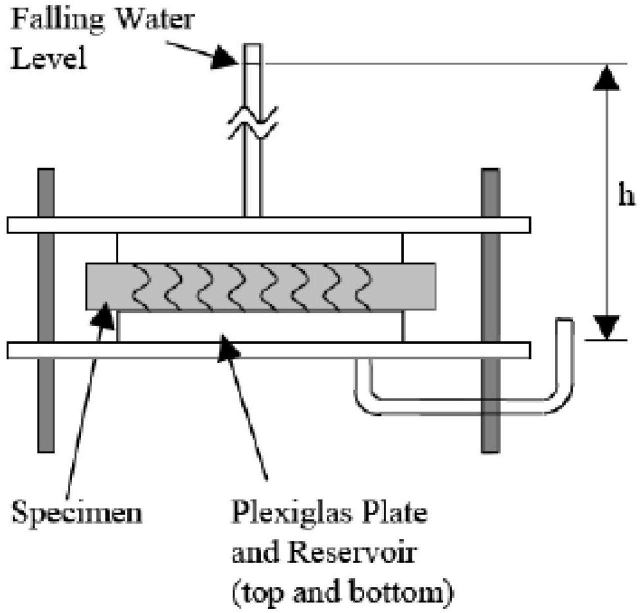


Figure 8 : Falling head test for permeability measurement

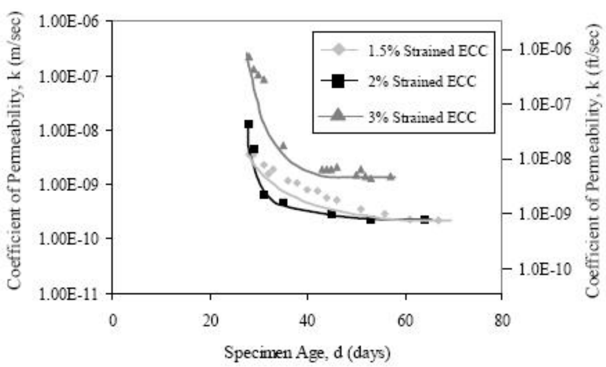
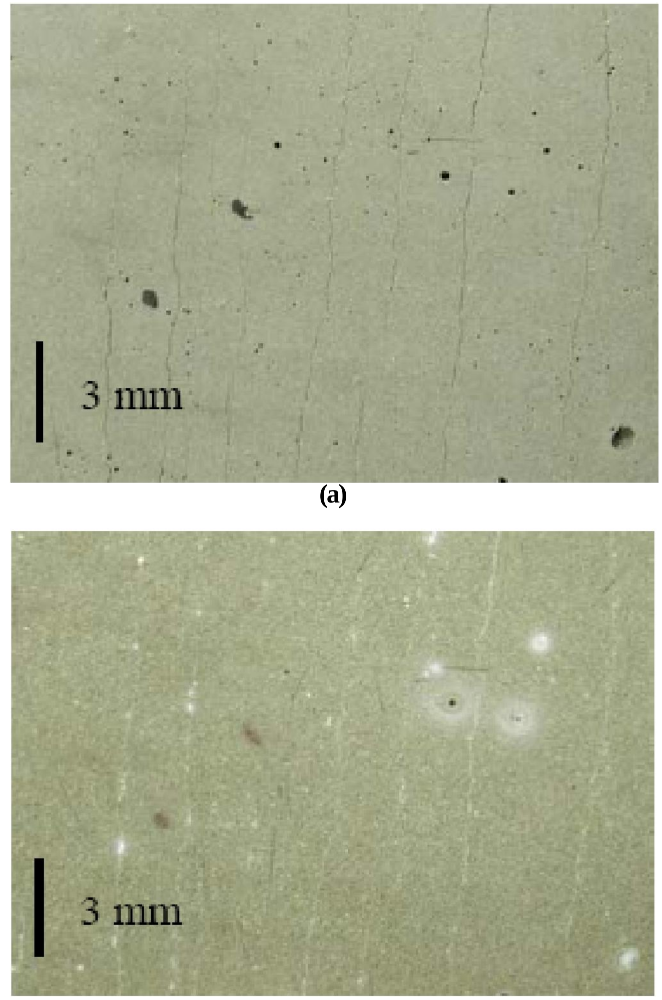


Figure 9 : Development of permeability for ECC strain to 1.5%, 2%, and 3%



(b)

Figure 10 : Appearance of ECC permeability speciemens (a) before permeability testing, and (b) after permeability testing

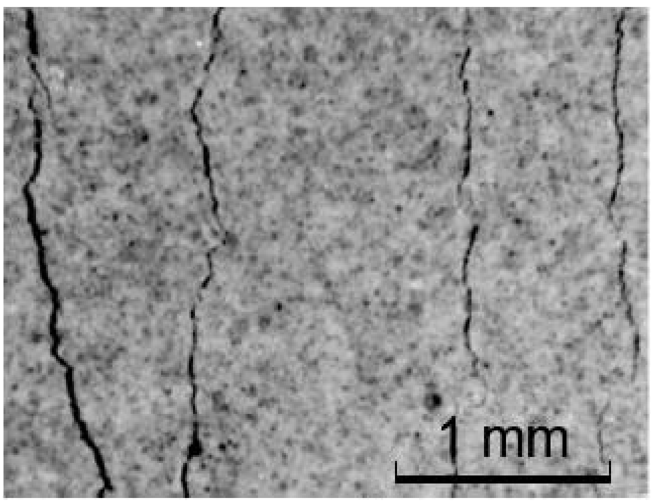
ing do not change. This phenomenon can be partially attributed to achieving complete saturation and further densification of the matrix throughout the testing period. However, ECC specimens were saturated in water for 14 days prior to permeability testing at an age of 28 days. By the time of testing, the specimens should have been nearly, if not completely, saturated and continued to undergo little matrix hydration.

Throughout the course of permeability testing, a white residue formed within the cracks and on the surface of the specimens near the cracks. These formations are shown in Figure 10. Figure 10(a) shows a saturated ECC specimen immediately prior to the beginning of permeability testing, while Figure 10(b) shows the same specimen after permeability testing.

The white residue forms both within the cracks, and within the pores on the surface of the ECC specimen. The effect of self-healing of cracks on permeability has been investigated by other researchers, and may be significant in the permeability determination of cracked ECC. This can be attributed primarily to the large binder content and relatively low water to binder ratio within the ECC mixture. The presence of significant amounts of unhydrated binders allows for autogeneous healing of the cracks when exposed to water. This mechanism is particularly evident in cracked ECC material due to the small crack widths which facilitate self-healing. However, this phenomenon is not observed while cracked ECC specimens are simply saturated in water (EC4). During the 14 days of saturation prior to permeability testing, cracked ECC specimens showed no signs of autogeneous healing of the cracks. After only 3 days in the permeability testing apparatus, evidence of self-healing became apparent. A similar phenomenon was also seen when cracked ECC specimens were partially submerged in water. Crack healing was only exhibited near the surface of the water, while no healing was observed above or below the water surface.

Microstructure observations

Figure 11(a) shows an ECC specimen subjected to 2% tensile pre-loading and Figure 12(b) displays the same spot on the specimen surface after undergoing autogenous healing through submersion in water (EC4). The distinctive white residue (reheal product) is abundant within the crack and near the crack face on the specimen surface.



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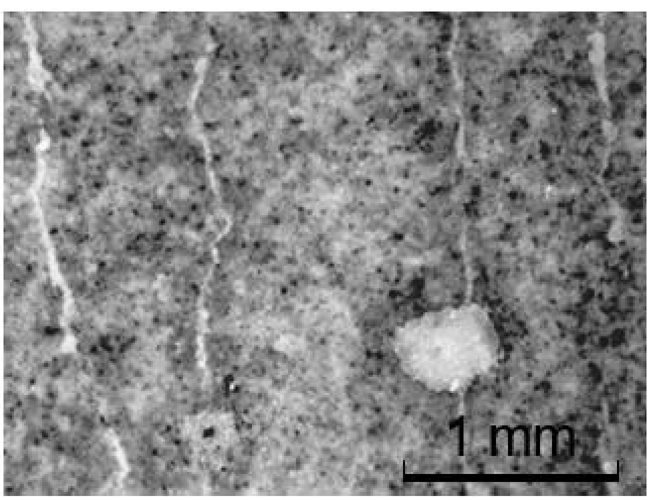
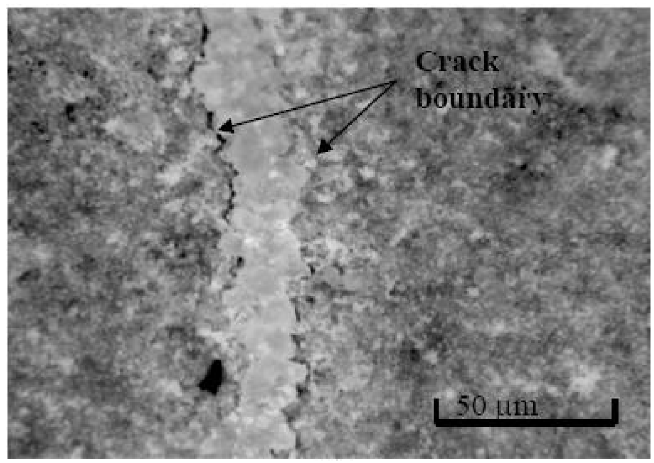


(b)

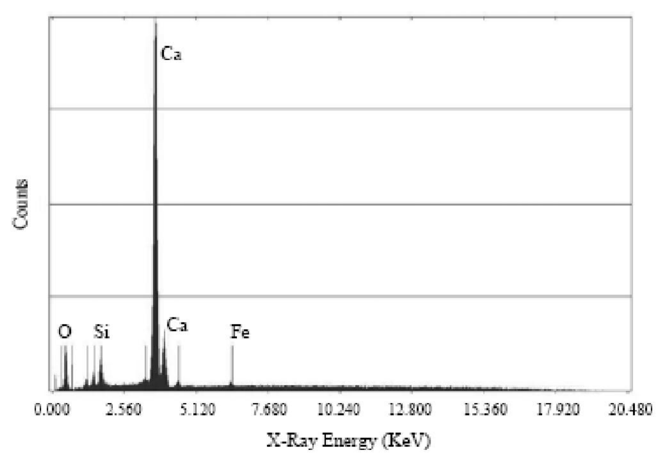
Figure 11 : (a)Controlled microcrack damage of preloaded ECC specimen and (b) autogenous healing of ECC specimen subjected to EC 4 (water)

Figure 12 gives a close-up view of the reheal product and its EDX spectrum. As can be seen, calcium is the main elemental composition of the reheal product. By conducting EDX quantification for oxides, the amount of calcium carbonate ($CaCO_3$) can be estimated by combining equal amount of CaO and CO_2. Interestingly, excessive CaO was found which implies the existence of calcium hydroxide (CaOH). Therefore, it is likely that the reheal product at early age is a compound of calcium carbonate (precipitation/crystallization) and calcium hydroxide (further hydration). Further investigation by x-ray diffraction analysis is necessary in the future in order to reveal the precise chemical composition of reheal products.

Surface chemical analysis (EDX) of the self-healing ECC specimens using an ESEM show that the crys-



(a)



(b)

Figure 12 : (a) Reheal product (b) ESEM Surface Chemical Composition Analysis (EDX) of Self-Healing Crack Formations

tals forming within the cracks, and on the surface adjacent to the cracks, are hydrated cement products, primarily calcium carbonate (Figure 11). These crystal formations within the self-healed cracks are shown in Figure 11. To facilitate healing of the cracks, and promote formation of calcium carbonate, a flow of water containing carbonates or bicarbonates must be present. Within the permeability testing, these carbonates were introduced by the dissolution of CO_2 in air into the water which flows through the specimens. In the case of the partially submerged specimens, the small amount of carbon dioxide dissolved at the water surface was sufficient to cause limited self-healing at that location. However, in the absence of this constant carbonate supply, as in the saturation tanks prior to permeability testing, no self-healing of the ECC micro cracks can occur. Ultimately, the formation of these crystals slows the rate of permeation through the cracked composite and further reduces the permeability coefficient.

The quality of self-healing is likely influenced by the type of self-healing products formed inside the crack. Analyses of these products were conducted using ESEM and EDX techniques. The crystalline and chemical properties of self-healing products were determined. These techniques are particularly useful in verifying the chemical makeup of self-healing compounds, essential in identifying the chemical precursors to self-healing and ensuring their presence within the composite.

CONCLUSION

This study presents the findings of an investigation on autogenous healing of ECC subjected to different environmental exposures at early ages. The inherent tight crack width less than 60μm, make ECC an ideal material to engage in robust self-healing at early age under proper environmental exposures when compared with concrete. Self-healed ECC shows substantial recovery of Resonant Frequency loss from damage (resonant frequency test) as well as adequate mechanical performance (uniaxial tensile test). It can be expected that autogenous healing of ECC will greatly benefit the long-term durability and safety of civil infrastructure. This study represents a systematic revelation of mechanical (not just transport) properties in cementitious material, and through the use of commonly encountered exposure environments in the experiments, demonstrates the realization potential of self-healing in concrete structures. A number of other specific conclusions can be drawn:

- Four to five cycles are necessary for engaging self-healing of ECC at early age under EC1 (water/air cycle), EC2 (water/hot air cycle), and EC4 (water). For different environmental exposures, water is the most determining environmental factor to engage autogenous healing of ECC at early ages.
- In general, ECC with lower initial damage level, i.e. low pre-determined tensile deformation, has higher level of recovery for the same conditioning regime. This confirms that the tighter the crack width and the lower the crack number, the higher the probability of self-healing. Crack width below 150μm and preferably 50μm leads to highly robust self-healing. This tight crack width, while difficult to control in normal concrete even with steel reinforcements, are readily attained in ECC when straining is below 1%.
- Resonant frequency measurement can be used as a rapid screening of the presence or absence of self-healing. This measurement reflects averaged bulk property change in the specimen under the different exposure conditions. Uniaxial tensile test, while more cumbersome, gives a true assessment of the quality of self-healing of the microcracks, and provides quantitative measurements of recovered mechanical properties.
- Under proper conditioning, self-healing can distinctly enhance the stiffness of cracked ECC resulting in the true mechanical self-healing of the composites. In some cases, the stiffness of the cracked ECC can recover completely.
- The tensile strain capacity of all re-healed ECCs remains above 1.5%, even higher if the residual strain due to preloading damage is accounted for.
- Higher conditioning temperature (EC2) tends to promote the degree of hydration at early age, and therefore increase matrix toughness as well as fiber/matrix interfacial bonding. This results in higher tensile strength and lower tensile strain capacity of self-healed ECC subjected to EC2 (water/hot air cycle).
- From the EDX results, the reheal product at early

age is likely a compound of calcium carbonate (precipitation/crystallization) and calcium hydroxide (further hydration). Further investigation by x-ray diffraction analysis is necessary in the future to reveal the chemical composition of reheal products.

Based on this study which employs a variety of environmental conditioning, 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. Thus ECC combines high ductility, high strength, and self-healing capability. Further studies in the field should be conducted to confirm this expectation in actual structures exposed to natural environments and the precise chemical composition of reheal products remains to be explored.

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