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Repeatability and Pervasiveness of Self-Healing in Engineered Cementitious Composites

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This paper investigates the intrinsic self-healing ability of engineered cementitious composites (ECCs) coupled with multiple microcrack formation under mechanical loading based on two robustness criteria: repeatability and pervasiveness. To this end, two different composites containing Class F fly ash and slag were investigated. To generate microcracks, specimens were repeatedly preloaded up to 70% of their deformation capacities under mechanical loading at the end of each specified cyclic wet/dry conditioning period. Resonant frequency (RF) and rapid chloride permeability tests (RCPT) were used to assess the extent of damage and self-healing, and final results were supported by microscope observations. RF measurements were recorded from two different parts of each specimen (the top and middle portions) to monitor whether self-healing takes place in certain regions or whether it is pervasive over the entire specimen. Results of the experimental study show that depending on the type of mineral admixture used and the duration of initial curing before deterioration, ECC specimens can recover up to 85% of their initial RF measurements, even after six repetitive preloading applications. The recovery rates observed in the middle portion are similar to those in the top portion for both ECC mixtures (to a slightly lesser extent), which implies that self-healing is quite pervasive. Furthermore, after repeated application of severe preloading, RCPT results for both mixtures satisfy low or moderate chloride ion penetrability levels in accordance with ASTM C1202. Due to the enhanced self-healing capability of specimens, maximum crack width observed over the specimen surfaces was restricted to 190 μm (0.008 in.), even after nine preloadings. These findings suggest that under certain conditions, the ECC materials produced in this study may significantly enhance the functionality of structures by reducing the need for repair and/or maintenance.

Keywords: engineered cementitious composite (ECC); repeatability; self-healing; supplementary cementitious material (SCM).

INTRODUCTION

It is highly desirable that concrete material used in transport structures lasts for a long time without sacrificing its mechanical and durability properties. However, due to a number of deteriorating mechanisms, concrete unavoidably cracks, which can lead to more severe degradation processes and significant reductions in service life. Such damage frequently requires repeated maintenance and/or repair of degraded sections to compensate for lack of performance. However, previous studies report that repairs are often short-lived and that half of repairs applied in the field end up failing.[1] To make matters worse, continuous maintenance and/or repair work is not always feasible, especially in the case of large-scale concrete infrastructures, due to the significant time, labor, and capital required. In addition, owing to structural restrictions and conditions, repairs can be difficult or even impossible to execute in some cases. Under such circumstances, self-repair or self-healing of concrete material without external human interference is an attractive alternative.

To date, many researchers have engaged in the study of emerging techniques including the use of hollow fibers,[2] chemical and bacterial encapsulation,[3,4] expansive agents and mineral admixtures,[5,6] and intrinsic self-healing with self-controlled tight crack width.[7] Although all of the approaches listed above hold promise, there are still a number of concerns related to the robustness of the self-healing mechanism.[8] One such concern is the repeatability of the self-healing phenomenon. Over the lifetime of an infrastructure, damage is likely to occur more than once due to multiple overloading and environmental conditions. Self-healing capability should therefore be repeatable to satisfy functionality, even in cases where crack opening occurs from the same location as previous occurences. Another concern related to self-healing robustness is the complexity posed by different loading types on structures. Because it is not always easy to predict cracking location, orientation, and depth, healing that is pervasive rather than only present in discrete parts of the structure is extremely appealing. Although the aforementioned self-healing approaches have limited effectiveness in fulfilling robustness criteria, the use of intrinsic self-healing, coupled with the formation of many closely spaced microcracks, seems to be the most promising technique in situations where repeatability and pervasiveness of the self-healing mechanism are of concern.[8] The formation of many closely spaced microcracks is very difficult to achieve in conventional and fiber-reinforced concrete (FRC) with the application of mechanical and environmental loading. Therefore, the possibility of attaining consistent and robust self-healing behavior with conventional concrete is small. However, a newly developed material called engineered cementitious composites (ECC) has superior characteristics that outperform conventional concrete and FRC[9,10] in terms of self-healing behavior.

ECC is part of a special category of high-performance fiber-reinforced cementitious composites developed in the last few decades. The optimization of ECC is based on the application of micromechanics-based design theory to attain

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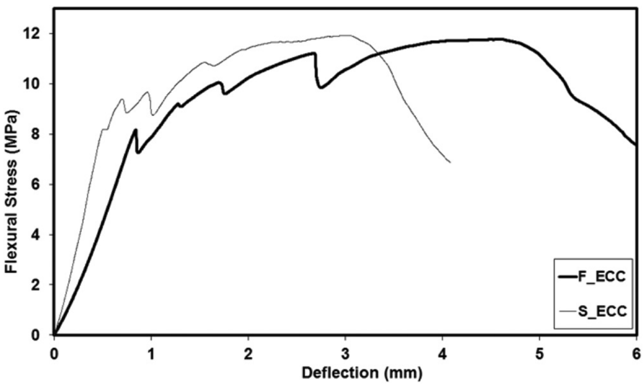


Fig. 1—Typical flexural stress: midspan deflection curves of ECC mixtures at 28 days.

high tensile ductility and tight microcrack formation under direct tension at moderate fiber content (2% by volume, or less).[11] The extreme ductility of ECC is several hundred times that of conventional concrete and FRC. Increased ductility leads ECC materials to exhibit exceptional enhancement in toughness values, similar to that of metals.[12] Along with the improvement in toughness value, average crack width remains at about 60 μm (0.002 in.) regardless of ultimate tensile strain. The availability of information related to this phenomenonm, especially in the case of multiple overloading conditions, is lacking in the literature, as is the answer to the question of whether self-healing occurs across entire structures or is limited to certain areas. To account for this knowledge gap, a detailed study was undertaken to gain an understanding of the self-healing phenomenon observed in ECC materials in terms of repeatability. To do so, 3- and 28-day ECC specimens incorporating different supplementary cementitious materials (SCMs) (Class-F fly ash and slag) were produced. Then, on specified days, specimens were subjected to repetitive preloading applications under splitting tensile loading and subsequent environmental conditioning to simulate self-healing behavior in cases where crack formation takes place multiple times. Repeatability of the self-healing mechanism was assessed by the use of rapid chloride permeability (RCPT) and resonant frequency (RF) tests. RF measurements were taken from different parts of the specimens (top, bottom, and middle portions) to understand the extent of self-healing in different regions of specimens. Crack characteristics of repeatedly preloaded specimens were also evaluated based on the results obtained from the surfaces of RF specimens.

RESEARCH SIGNIFICANCE

Although self-healing of cement-based materials is of interest to many researchers, there is a lack of information related to this phenomenon, especially in the case of multiple overloading conditions. The question of whether self-healing takes place in the entire structure or is limited to certain areas has also not been adequately answered in previous studies. To account for this knowledge gap, an experimental study was undertaken to examine the influence of different SCMs and initial curing times on two robustness criteria of self-healing: repeatability and pervasiveness.

Table 1—ECC mixture proportions

Mixture ID	Ingredients, kg/m^3						*MA/PC*	*W/CM*
	PC	MA	Water	PVA	Sand	HRWRA		
F_ECC	566	680	331	26	453	5.1	1.2	0.27
S_ECC	593	712	347	26	474	6.0	1.2	0.27

Note: 1 kg/m^3 = 1.685 lb/yd^3.

Table 2—Chemical and physical properties of portland cement, fly ash, and slag

Chemical composition	PC	F	S
CaO, %	61.43	3.48	35.09
SiO_2, %	20.77	60.78	37.55
Al_2O_3, %	5.55	21.68	10.55
Fe_2O_3, %	3.35	5.48	0.28
MgO, %	2.49	1.71	7.92
SO_3, %	2.49	0.34	2.95
K_2O, %	0.77	1.95	1.07
Na_2O, %	0.19	0.74	0.24
Loss on ignition	2.20	1.57	2.79
$SiO_2 + Al_2O_3 + Fe_2O_3$, %	29.37	87.94	48.38
Physical properties			
Specific gravity	3.06	2.10	2.79
Blaine fineness, m^2/kg	325	269	425

Note: 1 m^2/kg = 0.54 yd^2/lb.

EXPERIMENTAL PROGRAM

Materials and mixture proportions

Two different ECC mixtures incorporating Class F fly ash (F_ECC) and ground-granulated blast-furnace slag (S_ECC), which has the largest experimental dataset published to date, were prepared. Figure 1 shows typical stress-deflection graphs of both mixtures obtained under four-point bending loading, which exhibit superior deflection-hardening behavior of the specimens at the end of 28 days. To be used for bending tests, prism specimens with dimensions of 360 x 50 x 75 mm (14.2 x 1.9 x 2.9 in.) were used and during the tests span length of flexural loading was 304 mm (12 in.) with a 101 mm (4 in.) center span length. In both mixtures, the water-cementitious material ratio (*W/CM*) and mineral admixture (fly ash [F] and slag [S])-portland cement ratio (*MA/PC*) were kept constant at 0.27 and 1.2, respectively. The details of mixture proportions are shown in Table 1. Both ECC mixtures contain CEM I-42.5 cement (similar to ASTM Type I), fine silica sand with maximum aggregate size of 400 μm (0.016 in.), water, polyvinyl alcohol fibers, and a polycarboxylic-ether type high-range water-reducing admixture (HRWRA) with a solid content of 40%. Chemical composition and physical properties of cement, fly ash, and slag are presented in Table 2.

A series of cylindrical specimens with the dimensions of Ø150 x 300 mm (5.9 x 11.8 in.) was cast from a single batch produced, using a 65 L capacity (0.085 yd^3) gravity mixer in resonant frequency (RF) tests. Specimens were demolded after 24 hours and cured in plastic bags at 95 ± 5% RH and at 23 ± 2°C (73.4 ± 3.6°F) until the predetermined testing ages. For rapid chloride permeability tests (RCPT), cylinder specimens measuring Ø100 x 200 mm (3.9 x 7.9 in.) were produced and kept in plastic bags at 95 ± 5% RH and at 23 ± 2°C (73.4 ± 3.6°F). At the age of 28 days, cylindrical specimens with dimensions of Ø100 x 50 mm (3.9 x 2.0 in.) were extracted with a diamond blade saw and used for RCPT tests.

Precracking and methods to evaluate repeatability and pervasiveness of self-healing

Previous studies have shown that dynamic modulus measurement based on ASTM C215[13] and chloride ion penetrability measurement (RCPT) based on ASTM C1202[14] appear to be particularly promising and relatively simple techniques for monitoring the extent and rate of autogenous healing.[10,15] Although these test methods do not quantify damage, they provide useful ways of measuring the extent and rate of self-healing without disturbing specimens in cases where healing was observed as a reduction in material damage. For this reason, this study used these test methods to observe self-healing rates under repetitive preloading conditions. To monitor self-healing repeatability, transverse RF measurements based on ASTM C215[13] were taken from Ø150 x 300 mm (5.9 x 11.8 in.) cylinder specimens. Before being exposed to cyclic preloading, four 3-day-old and four 28-day-old cylinders from both mixtures were tested until failure under splitting tensile loading to define ultimate deformation capacities of the ECC mixtures. Ultimate splitting deformation capacities were in the range of 2.0 to 2.6 mm (0.079 to 0.102 in.), in accordance with mineral admixture type and initial curing period. The actual tests were started after 3 and 28 days of initial curing, and for each testing date, five cylindrical specimens were used for RF measurements. Two were kept as virgin (untouched) and the others were repeatedly preloaded up to 70% of their original measured deformation capacities under splitting tensile loading to achieve various amounts of microcracking. Preloading was applied in each group of 10 cycles up to 90 cycles for RF specimens. After each preloading, the longitudinal sides of the specimens were sealed to restrict water flow along lateral directions. Between each preloading interval, specimens were subjected to wet-dry (W/D) cycles consisting of submersion in water at 23 ± 2°C (73.4 ± 3.6°F) for 24 hours and drying in a laboratory medium at 50 ± 5% RH and 23 ± 2°C (73.4 ± 3.6°F) for 24 hours. This type of conditioning was selected with the intention of better simulating the actual field conditions since most of the structures in our daily lives are somehow subjected to actions of wetting and drying. Because each W/D cycle represents a period of 2 days, each 20-day interval corresponds with 10 complete W/D cycles. To determine whether self-healing is restricted to certain regions of the specimens or is widely dispersive over the entire area, RF measurements were taken from four different points, two at the top and bottom and two in the middle portions of the Ø150 x 300 mm (5.9 x 11.8 in.) cylinder specimens (Fig. 2). During the evaluation of the RF test results, the average of the top and bottom point measurements was accepted as the surface RF measurement, and the average of the two middle point measurements as the inside RF measurement. Additionally, changes in the widths of cracks formed over the specimens were observed with the help of microscope at the end of each 10 W/D cycles.

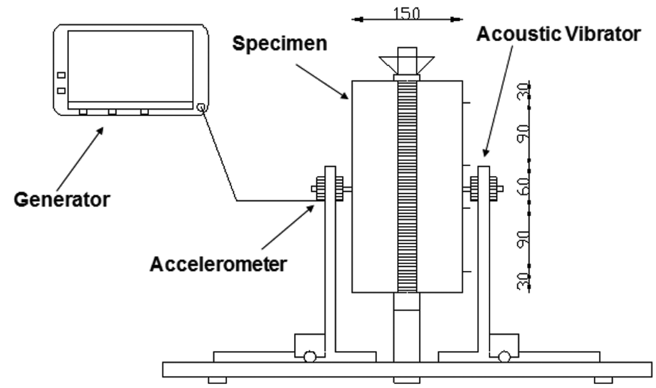


Fig. 2—Schematic diagram of resonant frequency measurements at different depths. (Note: units in mm; 1 mm = 0.039 in.)

Repeatability of self-healing was also monitored through RCPT tests. Ten 28-day-old cylindrical ECC specimens with the dimensions of Ø100 x 50 mm (3.9 x 2.0 in.) were tested from each mixture, based on ASTM C1202.[14] Before the application of preloading, the ultimate splitting deformation capacities of Ø100 x 50 mm (3.9 x 2.0 in.) cylinder specimens after 28 days were found by taking the average results of four specimens. The ultimate splitting deformation capacities of F_ECC and S_ECC specimens were 1.7 mm (0.067 in.) and 1.5 mm (0.059 in.), respectively. By taking the final results into consideration, it was decided to keep four of the specimens as virgin and to repeatedly preload the remaining six specimens up to 70% of their maximum splitting deformation capacity under splitting tensile loading. Preloaded specimens were then exposed to up to 100 W/D cycles; RCPT measurements were recorded every 20 days.

RESULTS AND DISCUSSION

Resonant frequency (RF) test

Unhealed specimens—Resonant frequency (RF) test results of ECC mixtures are presented in Fig. 3. In the figure, measurements taken from the top and middle portions of 3- and 28-day virgin and preloaded ECC specimens are illustrated. Results were achieved by using the initial RF measurements and percentage variations observed beyond 3 and 28 days until the end of 90 W/D cycles. When both figures are carefully examined, it can clearly be seen that there were changes in RF results depending on initial curing period, SCM type, and number of W/D cycles. Original RF values obtained from the top and middle portions of the 3-day-old F_ECC specimens were 1870 and 1515 Hz, respectively. In the case of 28-day-old F_ECC specimens, the same values were 2050 and 2000 Hz. Original RF values recorded from 3-day-old S_ECC specimens were 1925 and 1695 Hz for the measurements taken from top and middle portion of the specimens, respectively. The same values were 2070 and 2000 Hz in the 28-day-old S_ECC specimens. As seen from Fig. 3(a) and (e), an increasing trend in RF results was monitored for 3-day-old virgin specimens up to the end of 90 W/D cycles, irrespective of the point where the RF reading was taken and the type of SCM used.

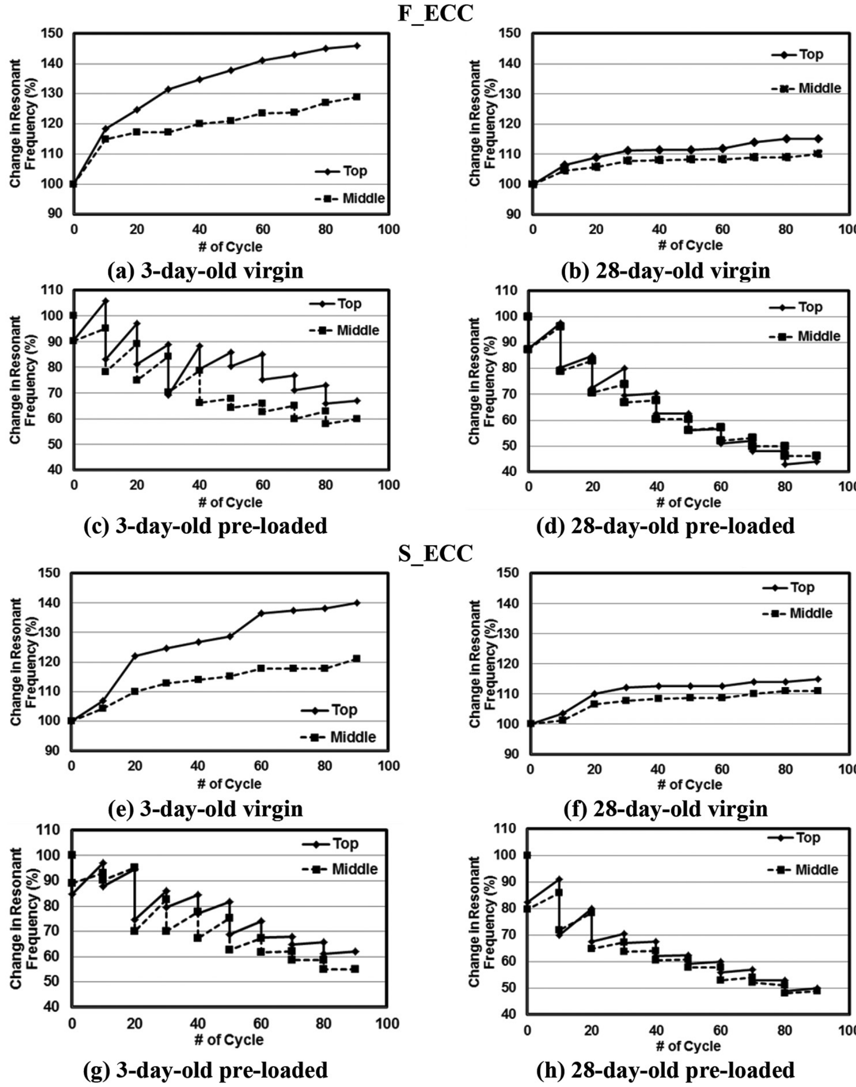


Fig. 3—Percent variations in RF measurements due to repetitive preloading and subsequent conditioning.

The increase in RF results was similar for both the F_ECC and S_ECC mixtures. For example, at the end of 90 W/D cycles for 3-day-old virgin specimens, increments in RF results reached up to 146% and 140% of the initial readings for measurements taken from the top portions of F_ECC and S_ECC specimens, respectively. The same modality held true for readings taken from the middle portions, with values reaching 129% and 121% for the same mixtures. Although the final RF results of 3-day-old virgin ECC specimens incorporating fly ash and slag were similar, F_ECC specimens showed slightly better performance than S_ECC specimens in terms of changes in RF measurements. The probable reason for this behavior in F_ECC specimens may be due to pozzolanic reactions, which could result in greater improvements in RF results compared with S_ECC specimens. This result implies that there is a higher probability of F_ECC specimens consuming higher amounts of the calcium hydroxide available in the hydrated matrix, especially at late ages, which could lead to greater improvements in RF measurements compared to S_ECC specimens.

The difference in RF results was less pronounced for virgin specimens initially cured for 28 days. As seen from Fig. 3(b) and (f), after 90 W/D cycles, 28-day-old specimens from both mixtures exceeded only 15% and 11% of the initial RF results for readings taken from the top and middle portions of specimens, respectively. It is also obvious from the same figures that 20 W/D cycles were almost adequate for complete stabilization of the results in the case of 28-day-old ECC specimens. These specimens may have showed lower enhancement in RF with increased initial curing time, due to lower amounts of SCMs readily available to participate in further hydration reactions and reductions in pore size, densification of matrixes and consequently lower overall transport of the moisture needed for further hydration.

Another point worth mentioning in the case of virgin specimens: regardless of initial curing time and type of SCM, RF measurements taken from the top portion of specimens were higher than those taken from middle portion at the end of any number of W/D cycles. This behavior was observed continually in nearly all of the specimens; the overall coefficient of variation was less than 7%, showing consistency of results. The behavior is most probably attributable to the changes in internal relative humidity of ECC specimens in different regions. According to Jiang et al.,[16] for cement pastes with a *W/CM* higher than 0.40, moisture diffusion is the only mechanism that leads to a reduction in internal relative humidity; moisture diffusion and self-desiccation due to chemical shrinkage of the paste have a simultaneous influence on the reduction of internal relative humidity in pastes with a *W*/*CM* less than 0.40. Therefore, for ECC specimens with a *W*/*CM* of 0.27, it can be stated that during the W/D cycles, diffusion of moisture to the short distances (points where the top and bottom RF measurements are taken) was faster compared to longer distances (points where the middle RF measurements are taken). This led to hydration kinetics being faster and self-desiccation being counteracted more easily at the top and bottom portion of specimens, which resulted in higher RF measurements at the end of specified testing cycles.

Effects of self-healing—Figures 3(c), (d), (g), and (h) show percentage changes in RF measurements taken from specimens repeatedly preloaded up to 70% of their maximum deformation capacities under splitting tensile loading at the end of each group of 10 W/D cycles until completion of 90 W/D cycles. Three specimens from each mixture were initially cured for 3 and 28 days, and were then used to investigate the likelihood of specimens exhibiting cracks in varying numbers and widths at the end of each preload application. The averaged results are shown in these figures. As the figures make it clear, the application of preloading and exposure to different numbers of W/D cycles resulted in fluctuations with varying rates. After each preloading at the end of each group of 10 W/D cycles, some reduction in RF results was monitored. Results reflecting the self-healing effect considerably changed depending on SCM type, number of W/D cycles and initial curing time. As seen from Fig. 3(c), (d), (g), and (h), the decrease in RF measurements with the application of preloading showed significant recovery at the end of different numbers of W/D cycles, although this trend was not long-lasting. Careful analysis of RF measurements from the top portion of 3-day-old ECC specimens shows that reduced RF measurements with the applied preloadings were not substantially recovered and recovery rates after each period were similar beyond exposure to 60 W/D cycles. The most probable reason for this equilibrium is the excessive widening of cracks upon preload repetitions and gradual exhaustion of unhydrated cementitious materials at late ages to account for crack closure through self-healing. However, despite the stabilization of RF results beyond a certain time period, after six preloading applications, average RF results were only 85% and 74% of the initial RF measurement for F_ECC and S_ECC mixtures, respectively. This shows that the ECC materials produced in this study performed well in terms of RF recovery under certain conditions, implying the occurrence of self-healing, which could substantially decrease repetitive repair and/or maintenance needs. As stated above, the recovery of RF results just before stabilization was higher for F_ECC (85%) specimens than for S_ECC (74%), and this tendency was observed to be true for each RF measurement taken after each interval of 10 W/D cycles. This result can be attributed to the changes in matrix composition of ECC specimens. As previously mentioned, the Class-F fly ash used in this study contains more SiO_2, which is important for pozzolanic activity, especially at later ages, while slag contains higher amounts of CaO, which is mostly responsible for self-cementing behavior. Therefore, it is possible to conclude that S_ECC specimens performed their cementing reactions earlier than F_ECC specimens, and this led to faster consumption of unhydrated cementitious materials in S_ECC mixtures. F_ECC specimens showed greater improvements in RF measurements after each preloading and subsequent W/D cycles owing to the abundance of unhydrated fly ash particles, even at later ages. According to Sahmaran et al.,[17] ECC specimens incorporating fly ash exhibited significantly tighter crack widths than specimens with slag due to increased limitations in matrix fracture toughness values. Thus, greater improvements in RF results in F_ECC specimens could also be attributed to the reduced crack widths occurring with the application of cyclic preloading.

It is clear from Fig. 3(d) and (h) that the repeatability of self-healing in terms of RF measurements decreased significantly in specimens initially cured for 28 days. As seen from the figures, the recovery results of both ECC mixtures showed stabilization after 30 W/D cycles, regardless of the point of measurement. Average recoveries obtained from the top portions were 80% and 71% of the initial RF measurements for 28-day-old F_ECC and S_ECC specimens after three preload repetitions and 30 W/D cycles, beyond which minimal or no further RF recovery was monitored. The explanation for this stabilization could be that due to increased initial curing time, the matrix maturity of specimens enhanced significantly, leaving fewer unhydrated cementitious materials available for the further hydration reactions needed to prompt self-healing behavior. Moreover, due to the continuity of hydration reactions, the matrix gets more compact at later ages, leading to an increase in matrix-fiber frictional bond strength and fracture toughness values. Increments in these parameters are detrimental to the attainment of sufficient multiple microcracking and could trigger localized crack formations with wider widths, which may negatively influence crack closure through self-healing at later ages.[17]

As previously mentioned, two RF measurements were taken from the surface and middle portions of the ECC specimens to better understand whether self-healing is limited to certain regions of specimens with repeated preload applications and subsequent W/D conditioning, or whether it is dispersive over the entire area. Generally speaking, RF measurements from the middle portions of specimens were lower compared to the recovery rates observed in the surface portions (Fig. 3). This behavior was more explicit for 3-day-old F_ECC, and to a lesser extent, for 3-day-old S_ECC mixtures. When the results from both surface and middle portions of 3-day-old mixtures are compared, it is evident that the number of recovered preload repetitions before the stabilization of results did not change, although the extent of recovery decreased in measurements taken from the middle portions, regardless of mixture type. For example, surface portion measurements taken after the application of six repetitive preloadings were 85% and 74% of recovery observed in RF results for 3-day-old F_ECC and S_ECC mixtures, respectively. The values were 66% and 67% for the same mixtures in measurements taken from the middle portions. The same modality was true for 28-day-old specimens, except

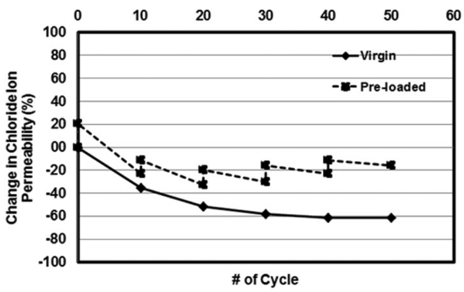
Table 3—Rapid chloride permeability test results of ECC specimens

Mixture ID		0 cycles		0 + 10 cycles		0 + 20 cycles		0 + 30 cycles		0 + 40 cycles		0 + 50 cycles	
		Zero	AP*	BP†	AP	BP	AP	BP	AP	BP	AP	BP	AP
F_ECC	Virgin	4154	4154	2673	2673	1995	1995	1749	1749	1613	1613	1597	—
		(9.7)	(9.7)	(2.3)	(2.3)	(3.3)	(3.3)	(4.0)	(4.0)	(7.2)	(7.2)	(5.3)	—
	Preloaded	4082	4925	3141	3611	2753	3279	2842	3440	3150	3626	3440	—
		(4.6)	(6.7)	(5.8)	(7.5)	(8.1)	(7.0)	(1.7)	(3.2)	(2.7)	(9.6)	(3.2)	—
S_ECC	Virgin	995	995	759	759	662	662	497	497	465	465	441	—
		(5.5)	(5.5)	(4.9)	(4.9)	(0.53)	(0.53)	(2.0)	(2.0)	(7.3)	(7.3)	(5.0)	—
	Preloaded	1064	1583	1160	1547	1302	1618	1409	1733	1639	1956	1885	—
		(7.3)	(6.5)	(6.1)	(5.7)	(6.9)	(5.5)	(5.5)	(6.1)	(6.5)	(5.6)	(6.4)	—

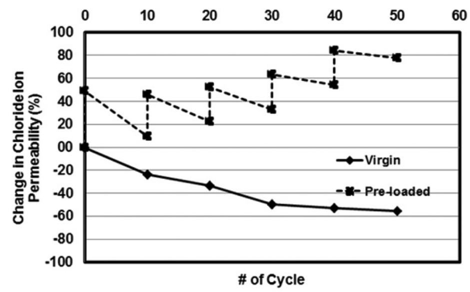
*After preloading.

†Before preloading.

Note: Numbers in parentheses are coefficient of variation (COV).



F_ECC



S_ECC

Fig. 4—Percent variations in chloride ion permeability values of ECC mixtures due to repetitive preloading and subsequent conditioning.

for the fact that the number of recovered preload repetitions decreased before the stabilization of RF results. In the case of the 28-day-old specimens, after three repetitive preload applications, RF results taken from the surface portions started to stabilize at 80% and 71% levels for F_ECC and S_ECC mixtures, respectively, while results from the middle portions reached 71% and 67% for the same mixtures. It can be deduced from these observations that near-surface cracks, which are more easily exposed to water, can be healed up to 85% even after six bouts of severe deformation exposure due to ease of moisture transport over shorter distances.

Rapid chloride permeability test (RCPT)

Unhealed specimens—Chloride ion permeability test results of ECC specimens are tabulated in Table 3. All data presented are averages of four specimens. The results are expressed in terms of total electrical charge in Coulomb, which reflects ECC materials' ability to resist chloride ion ingress. Because different SCMs show varying characteristics and low paste maturity at early ages, RCPTs were started after 28 days of initial curing to account for higher-than-normal results.

According to the results obtained from virgin specimens (Table 3), it is clear that undergoing 50 W/D cycles caused marked reductions in RCPT results, regardless of SCM type. Until the end of 50 W/D cycles, results showed a decreasing trend between 4154 and 1597 and 995 and 441 Coulomb in F_ECC and S_ECC mixtures, respectively. Another point that is visible at first glance is that the drop in chloride ion penetrability results of F_ECC specimens was significantly higher than it was for S_ECC specimens. This behavior of F_ECC specimens is more noticeable in Fig. 4, which shows the percentage changes in chloride ion permeability results with respect to the first measurement taken after 28 days of initial curing versus number of W/D cycles. As seen from the figure, the reduction in chloride ion charge/number of cycle slope was found to be more explicit for F_ECC specimens than for S_ECC specimens, especially after the first 20 W/D cycles. For example, at the end of 20 W/D cycles, average chloride ion penetrability results of F_ECC specimens dropped by 52%, while S_ECC specimens only decreased by 33%. As the number of W/D cycles increased, the chloride ion charge-number of cycle curve slope smoothed out for both ECC mixtures due to gradual diminishment of unhydrated cementitious materials with time. The probable reason for F_ECC virgin specimens exhibiting notable improvement in chloride ion penetrability results compared to S_ECC specimens could be due to the fact that specimens incorporating slag have a greater quantity of reacted particles in comparison to specimens incorporating fly ash. This behavior of F_ECC specimens is also evident in the fact that fly ash particles (especially Class-F fly ash) remained untouched in the system up to 30 to 40% without any chemical process,[18] requiring longer periods to react and produce hydration products such as calcium-silicate-hydrate (C-S-H) gels through pozzolanic reactions. Although the improvement in final values was more pronounced for F_ECC specimens at the end of each W/D cycle, the chloride ion penetrability of S_ECC specimens was significantly lower than in F_ECC specimens after any number of W/D cycles. S_ECC specimens may have shown lower RCPT results than F_ECC specimens due to finer particle size and the higher cementing behavior of slag particles. Table 2 shows that slag (425 m^2/kg [230 yd^2/lb]) is significantly finer than Class F fly ash (269 m^2/kg [145 yd^2/lb]). Therefore, it can be deduced that the microstructure of S_ECC specimens became more compact due to enhanced filler effect and the higher cementing capability of finer slag particles, especially during the early stages of hydration. Because RCPT is an electrochemical test method used for the evaluation of chloride ion permeability, the electrical conductivity of the pore solution should also be accounted for during the assessment of results. Electrical conductivity of the pore solution, which can be critical for chloride ion penetrability test results, can be reduced significantly by lowering the concentration of alkali ions (Na^+ and K^+) and associated hydroxyl ions (OH^-).[19] Thus, the lower alkali content of slag particles may also be a reasonable explanation for the reduced RCPT results of S_ECC specimens (see Table 2). It must be emphasized that although the rate of improvement in RCPT results was lower for virgin S_ECC mixtures, even without the application of further W/D cycles, average chloride ion permeability results of virgin S_ECC specimens were under 1000 Coulomb (995 Coulomb), regarded as the maximum value by ASTM C1202[14] in terms of resistance to chloride ion ingress. Virgin F_ECC specimens could never reach this level of chloride transport properties.

Effects of self-healing—Chloride ion permeability test results on repeatedly preloaded cylindrical (Ø100 x 50 mm [3.9 x 2.0 in.]) specimens are presented in Table 3. Data provided in Table 3 was obtained by taking the average of four specimens. With the application of repetitive preloading and the cyclic W/D curing regime, results varied significantly. Figure 4 indicates that both ECC mixtures showed self-healing, with decreasing chloride ion penetrability results after each W/D cycle. The rate of self-healing for S_ECC specimens was higher than for F_ECC specimens, which was constant throughout the entire W/D cyclic exposure. However, the difference between the healing rates of the ECC mixtures became less explicit due to a reduction of unhydrated cementitious particles over time. Although it is less prominent in the S_ECC specimens, the healing rate for both mixtures decreased significantly beyond 30 W/D cycles. This finding is in line with previously mentioned conclusions about RF measurements taken from specimens initially cured for 28 days.

Another striking point is that the application of five repetitive preloading and 50 successive W/D cycles led the average RCPT results of F_ECC specimens to decrease from 4082 to 3440 Coulomb, and the results of S_ECC specimens to increase from 1064 to 1885 Coulomb (Table 3). In other words, while the chloride ion penetrability test results of S_ECC specimens increased up to 77% of the initial reading after five preload applications and corresponding W/D cycles, F_ECC specimens decreased by 16% (Fig. 4). Although comparable self-healing took place in both mixtures, after each preloading and corresponding cyclic W/D curing, the RCPT results of F_ECC specimens showed a decreasing trend with respect to the first result taken at the end of 28 days, while S_ECC specimens showed the opposite. This behavior can be attributed to greater fiber-matrix frictional bond strength and fracture toughness of S_ECC specimens compared to F_ECC specimens, which may have significantly reduced the chance of multiple crack occurrence and increased crack widths with the application of repetitive preloading. Moreover, S-ECC specimens with an average 1064 Coulomb RCPT result after 28 days of initial curing already showed very high resistance to chloride ion penetration; beyond this time period, the possibility for further improvements was significantly reduced. Therefore, despite the self-healing performance of S_ECC specimens observed after each number of W/D cycles, the formation of cracks with wider widths (due to increased paste maturity) and faster consumption of inner materials (due to enhanced cementitious behavior) suppressed self-healing and caused inadequate sealing of cracks, which led to a marked increase in final RCPT results in S_ECC specimens (see "Crack characteristics of preloaded specimens" section). This finding is a strong indication of the dependence of self-healing on tight crack formation and availability of particles to be hydrated. However, it is important to point out that under certain environmental conditions, even after five repetitive severe preload applications, chloride ion penetrability results of both ECC mixtures produced in this study (3440 and 1885 Coulombs for F_ECC and S_ECC, respectively) satisfy the moderate or low chloride ion penetrability levels prescribed by ASTM C1202.

Crack characteristics of preloaded specimens

The number of cracks and minimum, maximum, and total crack opening measurements of 3- and 28-day-old ECC specimens subjected to repetitive preloading and W/D cycles are summarized in Table 4. Data presented in the table represent average measurements recorded over two opposite faces of three cylindrical specimens used for RF tests. As seen from Table 4, despite significant self-healing of cracks upon exposure to subsequent cyclic W/D conditioning, an increase in the number of preload applications increased both number of cracks and total crack widths to a certain level. A higher number of cracks was observed in F_ECC specimens than in S_ECC specimens. Although this characteristic of F_ECC specimens was observed to be true at the end of each preload application and W/D cyclic exposure, it was more explicit for specimens initially cured for 28 days. The probable reasons that might lead F_ECC specimens to show a higher number of cracks have already been explained in previous sections and will not be further discussed here.

Figure 5 shows the rate of total crack closure observed over entire crack surfaces in percentages, as well as the number of cycles applied to 3- and 28-day-old specimens. The values in Fig. 5 were calculated by the equation

$$\frac{PL_{TCW(t)} - SH_{TCW(t+10)}}{PL_{TCW(t)}} \times 100$$

where $PL_{TCW(t)}$ and $SH_{TCW(t+10)}$ denote preloaded total crack width at cycles t and self-healed total crack width at cycles

Table 4—Crack characteristics of preloaded and self-healed ECC specimens

Mixture ID	No. of cycles	3-day-old								28-day-old							
		Preloaded				Self-healed				Preloaded				Self-healed			
		No. of cracks	Crack width, μm			No. of cracks	Crack width, μm			No. of cracks	Crack width, μm			No. of cracks	Crack width, μm		
			Min.	Max.	Total		Min.	Max.	Total		Min.	Max.	Total		Min.	Max.	Total
F_ECC	0	7 to 11	10	100	470	—	—	—	—	5 to 8	20	110	520	—	—	—	—
	10	8 to 13	20	100	490	3 to 9	10	70	280	6 to 9	20	110	550	3 to 6	20	80	390
	20	8 to 14	20	110	530	4 to 10	20	80	300	6 to 11	10	120	580	4 to 7	20	90	450
	30	9 to 14	20	110	560	6 to 11	30	80	350	7 to 11	20	120	630	5 to 9	30	90	520
	40	10 to 14	10	110	610	7 to 12	20	80	390	8 to 11	20	130	710	7 to 10	10	110	600
	50	11 to 14	20	120	640	9 to 13	20	90	475	9 to 12	20	130	750	7 to 11	20	110	680
	60	11 to 15	10	120	670	9 to 14	10	100	550	9 to 12	30	130	790	8 to 11	20	120	720
	70	12 to 15	30	120	690	10 to 14	20	110	590	10 to 12	20	130	810	9 to 12	30	120	760
	80	12 to 16	20	120	710	11 to 14	20	100	660	10 to 13	20	140	830	10 to 12	10	120	790
	90	13 to 16	20	120	740	11 to 15	10	100	690	11 to 13	30	140	860	10 to 12	20	130	810
S_ECC	0	6 to 9	20	100	560	—	—	—	—	4 to 7	20	150	540	—	—	—	—
	10	6 to 10	10	100	530	3 to 6	10	60	280	5 to 8	30	150	570	3 to 5	20	110	310
	20	7 to 10	20	110	550	4 to 8	20	60	310	6 to 8	30	150	630	4 to 6	20	120	370
	30	7 to 10	20	110	580	5 to 8	10	70	370	6 to 9	40	150	740	5 to 7	10	120	570
	40	8 to 12	20	120	680	6 to 9	20	90	460	7 to 9	30	160	780	6 to 8	30	130	680
	50	9 to 12	30	120	720	7 to 10	30	110	580	8 to 9	20	160	810	7 to 9	20	130	720
	60	10 to 12	30	140	880	8 to 11	10	110	650	8 to 9	30	180	820	7 to 9	30	150	770
	70	11 to 13	30	150	920	9 to 12	20	110	820	8 to 9	40	180	890	8 to 9	20	170	800
	80	11 to 13	20	150	1010	10 to 12	10	140	870	8 to 9	40	200	990	8 to 9	30	190	870
	90	12 to 13	20	160	1070	10 to 12	10	150	980	8 to 9	40	200	1080	8 to 9	40	190	980

Note: 1 μm = 0.00004 in.

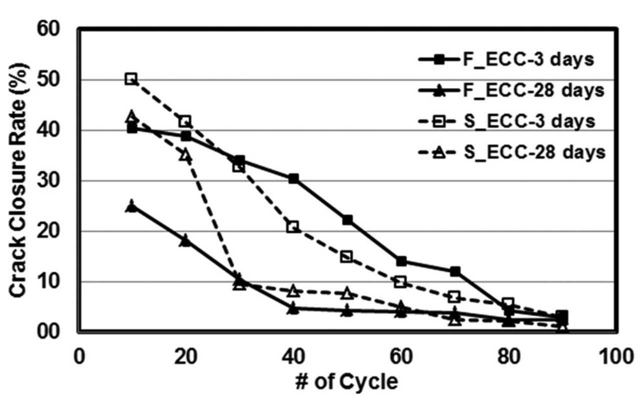


Fig. 5—Total crack closure rates of ECC specimens due to self-healing.

t+10, respectively. When the crack closure rates of 3-day-old ECC specimens exposed to cyclic preloading and subsequent W/D conditioning are evaluated, it is evident that comparable self-healing took place in both mixtures, although sealing of cracks was slightly better in F_ECC specimens (Fig. 6). For specimens initially cured for 28 days, however, the total healing of cracks over the surfaces of S_ECC specimens was more pronounced, especially at early ages. The extent of self-healing monitored in the 28-day-old S_ECC specimens is rather surprising, since the crack widths were larger than expected (Table 3), and significantly lower amounts of unhydrated cementitious materials were expected to be present in S_ECC matrixes, especially at later ages, due to the enhanced cementing behavior of slag. The increased self-healing capability of S_ECC specimens may be associated with the high pH value of the pore solution, which may contribute to higher amounts of Ca^{2+} ions being leached away from C-S-H gels and/or calcium hydroxide, leading to enhanced $CaCO_3$ growth and more evident final self-healing behavior.[7,20] Although the higher degree of self-healing was observed in the 28-day-old S_ECC specimens, the final self-healed condition of specimens was not as complete as that of the F_ECC specimens; at the end of nine preloadings and 90 W/D cycles, total crack opening measured over specimen surfaces was 810 μm (0.032 in.) and 980 μm (0.039 in.) for F_ECC and S_ECC specimens, respectively. This suggests that although self-healing kinetics are faster in bigger cracks,[21] the availability of required compounds and internal water limit the extent, and small crack widths are crucial for complete sealing. It is important to emphasize that under certain environmental conditions, even after nine repetitive preload applications, the maximum individual crack widths observed in 28-day-old specimens were limited to 130 μm (0.006 in.) and 190 μm (0.008 in.) for F_ECC and S_ECC, respectively. These values are still below the minimum

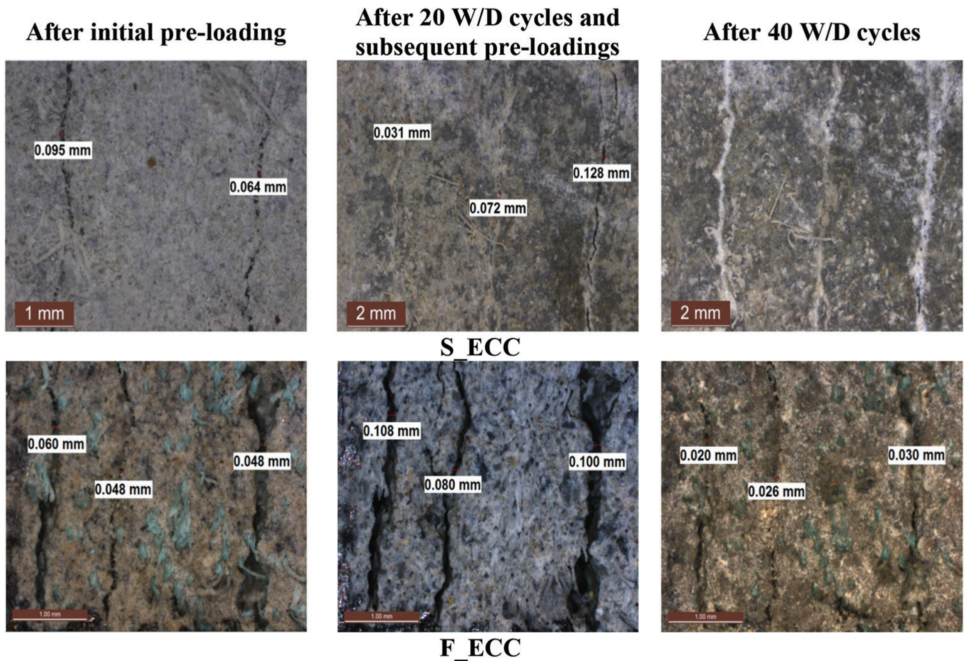


Fig. 6—Self-healing in the microcraks of 3-day-old ECC specimens.

threshold reported by different authors for the possibility of complete sealing of a crack.[22-24]

Initial aging of specimens caused differences in repeatability of crack sealing performance. After nine repetitive preloadings and cyclic W/D exposure, the cracks in 3-day-old specimens were healed from the same place four to five times, while in the 28-day-old specimens, self-healing of the same cracks was repeated two to three times, regardless of SCM type. This situation was true for cracks with widths of no more than 20 μm (0.0008 in.) and 30 μm (0.0012 in.) in F_ECC and S_ECC specimens, respectively. Moreover, in cases where crack widths were restricted to 20 to 30 μm (0.0008 to 0.0012 in.), new cracks that formed next to healed cracks were observed in most of the reloading situations. Although these new cracks were close to former crack sites, due to the availability of unhydrated cementitious materials in opposite crack faces, they were able to heal rapidly. However, when cracks widths were larger than approximately 100 μm (0.004 in.), cracks followed the same paths that were healed initially. Therefore, even under repetitive preloading, mechanical recovery is possible, although further investigations on related topics are needed for a more precise understanding.

CONCLUSIONS

This paper outlines a detailed study undertaken to gain an understanding of the self-healing phenomenon observed in ECC materials in terms of repeatability and pervasiness. Based on the test results and analysis of self-healed ECC specimens incorporating different SCMs (Class F fly ash and slag), the following conclusions can be drawn:

- Regardless of the point of measurement, for 3-day-old ECC specimens, recovery of RF measurements after the application of preloadings were not eye-catching and were similar to each other after approximately six preloadings and 60 W/D cycles were applied. In the case of 28-day-old ECC specimens, however, stabilization in RF recovery results started after the application of approximately three repetitive preload applications and 30 W/D cycles. Therefore, depending on the type of SCM used and initial curing time, the recovery rate in RF measurements might reach up to 85% of the initial measurement, even after six repetitive preloadings.
- For measurements taken from the middle portions of specimens, RF recovery rates were observed to be lower compared to measurements taken from the surfaces of all speciments, which can be attributed to the difficulty of having moisture diffuse across longer distances. Although RF recovery results dropped for the middle-portion measurements, the number of recovered preload applications stayed the same as the top-portion measurements, with slightly lower recovery rates. These findings show that even under repetitive loading conditions, the self-healing mechanism is widely dispersive over the entire area of the specimens rather than being limited to specific areas. However, the extent of self-healing is highly dependent on easy exposure of cracks to water.
- At the end of five repetitive preloading applications and 50 subsequent W/D cycles, RCPT results for F_ECC specimens decreased up to 16%, while results for S_ECC specimens increased up to 77% of the initial values, despite comparable self-healing occurrence observed in both mixtures. This behavior of S_ECC specimens can be attributed to the formation of larger cracks and faster consumption of unhydrated materials. While the two mixtures behaved in different manners, final RCPT results after five repetitive preloadings were 3440 and 1885 Coulombs for F_ECC and S_ECC mixtures, respectively, which are defined as moderate and low chloride ion penetrability levels in ASTM C1202.
- Self-healing behavior considering the crack closure performance was quite successful in the case of both mixtures, although the extent was more pronounced for

specimens cured for limited periods (3 days). Enhanced self-healing capability regarding with the closure of cracks was also observable by checking maximum crack widths over the specimens after self-healing so that even after the application of severe preloading for nine times followed by 90 W/D cycles, maximum crack widths monitored over the specimens with no respect to the age of specimens were 130 μm (0.006 in.) and 190 μm (0.008 in.) for F_ECC and S_ECC mixtures, respectively.

- When ECC specimens were further aged, there were reductions in self-healing rates in terms of RF and RCPT measurements regardless of SCM type used in the mixtures. This finding might look to be suggestive in that self-healing is not effective when the specimens were of higher maturity. However, it must be emphasized that likelihood for concrete material to be cracked during early ages is higher due to sensitivity against several shrinkage mechanisms, thermal movements and so on. This implies that concrete is much more in need of higher levels of self-healing to take place at the early ages in comparison to later ages. Moreover, preloading was repeated from the same point for nine repetitive times in the present study, which is a rather rare situation to be observed in real-life structures. Overall, these findings suggest that under certain environmental conditions, self-healing could be useful in real-life structures no matter what the age is although more detailed studies are needed on the related topic.

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