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SELF-HEALING OF ENGINEERED CEMENTITIOUS COMPOSITES UNDER CYCLIC WETTING AND DRYING

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ABSTRACT

Self-healing of Engineered Cementitious Composites (ECC) subjected to two different cyclic wetting and drying regimes is investigated in this paper. Uniaxial tension tests are used to generate multiple cracks in ECC specimens deformed to varying tensile strains. To quantify self-healing, resonant frequency measurements were conducted throughout wetting-drying cycles followed by tensile testing of self-healing ECC specimens. It was found that there exists a good relationship between change of resonant frequency and tensile strain deformation in ECC members. Further, through self-healing the resonant frequency of ECC can recover 76% to 100% of initial values while showing a distinct rebound in stiffness of cracked ECC after self-healing. For specimens pre-loaded to high levels of strain between 2% and 3%, the tensile strain after self-healing can recover from 1.8% to 3.1%. Also, the effects of temperature during cyclic regime can lead to an increase in the ultimate strength of the material while slightly decreasing the strain-hardening capacity of ECC due to further hydration of unreacted cement and fly ash.

Keywords: ECC; self-healing; cyclic wetting and drying; resonant frequency, tensile stress; tensile strain

1 INTRODUCTION

Cracks can occur during any stage of the life of a concrete structure. They can be due to the concrete material itself as in the case of volume instabilities, or due to external factors such as extreme loading, harsh environmental exposure, poor construction procedures, or design error. These cracks have many negative effects on the mechanical performance and durability of concrete structures. The development of concretes which can automatically regain this loss of performance is very desirable. Along these lines, self-healing of cracked concrete, commonly called autogenous healing, is an often studied phenomenon. Experimental investigation and practical experience has demonstrated that cracks in cementitious materials have the ability to seal themselves, e.g., water flowing through cracked concrete slows over time. In extreme cases, these cracks can seal completely. The complicated chemical/physical process of self-healing of cracks in concrete has been previously investigated by other researchers. The effects on self-healing by crack width, water pressure, pH of healing water, temperature, water hardness, water chloride concentration, and concrete composition have been discussed by many researchers [1-7]. For autogenous healing to occur, the following reasons have been cited: further hydration of the unreacted cement (C-S-H, CH, AFt), expansion of the concrete in the crack flanks (swelling), crystallization (calcium carbonate), closing of cracks by solid matter in the water (impurities) and closing of the cracks by spalling of loose concrete particles resulting from cracking [1].

Among these reasons, most researchers have indicated that crystallization of calcium carbonate within the crack was the main mechanism for self-healing of mature concrete [1-5]. Typically, a modified version of Poiseuille's Formula is used to describe water flow in concrete cracks. This model, derived from parallel plate flow theory of an incompressible fluid, along with experimental results show that crack width is the dominating factor in engaging the five mechanisms previously discussed which are responsible for self-healing. Therefore, much of this work will focus on the ability of cementitious materials to control crack widths [1-11].

2 MATERIAL DESIGN FOR SELF-HEALING

Engineered Cementitious Composites (ECC) are a unique type of high performance fiber reinforced cementitious composite (HPFRCC), which feature high tensile ductility with moderate fiber volume fraction, typically 2% by volume. Of special interest is the capability of ECC materials to deform to high tensile strains, commonly over 3%, while maintaining very tight cracks widths, shown to be on the order of 60 μm to 80μm on average. This crack width can be seen as an inherent material property, similar to compressive strength or elastic modulus, as it is a feature of the fiber-matrix interaction within the material. This interaction, which is deliberately tailored for strain-hardening performance, is innate within the response of the ECC resulting in tight crack widths while subjected to tensile strains over 3% [12]. As mentioned previously, these tight crack widths are essential for engagement of self-healing abilities.

Investigating water permeability in cracked concretes, Wang et al reported that as crack widths increased from 100μm to 500μm, the permeability coefficient increased nearly seven magnitudes from 1.0×10^{-11} m/sec to 1.0×10^{-4} m/sec [13]. However, for crack widths under 100μm, the permeability coefficient remains nearly identical to that of sound concrete, suggesting that for crack widths below this threshold there is no significant increase in permeability after cracking. Studies by Lepech and Li examined the permeability of ECC subjected to different tensile strains ranging from 0% to 3% [14]. Even as the tensile strain deformation approaches 3%, the permeability of cracked ECC material still remains quite low (7.74×10^{-10} m/sec), due to the inherently tight crack widths in ECC regardless of the level of strain.

With respect to the related theory and experimental results mentioned above, the requirements of self-healing are completely satisfied by ECC material due to the inherently low crack width and permeability. Therefore, the self-healing process with ECC should be easily engaged. This self-healing looks to improve the long term ductility and durability of ECC after cracking, and to establish a much more durable civil engineering material subjected to various environmental conditions.

However, little information is currently available on the long-term performance of ECC in the cracked and uncracked states. While knowledge of the process of self-healing itself is available, specifics with regard to ECC are limited, especially in the case of exposure to various environmental conditions. These conditions can vary greatly and include: the drying action of wind and sun; rain-water containing dissolved sulphurous compounds from industrial pollutants (i.e. acid rain); bridge-deck run-off or road spray contaminated with chlorides from deicing salts; freezing and thawing action; sulfate attack and carbonation. This investigation focuses on the self-healing of pre-loaded ECC materials under cyclic wetting and drying. Wetting and drying was used as an accelerated test method to simulate outdoor environmental conditions in which ECC surfaces are subjected to the drying action of wind and sun and wetting by rain runoff or snowmelt. Experimental data on the extent and rate of self-healing for ECC material pre-loaded to various strain level, along with and the mechanical properties of ECC after self-healing, are presented.

3 EXPERIMENTAL PROGRAM

3.1 Mix Proportions

The mix proportions of ECC material used in this investigation are given in Table 1. To prepare the ECC, Type I ordinary portland cement, sand with 110μm average grain size, Class F normal fly ash supplied by Boral Materials Technologies, 12mm Kuralon-II REC-15 poly-vinly-alcohol fibers supplied by Kuraray Company, and a polycarboxylate-based high range water reducer (Advacast 530) from Degaussa, Inc. were used. A series of coupon specimens were cast from a single batch prepared using a forced-based Hobart mixer. The fresh ECC was then covered with plastic sheets and demolded after 24 hours. The specimens were left to air cure under uncontrolled conditions of humidity and temperature for 6 months.

Table 1 Mix Proportions of ECC

Materials	**Unit weight** (kg/m^3)
Cement	578
Aggregate	462
Fly ash	694
Water	319
HRWR	17
Fiber	26

3.2 Wetting and Drying Cycles

The experimental program consisted of two cyclic wetting and drying regimes. One cyclic regime (CR1) subjected ECC specimens to submersion in water at 20°C for 24 hours and drying in laboratory air at 21±1°C for 24 hours, during which no temperature effects are considered. The second cyclic regime (CR2) consisted of submersion in water at 20°C for 24 hours, oven drying at 55°C for 22 hours, and cooling in laboratory air at 21±1°C for 2 hours. This is used to simulate cyclic outdoor environments such as sunshine and high temperatures.

3.3 Pre-loading of ECC

In previous self-healing research, feedback-controlled splitting tests were used to induce a controlled crack width in concrete, as fully described by Wang et al [13]. Due to this loading geometry, a single crack is typically generated in each concrete specimen along the loading direction. The displacement (i.e. crack width) was monitored on both sides of the specimen by using two linear variable displacement transducers. While this may be appropriate for concrete materials which form a single crack under tension, to examine the self-healing properties of ECC material which develops large numbers of microcracks under tensile strains, splitting tests were not performed. Instead, uniaxial tensioning of ECC specimens was applied as the pre-loading. In this fashion, the true self-healing performance of multiple-cracked ECC subjected to various strain levels could be evaluated.

Direct uniaxial tensile tests were conducted to pre-crack ECC specimens using an MTS 810 material testing system (Figure 1). Specimen dimensions were 230mm x 76mm x 12.5mm. To facilitate specimen gripping during the test, aluminum plates were glued at each end of the coupon specimen. Tests were conducted under displacement control at a loading rate of 0.005mm/s. When the tensile strain reached the required pre-determined test value, the tensile load was released, and the specimens were removed to prepare for cyclic wet-dry exposure. After release of the load, crack widths within the ECC close slightly, by approximately 15% of the crack width, as compared to the loaded state. To account for this, all crack width measurements are conducted in the unloaded state. While self-healing in structures will take place in the loaded state, this unloading has minimal impact on ECC self-healing capabilities as cracks in ECC material subjected to tensile load typically exhibits a similar width to that in the unloaded state, between 60μm and 80μm on average [15].

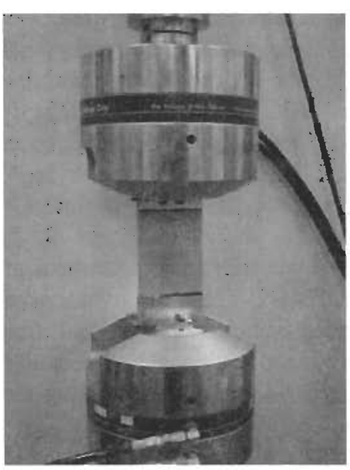


Figure 1: Uniaxial tension testing setup

3.4 Determination of Self-healing of ECC

Permeability testing and acoustic emission technologies are two testing methods which have commonly been used by others to monitor the process of self-healing in cementitious materials [1-5,8,9,11,13-14]. In many studies, a falling head or constant head test is used to examine the extent of self-healing by monitoring the flow rate or quantity of water passing through the cracked specimens. The change in the coefficient of permeability of concrete with respect to time is used to measure the amount of self-healing which has occured. Acoustic emission technology based on ultrasonic pulse velocity (UPV) measurements has also been used to assess crack healing. Although UPV measurement can detect the occurrence of crack healing, it has been shown that this method cannot accurately determine the extent of crack healing[11]. Resonant frequency or dynamic modulus measurements have also been used by a small number of researchers to quantify the self-healing process [8-10]. Recently, one-sided stress wave transmission measurements were used to characterize the process of self-healing, however this transmission measurement is unable to clearly distinguish among crack widths above 100μm [11]. In this investigation, resonant frequency measurements based on ASTM C215 (Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequency of Concrete Specimens) were used to monitor the extent and rate of ECC self-healing after cyclic wetting and drying.

Prior to using resonant frequency as an accurate measure of "healing" within a cracked ECC specimen, it is essential to verify it as a valid measurement of internal damage. Therefore, a series of ECC specimens were subjected to varying levels of strain deformation ranging from 0% to 4%. After unloading, the resonant frequency of each cracked specimen was determined.

From this series of tests, a singular relationship between tensile strain and change in resonant frequency was determined. Further, this relation extends to the number of crack within a specimen versus resonant frequency. These relations are shown in Figures 2 and 3, respectively. The resonant frequency has been normalized by that at zero strain. Due to stable formation of multiple steady-state cracks within ECC, most of which exhibit similar crack widths, there exists a strong bi-linear relationship between the resonant frequency and the tensile strain deformation or number of cracks. Below approximately 1% strain, a sharp drop in resonant frequency can be seen, while above 1% strain this trend softens. The bi-linear relationship may be attributed to the increase in number and crack opening size of the multiple-microcracks at low strain level, while only increase in crack number at steady state crack width has been observed after about 1% strain. These results indicate that a change of resonant frequency can be used to quantify the degree of damage (i.e. tensile strain beyond the first crack) to which an ECC specimen has been subjected. Therefore, this technique should prove useful in quantifying the both the rate and extent of self-healing, or "negative damage", within cracked ECC specimens.

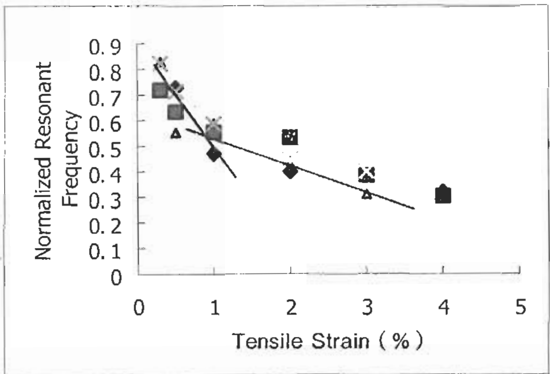


Figure 2: Relationship between normalized resonant frequency and tensile strain

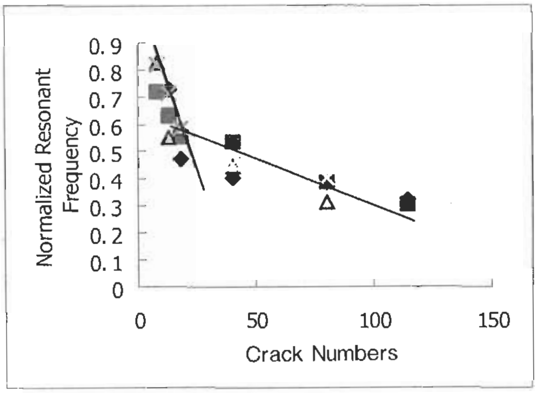


Figure 3: Relationship between normalized resonant frequency and crack numbers

4 RESULTS AND DISCUSSIONS

4.1 Cracks Characteristics

After pre-loading, the specimens were examined using an optical microscope. Table 2 shows the average number of cracks within two pre-loaded specimen series and their corresponding crack widths. The number of cracks on each specimen was determined by averaging the number of cracks intersecting a series of three regularly spaced lines running along the length of the pre-loaded specimen. These results indicate that the number of cracks within pre-loaded ECC ranges from 9 to 80 as the tensile strain increases from 0.3% to 3%, with the first crack occuring at approxmiately 0.1% strain. As expected, a higher tensile strain coincides with more cracks. While, cracked ECC specimens exhibited tightly spaced micro-cracks, there exists a significant variation in crack widths within each specimen, most likely reflecting material inhomogeneity. Crack widths were measured using a Hirox optical microscope with magnification of 100x. The maximum crack width within this study remains below 90μm even as the tensile strain reaches 3%. The maximum, rather than the average crack width, is reported here to highlight the extremely tight crack widths inherent in ECC as compared to concrete. As mentioned previously, these tight crack widths should easily engage the self-healing process.

Table 2: Crack numbers and maximum crack widths of pre-loaded ECC

Tensile strain	**Crack number**	**Maximum crack widths (μm)**
3%	80	83.25
2%	48	46.4
1%	15	67.6
0.5%	12	89.7
0.3%	9	43.15

4.2 Self-healing of ECC

From Fig. 4 and Fig. 5 it can be seen that the resonant frequencies of all pre-loaded ECC specimens gradually recovers under cyclic wetting and drying. Ultimately, the resonant frequencies stabilize after 4 to 5 cycles. These results demonstrate that roughly 4 to 5 wetting-drying cycles are adequate to engage noticeable self-healing of cracked ECC material. Specimens subjected to higher tensile strains exhibit a lower initial frequency after cracking, due to a larger number of cracks, and ultimately lower recovery values after wetting-drying cycles.

Fig. 6 and Fig. 7 show the extent of self-healing within pre-loaded ECC specimens. By calculating the ratio of the final resonant frequency after wet-dry cycles to the initial uncracked resonant frequency, the extent of self-healing can be deduced. Along with the two series of pre-loaded ECC specimens, the change in resonant frequency of six uncracked ECC specimens was monitored under cyclic wetting and drying. The change in resonant frequency for these specimens subjected to wet-dry cycles is shown in Figure 8. The average increase for the six uncracked specimens after 10 wet-dry cycles is 70 Hz. These results are used to remove any unintended increase in resonant frequencies of cracked ECC due to additional hydration during wetting and drying cycles thereby standardizing the results for conditioning.

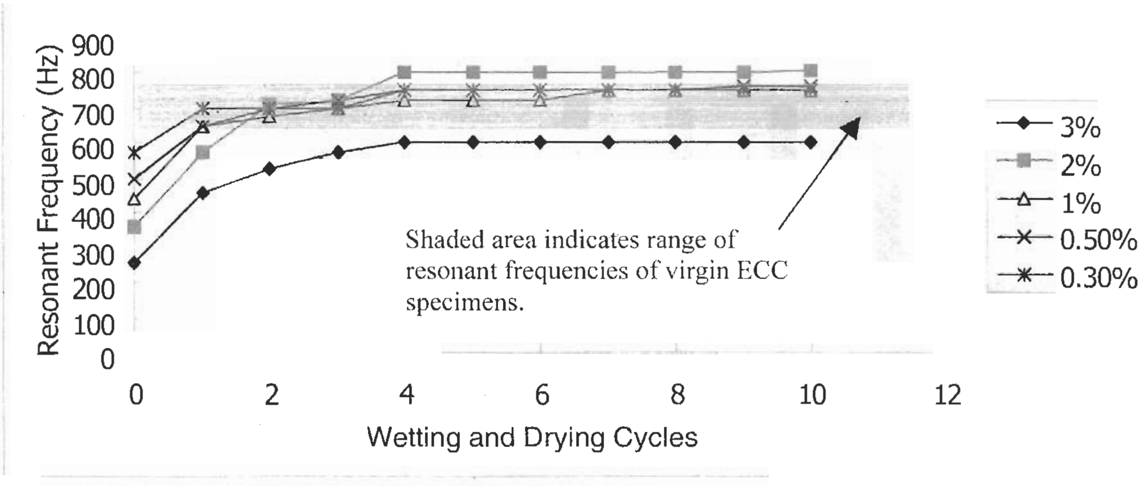


Figure 4: Relationship between resonant frequency and wetting-drying cycles (CR 1)

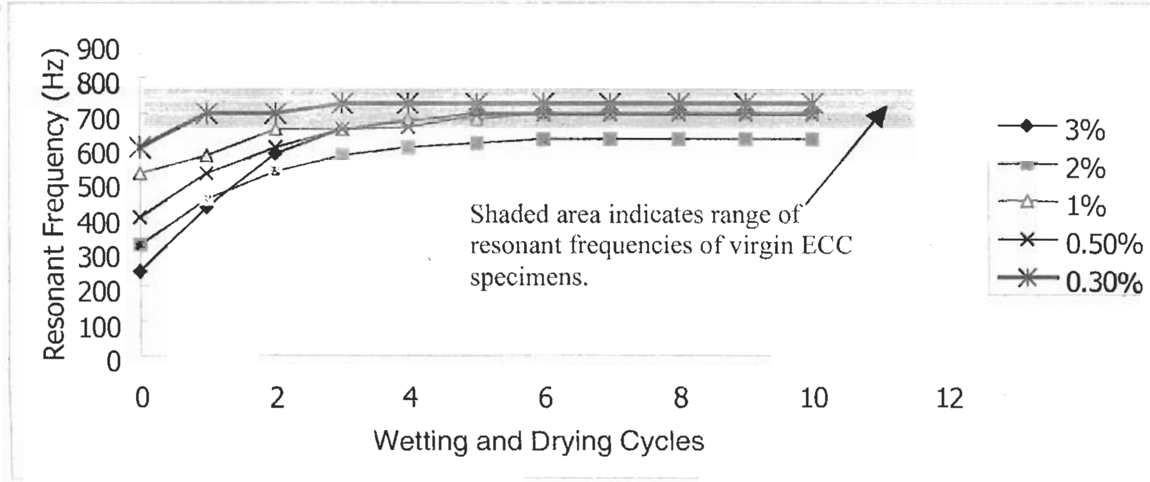


Figure 5: Relationship between resonant frequency and wetting-drying cycles (CR 2)

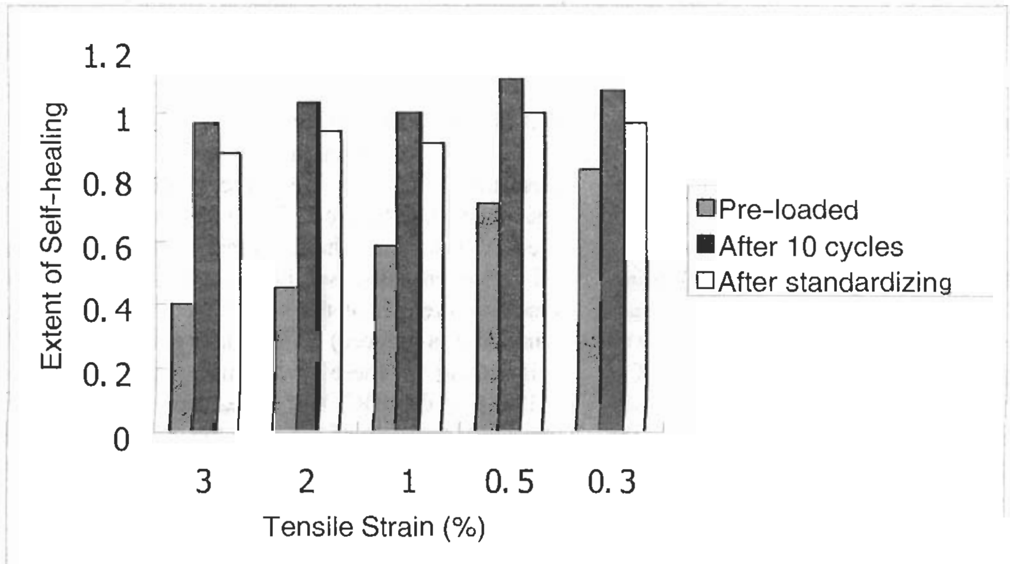


Figure 6: Extent of self-healing of pre-loaded ECC under cyclic regime 1

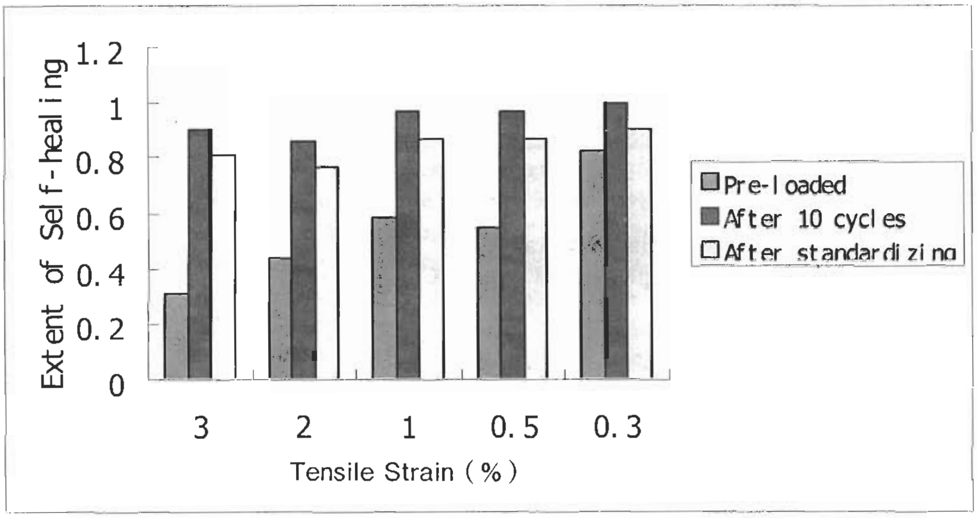


Figure 7: Extent of self-healing of pre-loaded ECC under cyclic regime 2

From Fig. 6 and Fig. 7 it can be seen that the resonant frequencies of most specimens approached or exceeded 100% of their initial resonant frequency when increases due to additional hydration were not considered, the highest of which reaches 110% of the initial frequency. After standardizing the results by removing hydration increases, the resonant frequencies for CR1 tests after pre-loading were 40~82% of initial, while after wet-dry cycles had regained stiffness to 87~100% of initial values. For CR2 specimens, the resonant frequencies after pre-loading were 31~83% of the initial value, and after self-healing had stabilized at 77~90% of initial.

Of particular interest is the relation between the extent of self-healing and level of strain in the pre-loaded ECC specimens under CR1. Pre-loaded testing series with tensile strain of 0.5% exhibited a reduction in resonant frequency of only 18%, while those pre-loaded to 3% strain showed an initial reduction of 60%. Self-healing in 0.5% strained specimens showed rebounded resonant frequencies back to 100% of initial values, while specimens pre-loaded to 3% strain returned to only 87% of initial frequencies. This phenomenon is captured in Figure 9, which highlights the rebound in resonant frequency versus number of cracks within the ECC specimen. As the number of cracks grows, both the rate and amount of self-healing grows. However, the ultimate self-healed condition may not be as complete as in specimens strained to a lower deformation. This is likely due to the presence of a greater number of cracks within the highly strained specimens. Within self-healed ECC specimens, the material which heals the cracks is typically much weaker than the surrounding mortar matrix. With an increasing number of cracks, while the opportunity for a greater amount of healing exists, the likelihood of healing all these cracks to a level similar to the uncracked state drops. Therefore, the accompanying reduction in ultimate self-healing state (i.e. final resonant frequency) with an increase in strain capacity is not altogether surprising.

In addition to this, a noticable difference exists in the extent of self-healing within specimens subjected to CR1 and CR2. This is most evident in Figures 6 and 7. While most specimens subjected to CR1 recovered their full initial stiffness, those subjected to CR2 did not. This may be due to the temperature effects associated with the CR2 conditioning regime. After submersion in water at 20°C, these specimens are then oven dried at 55°C. During this process, moisture escapes from the specimens through evaporation. As the water evaporates, steam pressure builds up within the pores, resulting in internal damage and potential microcracking. This additional damage which takes place during the self-healing process, coupled with the initial damage due to cracking, may handicap CR2 specimens and ultimately result in lower amounts of self-healing when compared to CR1 specimens.

4.3 Mechanical Properties of Self-healed ECC

Figures 10 through 11 show the results of mechanical testing (i.e. direct tension) of ECC specimens both before and after self-healing cycles. For the CR1 test series, the first-cracking strength of nearly all specimens after self-healing falls below the first-cracking strength of the virgin specimens (before any damage was induced) . The tensile strain capacity after self-healing for these specimens ranges from 0.9% to 3.1%. For the CR2 test series, once again the first-cracking strength of all specimens after self-healing remains below the first-cracking strength of virgin specimens. The tensile strain after self-healing for CR2 specimens ranges from 0.8% to 2.2%.

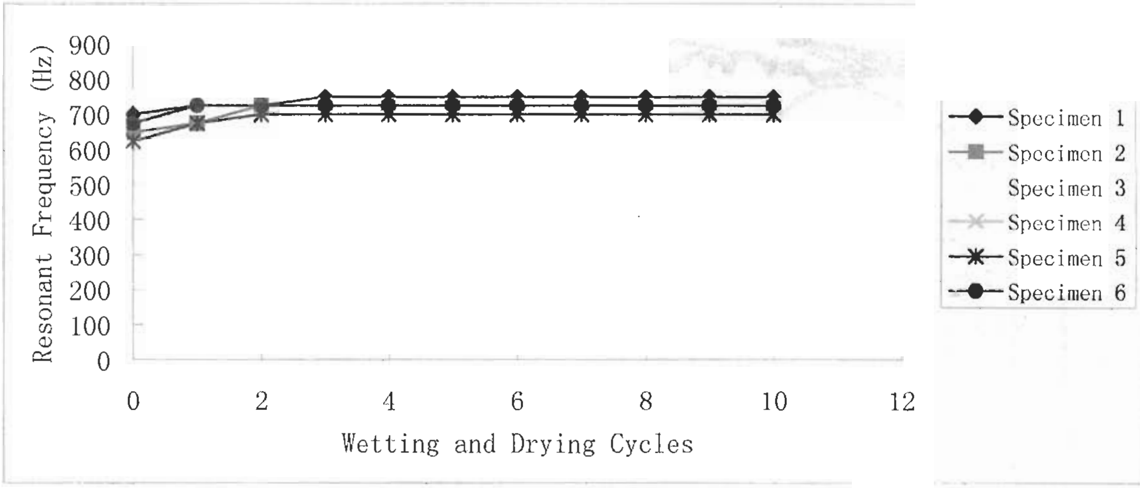


Figure 8: Relationship between resonant frequency and wet-dry cycles for intact ECC

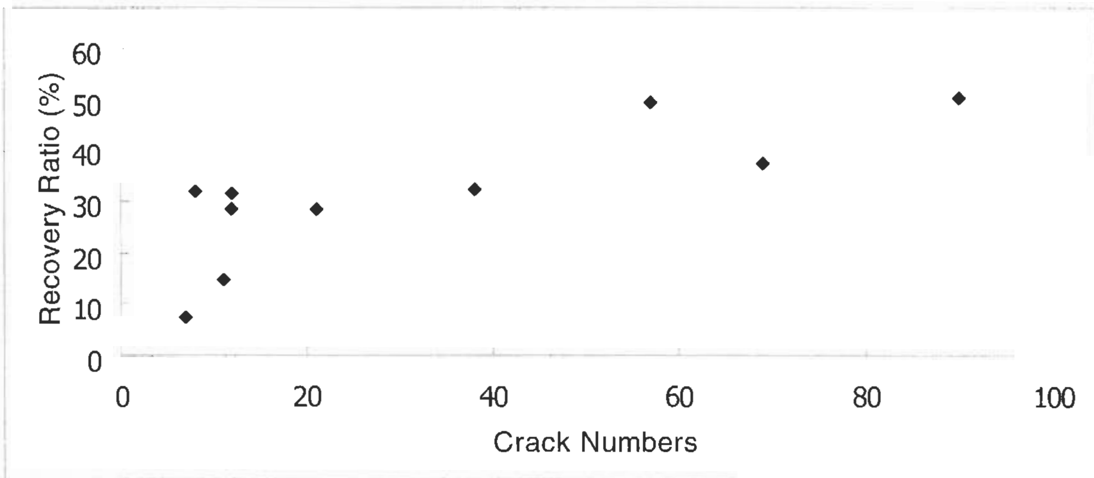
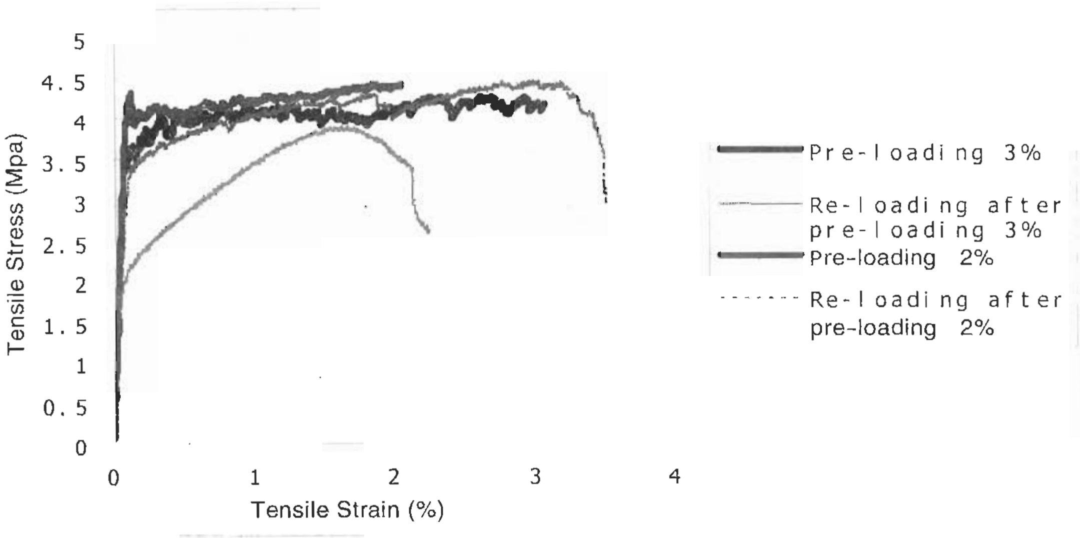
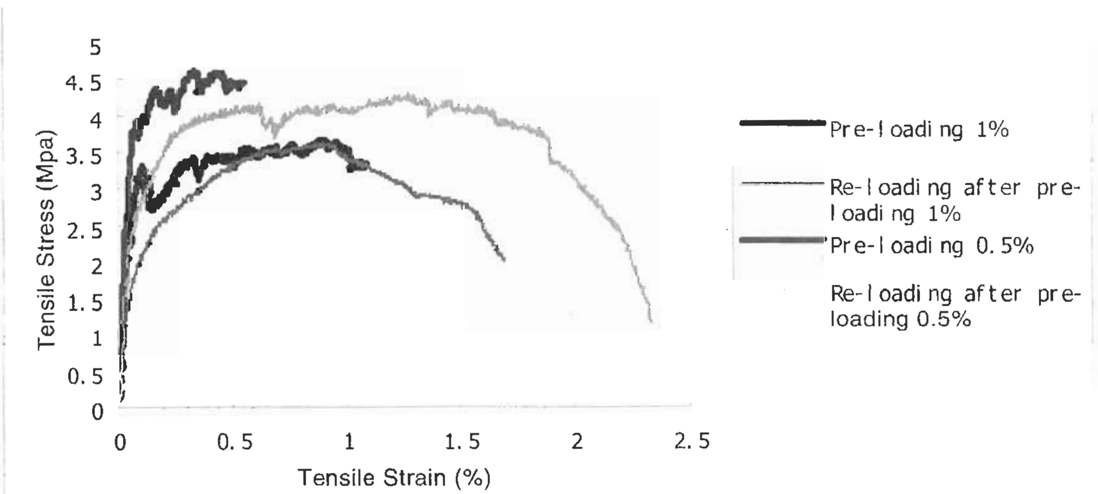


Figure 9: Relation between number of cracks and self-healing recovery ratio

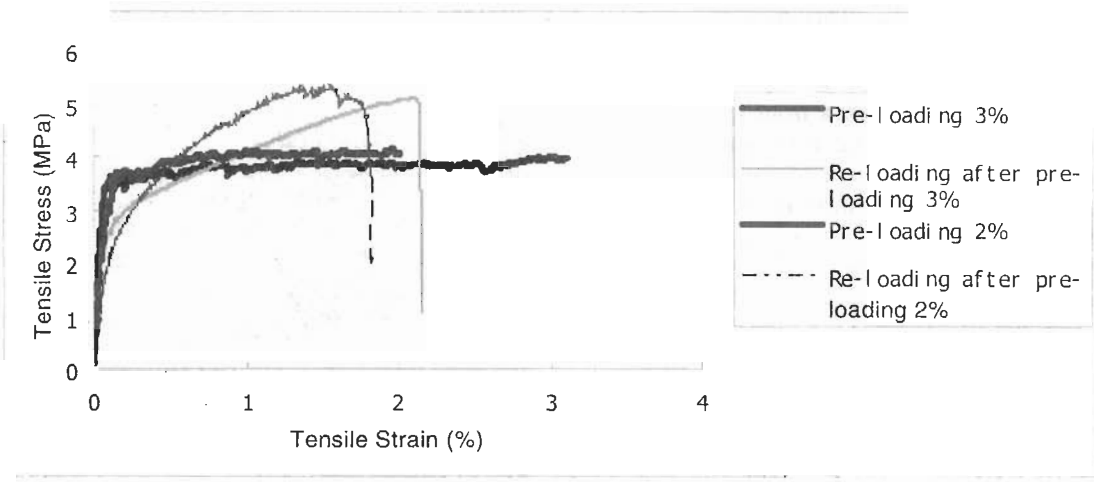


(a) Specimens pre-loaded above 1%

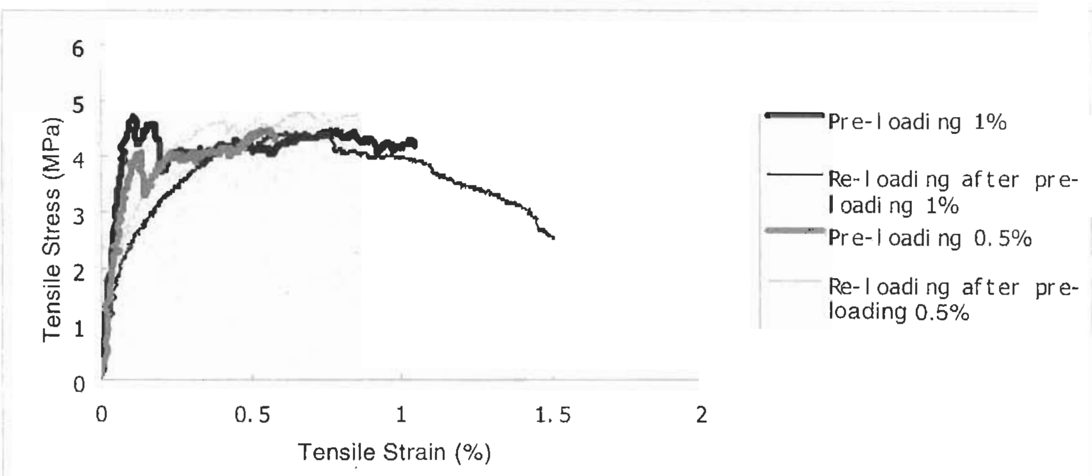


(b) Specimens pre-loaded below 1%

Figure 10: Tensile stress-strain curves of ECC specimens before and after self-healing (CR1)



(a) Specimens pre-loaded above 1%



(b) Specimens pre-loaded below 1%

Figure 11: Tensile stress-strain curves of ECC specimens before and after self-healing (CR2)

However, the ultimate strength after self-healing for these specimens remains higher than that of the pre-loaded specimens, especially for the specimens pre-loaded to 2~3%. The difference of the ultimate strength after self-healing can likely be attributed to the different cyclic conditioning regimes. Recall that specimens subjected to CR2 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 the ECC matrix. This results in the slightly lower self-healed strain capacity of specimens subjected to CR2 as compared to CR1.

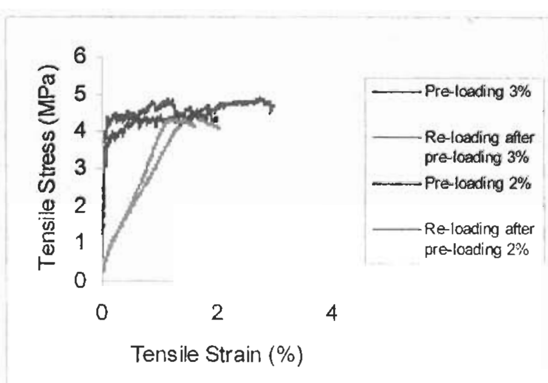


Figure 12: Tensile stress-tensile strain of virgin and pre-loaded ECC specimens without self-healing

As the mortar matrix gains strength, the tendency to rupture fibers crossing newly formed cracks increases, and does not allow the composite to develop saturated microcracks. This type of crack formation is essential to the strain-hardening performance of ECC. Additionally, a greater self-healed strain capacity was expected from specimens preloaded to a low strain level (i.e. 0.3% and 0.5%), as they should have retained much of their original strain-capacity during tensile loading after self-healing cycles. It is possible that the condition of self-healed specimens had some effect on this outcome, but further testing will help to confirm or rule out this experimental trend.

Figure 12 shows the tensile properties of ECC specimens which have been preloaded to 2% or 3% strain levels, then unloaded, and immediately reloaded. Thus these specimens have no opportunity to undergo any self-healing. As expected, there is a remarkable difference in initial stiffness between an virgin specimen and a pre-loaded specimen under tension. This is due to the re-opening of cracks within pre-loaded specimen during reloading. The opening of these cracks offers very little resistance to load, as the crack simply opens to its previous crack width. Once these cracks are completely opened, however, the load capacity resumes, and further tensile straining of the intact material (between adjacent microcracks) can take place. By comparing the initial material stiffness of self-healed specimens in Figures 10 and 11 with that shown for the pre-loaded specimens without self-healing in Figure 12, it can be seen through the initial stiffness of ECC specimens after self-healing that all exhibit a significant recovery of mechanical properties. In other words, self-healing of ECC material can result not only in possible sealing of cracks as shown by others, but in true rehabilitation of tensile properties, in this case the initial stiffness of the material under tensile load.

This finding is also supported by the rebound of resonant frequency seen in the self-healed ECC specimens. As outlined in ASTM C215, resonant frequency is directly related to the dynamic modulus, or stiffness, of a material. The self-healing shown through resonant frequency measurements demonstrates the same self-healing as that shown in the stiffness gain of tensile coupons. This congruent finding can be used to validate the results of both test series, resonant frequency and direct tension testing.

Figure 13 shows an ECC specimen subjected to tensile loading after undergoing self-healing through the CR1 conditioning regime. This specimen was initially subjected to 2% strain before being exposed to wet-dry cycles. The distinctive white residue, characteristic of crystalization of calcium carbonate crystals, is abundant within the crack and near the crack face on the specimen surface. Further, it can be seen that the majority of cracks which form in self-healed specimens tend to follow previous crack lines and propagate through the self-healed material. This is not surprising due to the relatively weak

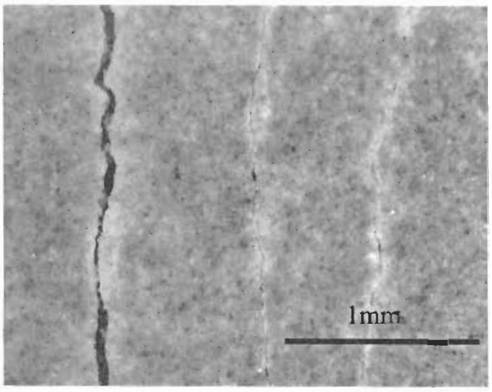


Figure 13: Cracks through self-healed material due to reloading after wet-dry cycles

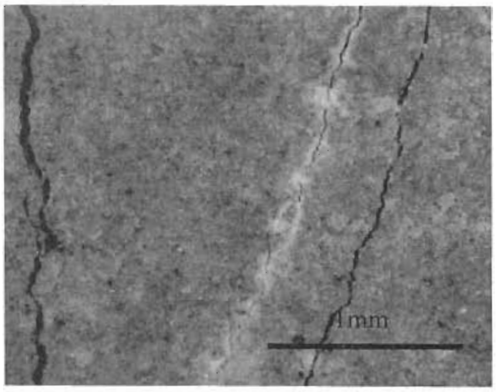


Figure 14: Cracks through virgin ECC material adjacent to a self-healed crack held tight by self-healing material

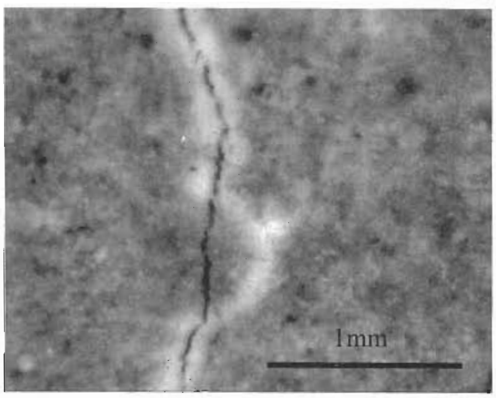


Figure 15: Meandering new crack path through previously self-healed crack

nature of calcium carbonate crystals in comparison to hydrated cementitious matrix.

However, this is not always the case. As can be seen in Figures 14 and 15, new cracks and crack paths have been observed to form adjacent to previously self-healed cracks which now show little or no new cracking. The possibility of this event depends heavily upon the cracking properties of the matrix adjacent to the self-healing, and the quality of the self-healing material itself. However, this phenomenon serves as testament to the real possibilities of mechanical self-healing within ECC material.

5 CONCLUSIONS

From this work, two broad conclusions can be drawn regarding the self-healing of ECC materials subject to wet-dry cycles. Foremost, the deliberate strategy used to enhance self-healing through design of cementitious materials with inherently tight crack widths is effective. In the future, this methodology can be further refined to develop new versions of ECC with tighter crack widths, which should lead to more effective and complete self-healing. Second, the use of resonant frequency has been established as a non-destructive test method to uniquely determine the level of damage (i.e. strain deformation) to which a specimen has been subjected. While the technology was primarily used within this study to quantify self-healing, or "reverse damage", it has promising prospects in future research to quantify the extent of damage within ECC subjected to unknown strain levels.

Additionally, a number of other specific conclusions can be drawn.

(1) The necessary number of wet-dry cycles to fully engage the self-healing process within ECC is about 4 or 5 cycles.

(2) Self-healing in specimens subject to a tensile strain of 3% and 0.3% brought the resonance frequencies back to 76% and 100% of initial values, respectively. This exhibits the relation between the extent of self-healing within cracked ECC specimens, and the level of strain to which they have been subjected.

(3) Effects of temperature may lead to damage and microcracks during the self-healing process, ultimately resulting in lower levels of self-healing within the specimens.

(4) Self-healing can distinctly enhance the stiffness of cracked ECC resulting in true mechanical self-healing of the composite.

(5) Effects of temperature during self-healing can lead to the increase of the ultimate strength and the slight decrease of capacity of strain-hardening of ECC due to further hydration of unreacted cement and fly ash.

(6) For ECC specimens subjected to a high level pre-load strain, roughly 2% or 3%, post-self-healing tensile strain capacity can reach from 1.8% to 3.1%.

A broader range of ECC mix design and a larger database is needed to confirm these tentative conclusions.

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