- Technical Paper -

EFFECT OF WET-DRY CONDITION ON SELF-HEALING PROPERTY OF EARLY-AGE ECC

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ABSTRACT

This paper describes the results of self-healing behavior in early-age Engineered Cementitious Composites (ECC). Uniaxially pre-loaded ECC with tensile strain of 0.3 or 0.5% were subjected to different number of wet-dry cycles. Then, resonant frequency test and reloading test were carried out to assess the recovery rate as a function of the number of wet-dry cycles. These two tests indicated different recovery rates and it was revealed that the number of wet-dry cycles, crack width and dispersion were crucial to attain recovery in mechanical characteristics.

Keywords: ECC, self-healing, wet-dry cycle, early age, resonant frequency

1. INTRODUCTION

With the deterioration of numerous public facilities and infrastructures, maintenance and repair cost is estimated to increase. To achieve life expansion of structures with a limited budget is a socially desirable task.

As a next-generation solution, the self-healing of concrete has been actively studied. Recently, various findings on self-healing in concrete have been revealed. For example, the main mechanism of self-healing was experimentally-indicated to include (a) calcite precipitation and (b) continuous hydration of unhydrated binder [1, 2]. Another finding is that tight crack width is crucial to enable robust self-healing [3]. Yet control of crack width using steel reinforcement has not been reliable in normal reinforced concrete.

ECC, a type of high performance fiber reinforced cementitious composites micromechanically designed, attains self-controlled tight crack width under uniaxial tensile stress. While pseudo-strain increases to several percent with creation of multiple fine cracks, the crack width is limited to about 0.060mm [3]. Thus expectations for self-healing of ECC were raised and some earlier studies have shown that the fine cracks of ECC can be healed throughout wet-dry cycles [3] and it will improve the permeability. In addition, it is also found that resonant frequency which is an evaluation index for dynamic elastic modulus increases of ECC with the number of wet-dry cycles. That is, this crack healing raises expectations of mechanical performance recovery. For these reasons, self-healing of ECC will be beneficial in reducing the time and effort for maintenance and repair.

This research intends to assess the effect of the number of wet-dry cycles on the mechanical property of self-healed ECC. Two test methods: the resonant frequency test and the tensile reloading test are introduced and recovery rates obtained from those tests are compared. Regarding the tensile reloading test, stiffness and strength are focused on.

2. EXPERIMENTAL PROCEDURES

2.1 Specimen casting

The experimental flow is schematically illustrated in Fig. 1. The first step is specimen casting with the mix proportion summarized in Table 1. After casting, specimens were covered with plastic sheets and kept in a laboratory for 24 hours until demolding. Afterward, specimens were air cured for 48 hours.

2.2 Resonant frequency test

Before and after pre-loading tests (i.e. step 2 and step 4 in Fig. 1), resonant frequency tests were carried out in accordance with ASTM C215 as shown in Fig. 2. Resonant frequency was measured three times for one specimen and the averaged value was adopted as the representative value.

2.3 Uniaxial tensile pre-loading

The specimens were subjected to uniaxial tensile pre-loading at age of three days, to induce specific levels of crack damage in the specimens. On the day before pre-loading, alumina plates were glued on the shaded areas in Fig. 3 with epoxy resin. Specimens were gripped over these alumina plates by the loading machine in order not to be crushed by direct gripping. This gripping method enables the tensile force to transfer to specimens perfectly without slip between alumina plates and grips of the loading machine.

Specimens were loaded in displacement

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controlled mode with a speed of 0.025mm/sec. The measurement items were load and elongation of the specimen mid-span. As shown in Fig. 4, two LVDTs were attached to both sides of a specimen for elongation measurement. The averaged measurements by two LVDTs provide the (pseudo) tensile strain of ECC, ε_t , as follows:

$$\varepsilon_t = \frac{\Delta L}{L} \times 100 \quad (\%) \tag{1}$$

where ΔL is the averaged elongation of measured by two LVDTs and L is the original gage length (=100mm). Pre-loading was continued until ε_t reached the designated value, ε_{pre} (=0.3 or 0.5%). These values of ε_{pre} were determined in reference to [3]. It is confirmed that ECC with ε_{pre} =0.3% and 0.5% show great recovery of resonant frequency and crack healing through 10 times of wet-dry cycles [3]. By using the same ε_{pre} as [3] and varying the number of wet-dry cycles as will be explained in 2.4, this study intends to investigate the effect of number of wet-dry cycles on mechanical property recovery in addition to resonant frequency and crack healing. After pre-loading, specimens were unloaded and the residual strain, ε_{rs} , was measured. Numbers and widths of all cracks were recorded after the unloading. Number and width of cracks were observed along with three dot lines shown in Fig. 3 using a microscope with the resolution of 0.010mm.

2.4 Environmental conditioning stage

After crack observation, specimens were subjected to any of the following two cyclic environment conditions: Wet-dry (WD) and lab air (LA). Each cycle is 48 hours in both conditions. Each WD cycle comprises water immersion at 20°C for 24 hours and air drying in laboratory at 21±1°C for 24 hours. Each LA cycle is 48 hours of air drying at 21±1°C in laboratory. In this study, specimens were subjected to 1, 5, 10 or 20 cycles of WD or LA condition. Resonant frequency was measured after each environmental conditioning cycle was completed.

2.5 Uniaxial tensile reloading

Reloading tests were conducted until test length of specimen attained the same measurement as pre-loading tests. Here, by taking into account residual strain caused by pre-loading, ε_{re} , imposed strain on reloading, is expressed in the following formula:

$$\varepsilon_{re} = \varepsilon_{pre} - \varepsilon_{rs} \tag{2}$$

2.6 Specimen series

As stated above, experimental parameters were the magnitudes of ε_{pre} (0.3 or 0.5%), environmental conditioning regimes (WD or LA) and cycle number of

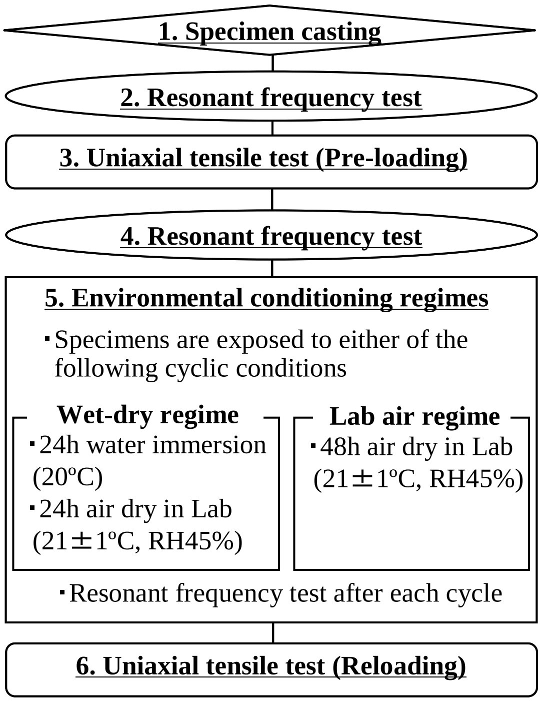


Fig. 1 Experiment flow chart

Table 1 Mix proportion for ECC

W/C	*W/B*	*W*	*C*	*S*	*FA*	*Ad*	*F*
(%)		(kg/m^3)					(Vol. %)
57.1	26.0	328	574	459	689	7	2.0

Note:
W: Water, *C*: Ordinary portland cement,
S: Silica sand, average grain size=0.110mm,
FA: Fly ash (Class F),
Ad: High-range water reducer (Polycarboxylate-based),
F: PVA fiber, length=12mm, nominal tensile strength=1600N/mm^2.

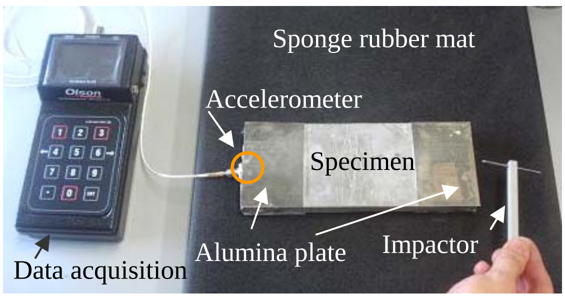


Fig. 2 Resonant frequency test

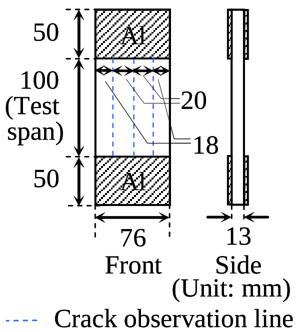


Fig. 3 Specimen shape

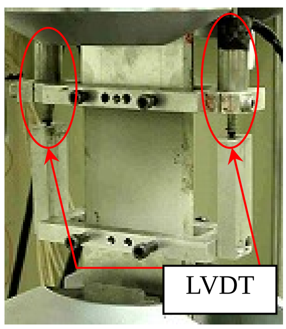


Fig. 4 Uniaxial tensile test

Table 2 Experiment series

Name	ε_{pre}	Environmental condition		No.
		Type	Cycle number	
0.5-WD-20	0.5%	Wet-dry	20	2
0.5-WD-10			10	2
0.5-LA-20		Lab. air	20	1
0.5-LA-10			10	1
0.3-WD-20	0.3%	Wet-dry	20	2
0.3-WD-10			10	2
0.3-LA-20		Lab. air	20	1
0.3-LA-10			10	1
0.3-WD-5		Wet-dry	5	1
0.3-WD-1			1	1
0-WD-20	0%	Wet-dry	20	1
UTST	-	-	-	3

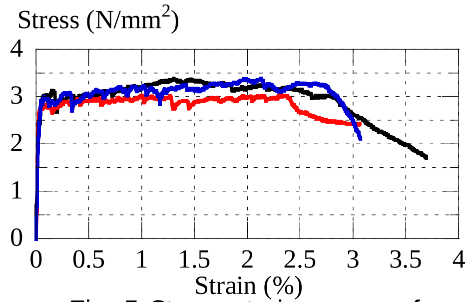


Fig. 5 Stress-strain curves of 3-day age ECC

Table 3 Crack properties of ECC pre-loaded at 3-day age

Imposed strain (%)		0.3	0.5
Crack number		4	6
Crack width (mm)	Max.	0.030	0.050
	Average	0.012	0.016

environmental conditioning1, 5, 10 or 20). A total of 17 specimens with combinations of these three parameters are listed in Table 2. The rightmost column of the table gives the number of specimens for each experimental series. 0-WD-20 was a control specimen for confirmation of resonant frequency of virgin specimen (i.e. specimen without pre-loading). UTST series were loaded up to failure to check the uniaxial tensile behavior of ECC at 3-day age.

3. TEST RESULTS

3.1 Material property of ECC at 3-day age

Figure 5 shows the results of uniaxial tensile tests. The ductile behavior was observed and the maximum uniaxial tensile strain was 2.6% in average. Crack number and width of ECC pre-loaded up to 0.3% or 0.5% are presented in Table 3. As seen in this table, fine cracks unlike normal concrete were achieved even in specimens with $\varepsilon_{pre} = 0.5\%$.

3.2 Resonant frequency test

Figure 6 shows the change of resonant frequency with the increase in the number of environmental conditioning cycles. Some data present the average of two specimens. This figure shows sharp drops of the resonant frequency due to pre-loading damage in all specimens. The reduction in resonant frequency is significant in specimens with $\varepsilon_{pre} = 0.5\%$.

After the sharp drop, resonant frequency shows different changing tendency according to the environment conditioning regime. The resonant frequency of specimens exposed to LA was nearly constant through all cycles. Meanwhile, the resonant frequency drastically recovered through only one WD cycle. Subsequently, the resonant frequency recovered gradually with additional conditioning cycles. This recovery tendency of resonant frequency does not depend on magnitude of ε_{pre}.

Here, a new index "Normalized RF" is introduced for evaluating quantitatively this recovery rate of resonant frequency. This index represents the ratio of resonant frequency between the target and the virgin specimens experienced the same number of WD cycles. This is calculated from Eq. (3) as:

$$Normalized\ \ RF(\varepsilon_{pre}, N) = \frac{RF(\varepsilon_{pre}, N)}{RF(0, N)} \times 100 \quad (\%) \quad (3)$$

where $RF(\varepsilon_{pre}, N)$ and $Normalized\ \ RF(\varepsilon_{pre}, N)$ represent the resonant frequency and Normalized RF of the considered specimen with ε_{pre} at the completion of N times of WD cycles. This normalization removes the effect of wet-dry cycles on the bulk material (e.g. continued hydration and/or moisture content) that may affect the resonant frequency measurements. In this manner, changes in the normalized RF can be attributed to self-healing of the microcrack damage.

Figure 7 shows the measured normalized RF. The normalized RF immediately after unloading without conditioning is decreased to 72% in specimens with $\varepsilon_{pre} = 0.3\%$ and 63% in specimens with $\varepsilon_{pre} = 0.5\%$. After the first WD cycle, however, the normalized RF rapidly increased up to 95% in both specimens. Subsequently, the normalized RF approaches 100% as the number of WD cycles increases.

3.3 Decrease of crack width with WD cycles

Decrease of crack width with WD cycles is shown in Fig. 8. A Y-shaped crack with width of 0.020mm is seen in the left-hand picture taken shortly after pre-loading test. After one WD cycle, the crack is filled in with white self-healing products inferable as calcite. The precipitation of the self-healing products is more distinguished at the end of 20 WD cycles. This suggests that resonant frequency recovery is ascribable to crack healing with self-healing products.

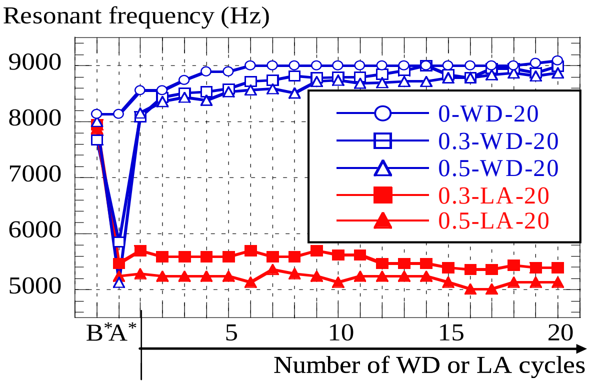


Fig. 6 Resonant frequency change with WD cycles (B*- Before pre-loading, A*- After pre-loading)

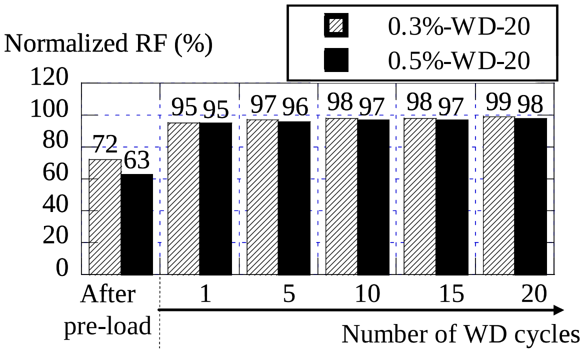


Fig. 7 Normalized resonant frequency

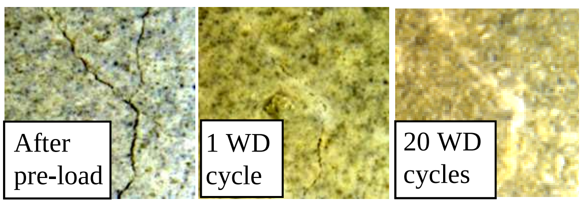


Fig. 8 Decrease of crack width with WD cycles (Crack width=0.020mm)

3.4 Uniaxial tensile test

Stress-strain curves obtained from pre-loading and reloading tests are shown in Fig. 9. Regarding experiment series which have two specimens, the stress-strain curve of either specimen is illustrated because similar shapes were obtained. There can be seen some features in reloading curves by each experimental parameter: environmental conditioning regime, cycle number of environmental conditioning and magnitude of ε_{pre} .

(1) Effect of environmental conditioning regime on reloading behavior

From Fig. 9, it is found that different environmental conditioning regimes lead to clear differences in reloading curves. Compared with the pre-loading curves, an obvious decrease in initial stiffness is observed in all LA-conditioned specimens. This is irrespective of the magnitude of ε_{pre} and the number of LA cycles. Likewise, the stress magnitude when the strain attained the maximum on reloading is also smaller than that on pre-loading. These can be thought to result from crack opening, which stayed

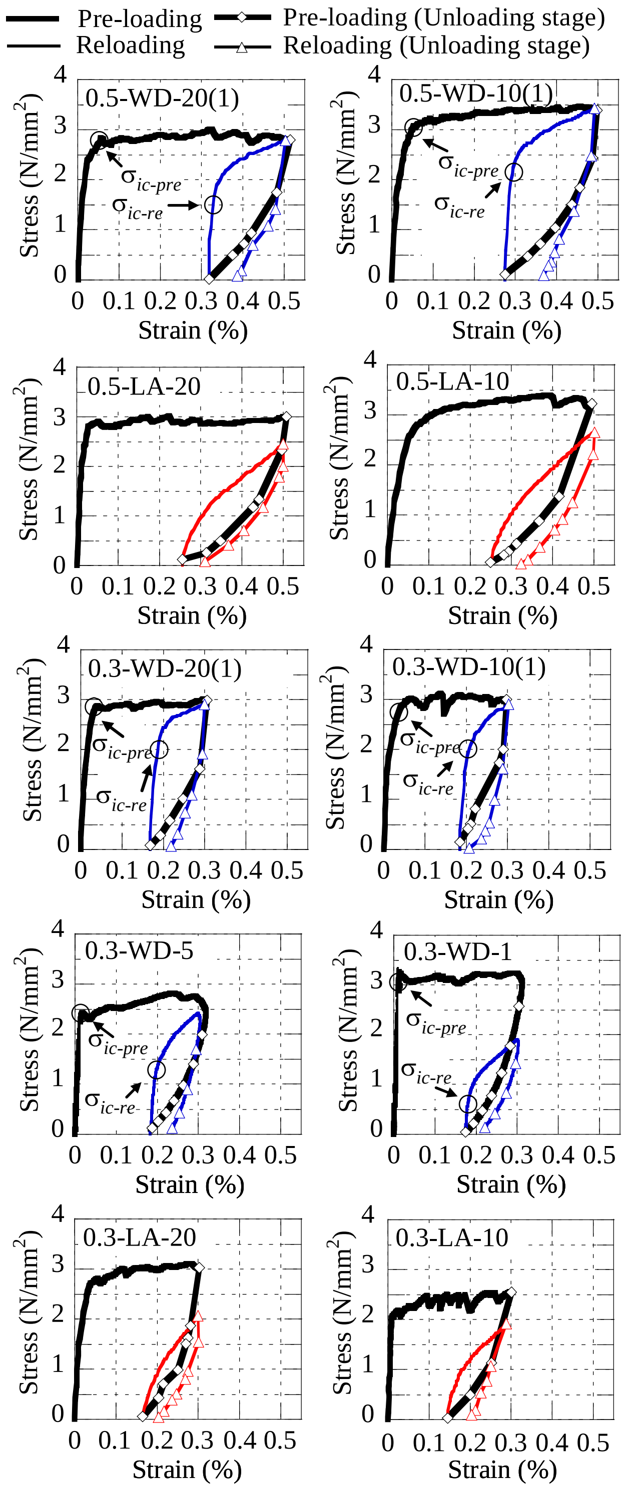
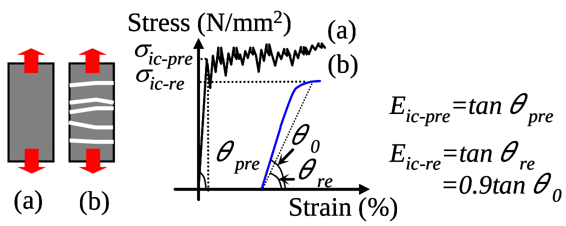


Fig. 9 Stress-strain curves



(a) Pre-loading (3-day age)
(b) Reloading (3-day age + N times of WD cycles)

Fig. 10 Concept of $\sigma_{ic\text{-}pre}$, $\sigma_{ic\text{-}re}$, $E_{ic\text{-}pre}$, and $E_{ic\text{-}re}$

Table 4 Initial cracking stress and initial stiffness on pre-loading and reloading

Specimen	σ_{ic-pre} (N/mm^2)	σ_{ic-re} (N/mm^2)	σ_{ic-re} / σ_{ic-pre}^* (%)	E_{ic-pre} (kN/mm^2)	E_{ic-re} (kN/mm^2)	E_{ic-re} /E_{ic-pre}^* (%)
0.3-WD-1	3.0	0.6	21.4	7.6	9.3	149.5
0.3-WD-5	2.4	1.3	46.4	7.0	9.7	155.9
0.3-WD-10(1)	2.8	2.0	71.4	5.5	10.7	172.0
0.3-WD-10(2)	2.6	2.3	82.1	7.6	11.4	181.7
0.3-WD-20(1)	2.9	2.0	71.4	6.4	7.6	122.2
0.3-WD-20(2)	2.6	2.0	71.4	6.3	8.3	133.4
0.5-WD-10(1)	3.1	2.2	78.6	4.8	9.4	151.1
0.5-WD-10(2)	2.6	2.0	71.4	4.0	8.9	143.1
0.5-WD-20(1)	2.8	1.5	53.6	5.1	6.4	102.9
0.5-WD-20(2)	2.8	1.5	53.6	7.9	6.2	99.7
Average	2.8	-	-	6.2	-	-

Note: σ_{ic-pre}^* =average value of σ_{ic-pre}(=2.8N/mm^2), E_{ic-pre}^* =average value of E_{ic-pre}(=6.2kN/mm^2)

close during unloaded state.

In contrast, reloading curves of specimens exposed to WD cycles showed entirely-different characteristics. Decrease in initial stiffness like LA-conditioned specimens was not observed. Initial stiffness, instead, seems equivalent or higher than that on pre-loading. Another point to note here is that reloading curves bend over when stress attains a certain value. These two characteristics, that is, initial stiffness and stress at bending point on reloading, are used for evaluation of mechanical characteristics of self-healed ECC through WD cycles.

Hereinafter, initial stiffness and stress at bending point on reloading curve will be denoted as E_{ic-re} and σ_{ic-re}, respectively. Here, the bending point is determined to satisfy the condition that the slope of the line connecting the bending point and the starting point of reloading curve is 90% of initial stiffness on reloading. E_{ic-re} and σ_{ic-re} are weighed against E_{ic-pre} and σ_{ic-pre}, which represent initial stiffness and the stress at the first convex point on pre-loading curve. The concept and derivation method of these four evaluation items will be explained below.

First, the cause for bending of reloading curves is considered. Microscopic observations clarified that cracks created by the reloading test were observed only in the same location with pre-loading test in any specimen. In other words, cracks induced by pre-loading test were vulnerable parts and reopened during reloading test. This indicates that the bending point of reloading curve corresponds to the first cracking point of a specimen which includes self-healed cracks. Hence, σ_{ic-pre} and σ_{ic-re} are contrasted in order to compare the initial cracking stress on pre-loading and reloading.

Next, E_{ic-pre} and E_{ic-re} were defined respectively as a slope of a segment which connects the origin and σ_{ic-pre} or σ_{ic-re}. E_{ic-pre} and E_{ic-re} reflect the average stiffness of the specimen from beginning of loading to the initial cracking point. The conceptual diagram of these four characteristics is shown in Fig. 10. Table 4 summarizes the values of σ_{ic-pre}, σ_{ic-re}, E_{ic-pre}, and E_{ic-re}. Here, (1) or (2) is attached to some specimen names in the experiment series with two specimens.

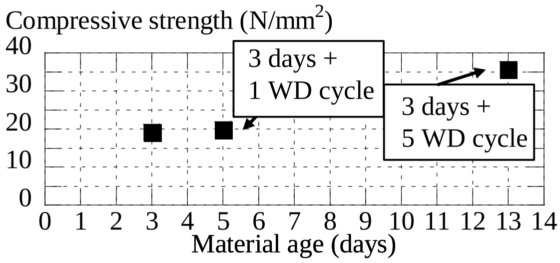


Fig. 11 Compressive strength change with material age

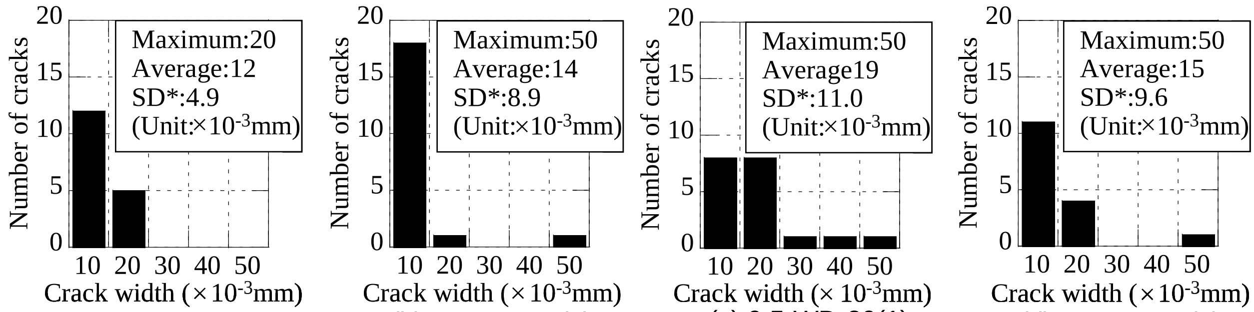
(2) Effect of the number of WD cycles on reloading behavior

Specimens with $\varepsilon_{pre} = 0.3\%$ are focused on in this section. In Table 4, the ratio σ_{ic-re}/ σ_{ic-pre}^* is nearly constant and runs from 71.4 to 82.1% for specimens that underwent 10 and 20 WD cycles. The ratio, however, shows a remarkable decline as the number of WD cycles decreases. This result is as opposed to the resonant frequency which presented noteworthy recovery just after one WD cycle. The cause of this discrepancy will be discussed below.

The resonant frequency reflects the density and the stiffness of targets. That is to say, the reasons for resonant frequency recovery in cracked specimens will be classified into the followings: (a) crack filled by self-healing products, and (b) densification of uncracked area with progress of hydration.

Which of these is the main contributor of resonant frequency recovery will be discussed. Figure 11 shows that the compressive strength of ECC changes with the number of WD cycles. No significant enhancement of the compressive strength is seen from 3-day age to 5-day age (3 days + 1WD cycle). For this reason, the densification effect of uncracked area on the resonant frequency is thought to be negligible and that crack healing can be the main factor in the resonant frequency recovery. When taken together, adequate amount of self-healing products to give larger resonant frequency can be attained with only one WD cycle.

On the other hand, given the fact that σ_{ic-re} of 0.3-WD-1 and 0.3-WD-5 is much smaller than that of 0.3-WD-10 and 0.3-WD-20, the mechanical performance of self-healed areas can be said to be affected by number of WD cycles. That is to say, it is



(a) 0.5-WD-10(1) (b) 0.5-WD-10(2) (c) 0.5-WD-20(1) (d) 0.5-WD-20(2)

Fig. 12 Crack width distribution in specimens with $\varepsilon_{pre} = 0.5\%$ (*SD- Sample standard deviation)

indicated that (a) the boundary of self-healing products grown from opposite crack surfaces, (b) the interface between self-healing products and crack flank surfaces or (c) both, may be strengthen with the number of WD cycles.

In all specimens with $\varepsilon_{pre} = 0.3\%$, the value of $E_{ic\text{-}re}$ exceeds $E_{ic\text{-}pre}$. For this reason, self-healed cracks in 0.3% series are imperceptible when focusing on the entire behavior of a specimen. And enhancement of strength due to continuous hydration in uncracked part is also thought to contribute to this stiffness increase. $E_{ic\text{-}re}$ in 0.3%-WD-20 series is, however, lower compared to that in 0.3%-WD-10 series. This is just as valid for 0.5% series. In other words, this indicates that the effect of WD cycles on specimens changes depending on cyclic number.

(3) Effect of ε_{pre} on reloading behavior

Specimens with $\varepsilon_{pre} = 0.5\%$ are taken into account. The $\sigma_{ic\text{-}re}$ of 0.5-WD-20 series is clearly smaller than that of 0.5-WD-10 series. Furthermore, $E_{ic\text{-}re}$ of 0.5-WD-20(2) fell a little below $E_{ic\text{-}pre}$. The causes of these mechanical characteristics degradations could be a result from larger crack width.

The crack width distribution in 0.5% specimen series is shown in Fig. 12. In this figure, all cracks observed along three lines shown in Fig. 3 are presented. Maximum crack width, average crack width, and sample standard deviation of crack width in each specimen are presented in conjunction with the crack distribution. According to Fig. 12(c), 0.5-WD-20(1) has some large cracks with width of 0.030 to 0.050mm. There is a possibility that these cracks behaved as local defects. Consequently, $\sigma_{ic\text{-}re}$ and E_{ic} resulted in smaller values.

In addition, by comparison of Fig. 12(b) and (d), there are greater amount of cracks with more than 0.010mm width in 0.5-WD-20(2) than in 0.5-WD-10(1). Yet maximum value, average value and sample variance of crack width are nearly equivalent between these specimens.

Based on the discussions above, smaller crack width and high crack dispersion can contribute to recovery of $\sigma_{ic\text{-}re}$ and $E_{ic\text{-}re}$. Given that crack width itself and crack width variation will be larger as ε_{pre} increases, $\sigma_{ic\text{-}re}$ and $E_{ic\text{-}re}$ will have higher variation from specimen to specimen when larger deformation is imposed.

(4) Comparison between the two test methods

The results from resonant frequency test and reloading test can be summarized as below. Resonant frequency test and reloading test showed different recovery rates. While resonant frequency test can be an effective method to evaluate the crack filling level, it cannot detect recovery in mechanical properties like initial cracking stress and initial stiffness. It will be necessary to select a proper test method depending on desired evaluation items.

4. CONCLUSIONS

(1) Resonant frequency test and reloading test were carried out to assess the self-healing rate of early age ECC under wet-dry cycles. While resonant frequency test showed a great sensitivity to the presence of self-healing products, it is inadequate as a measure of mechanical property recovery.

(2) Resonant frequency showed a drastic recovery after just one wet-dry cycle. This corresponds to the precipitation of self-healing products in cracks.

(3) Initial cracking stress on reloading recovered with increase in wet-dry cycles.

(4) The initial cracking stress and average stiffness up to initial cracking on reloading cannot be expected to recover when the imposed strain is large. This can be due to the presence of larger crack width.

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