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Selecting an adequate procedure for the electrochemical chloride removal

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Abstract

This paper concerns the proper selection of the electrochemical chloride removal (ECR) current and the ECR duration. Four constant ECR current densities, 100, 200, 400 and 800 $\mu A/cm^2$, were applied to concretes for 1 to 6 weeks. The remaining chloride contents nearby the reinforcement were measured to understand how fast the ECR process can remove chloride ions out of concrete. The open circuit potential and corrosion rate measurements were performed to examine the corrosion status after ECR. In addition, the pH value nearby the reinforcement and the apparent electrical resistivity of concretes after ECR were measured. The results showed that the remaining chloride ions decreased, the open circuit potential became nobler, the corrosion rate decreased, the pH value increased and the apparent electrical resistivity increased as the ECR parameter, defined as the product of the ECR current density and the ECR duration, increased. The ECR parameter can provide a direction in designing the ECR process.

Keywords: Eletrochemical chloride removal; Chloride ions; pH (A); Open circuit potential; Resistivity

1. Introduction

It is well known that the chloride ion can result in a catastrophic failure of reinforced concrete structures [1]. The use of deicing salts during winter and intentional addition of chloride-based admixtures to concrete lead to substantial amounts of this aggressive species. Other sources include the use of chloride contaminated aggregates or mixing water, and the exposure to industrial brine and seawater. Chloride ions can destroy the passive film of reinforcements and, consequently, result in corrosion damage in the reinforced concrete structure. It is well known that, after reaching a threshold concentration, the chloride ions result in collapse of the passive film. Ethesham and Al-Sandoun [2] expressed the chloride threshold by the following equation: $[Cl^-]/[OH^-] \geq 0.6$. Nowadays, it is believed that there is no such exact thing as "chloride threshold" and there is no collapse of the passive film in chloride attack. Corrosion induced by chloride occurs locally (pitting) and is driven by large relatively cathodic areas often with macrocells. There is no clear "chloride threshold" for corrosion. That depends upon voids (where corrosion is initiated) and the chemical suppressing effect of alkaline solids—not just pH. In brief, electrochemical treatment raises the chloride threshold. Hence, the issue is not simply chloride removal but the creation and sustainability of passivation. That is worthy of further research. It then has no doubt that the chloride removal becomes a very important issue nowadays.

Since the chloride removal method was investigated [3,4] (though the earlier investigation had drawbacks, not the least the severe cracking of concrete caused by excessive current density realised at high voltage), the application of an electrical filed to concrete as a nondestructive repair method has been increasingly conducted [5,6]. Assessments and guidelines for treatment were issued following the Strategic Highways Research Program (SHRP in the USA)—such as SHRP-S-347 and SHRP-C-620. These can be downloaded from the web. In addition, the processes were the subject of several patents which formed the basis of the "Norcure" processes that have been fairly widely known and used in USA, Europe and Japan. The removal of chloride ions was named as ECR or electrochemical chloride extraction (ECE) in literatures. The idea of ECR involves mounting an anode surrounded by a liquid electrolyte (usually NaOH or Na_3BO_3) on the surface of concrete and

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driving direct current into the embedded reinforcement, which acts as a cathode. (The usual electrolyte is calcium hydroxide provided as a saturated solution by mixing cellulose pulp with solid hydrated lime. This captures some fugitive chlorine and also prevents acid etching of the concrete surface.) The current pushes chloride ions away from the reinforcement and extracts them towards the anode. Once reaching the concrete surface, the ions eventually pass into the anolyte and are thereby removed from concrete. The chemical reactions that occurred at the cathode are expressed as:

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^- \tag{1}$$

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \tag{2}$$

Anodic reactions are expressed as:

$$4OH^- \rightarrow 2H_2O + O_2 + 4e^-$$

$$2H_2O \rightarrow 4H^+ + O_2 + 4e^-$$

$$2Cl^- \rightarrow Cl_2 + 2e^-.$$

Ihekwaba et al. [7] reported that ECR was considerably retarded by the presence of a carbonation front. Ihekwaba et al. [8] also reported that the ECR current reduced the concrete compressive strength, especially for the concrete near the cathode. By investigating the rehabilitation of several vertical structures, Ihekwaba et al. [9] concluded that circular columns containing spiral reinforcements showed better ECR performance than structures with planar surfaces. They [10] also reported that a pullout bond degradation of steel rebars in ECR concrete with a maximum decrease of 44% bond degradation was found. A recent report from the author [11] claimed that a serious bond degradation was found for seriously contaminated concrete even when one only wanted to push the chloride ions away from rebar instead of extracting them out. Marcotte et al. [12] found that calcium–silicon-rich phases existing in untreated concrete disappeared after ECR. Instead, sodium-rich, calcium-rich, iron-rich and calcium–aluminum-rich phases were observed. They [13] also reported that, although ECR halted chloride-induced pitting corrosion, it increased the overall corrosion rate for all specimens studied because of the passive film reduction and significant changes in the pore solution and cement chemistry adjacent to the reinforcements. Glass and Buenfeld [14] have reported that electrochemical process can inhibit corrosion, which is controversial with [13]. Ayra et al. [15] investigated several parameters that influenced electrochemical removal including the applied potential gradient, reinforcement arrangement, initial chloride content, depth of chloride contamination with respect to the cover reinforcement, use of an external cathode and soffit ECR, i.e., chloride removal from the source of contamination. Andrade et al. [16] proposed a mathematical model to describe an ECR process in which the Nernst-Plank equation was adopted. Hassanein et al. [17] reported a mathematical model that incorporated a term describing the rate of release of bound chloride. Sa'id-Shawqi et al. [18] later used a numerical method to predict the electrochemical removal based on Nernst-Plank and Laplace equations. Castellote et al. [19] have used a simplified mathematical model to investigate the ECR efficiency. Polder [20] applied the ECR of an electrochemical chloride removal technique for 39 days to treat concrete with a 16-years submersion in the North Sea. It was reported that 70% to 90% of the chloride content was removed. Chess et al. [21] suggested that a 1–10 A/m^2 current density is suitable for ECR. A high current density such as 10 A/m^2 may not be adequate for concrete since it may result in cracking.

ECR, which uses an electrochemical process similar to that of the cathodic protection, employs a higher current density in order to complete its goal in a short period. However, the electrical field may reduce the bond characteristics and the compressive strength since it changes the chemistry and the nature of the steel/paste interface. Therefore, how to select a suitable ECR process including the ECR period and ECR current density becomes nontrivial. In previous studies [22,23], we have used a parameter to understand the proper selection of cathodic protection. The success in these studies stimulates the current research. It is our goal to propose a guide for engineers to select a proper ECR process. Another goal of this study is to re-examine whether or not an ECR process induces an overall corrosion as mentioned in reference [13]. It is well known that the realkalization process, which basically is applied to heal the damaged passive film of reinforcement due to the carbonation effect, uses the cathodic current that is the same as the ECR process. It then becomes puzzling why an ECR process will result in an overall corrosion when the realkalization will not. The reason of controversial claims will be clarified in this study.

2. Experimental scheme

2.1. Materials and mixes

The type I cement, fine aggregates with a fineness modulus of 3.01 and coarse aggregates from local sources were used. The #3 deformed rebar used has the Young's modulus of 203 GPa (29,435 ksi) and the yield strength of 410 MPa (59.5 ksi). The electrolyte used was a 0.1 M NaOH solution. A titanium mesh having an electrical resistivity of 5.6×10^{-5} Ω cm was used as the anode. Mix designs for concretes are shown in Table 1, in which two water/cement ratios (0.4 and 0.66) are used. For contaminated specimens, a 3% NaCl dosage was added by weight of cement.

2.2. Design of ECR parameters

In the ECR process, two factors should be considered: the ECR current density and ECR duration. In applying an electrical

Table 1
Mix proportions of concrete (kg/m^3)

W/C	Water	Cement	NaCl	Fine aggregate	Coarse aggregate
0.66	195	295	8.85	719	1161
0.4	195	488	14.64	554	1161

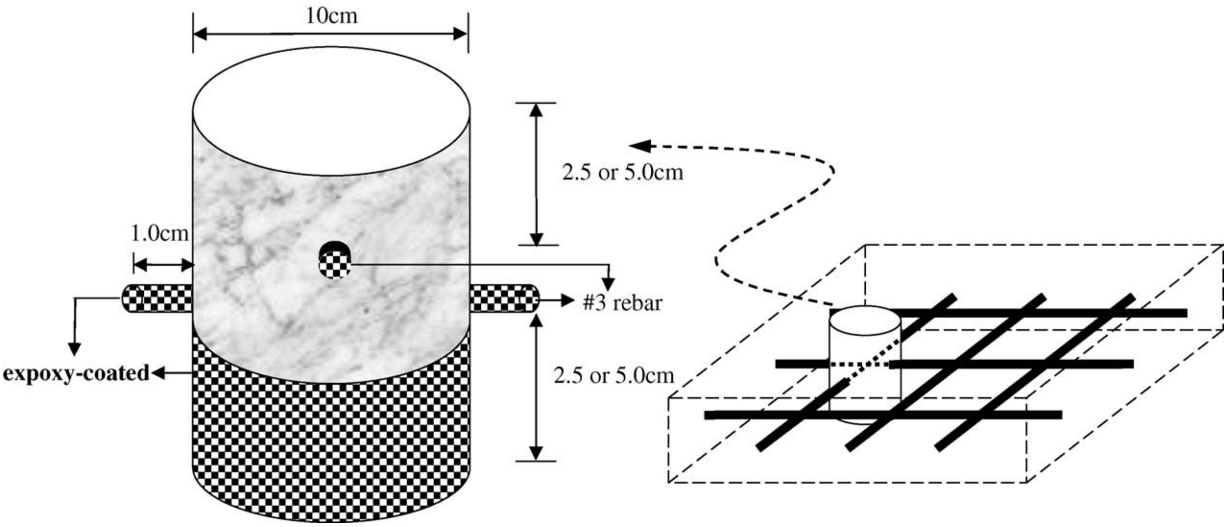

Fig. 1. Specimen designation and its purpose of simulating a concrete slab.

field to the reinforcements, two methods including the constant applied electric field intensity or constant applied electric current density have been used. In this study, the concept of using a constant applied current density was used. Four constant current densities including 100, 200, 400 and 800 μA/cm^2 were adopted for ECR. The area for the above-mentioned current densities corresponds to the protected area of rebar. The ECR durations were 1, 2, 3, 4, 5 and 6 weeks. An ECR parameter (ϕ), which is defined as the product of the ECR current density and the ECR time, was used.

2.3. Experimental procedures

The idea of designation for concrete specimens is sketched in Fig. 1. Our purpose is to simulate the ECR process for a reinforced concrete slab. Instead of using a full-scale slab, a simplified concrete cylinder was used. The distance from the bottom of the specimen to the nearest reinforcement is defined as the cover thickness. The cover thickness was 2.5 cm or 5 cm. Two reinforcements were used with a rectangle intersection and the electrical connectivity between them was introduced. The concrete specimen has a dimension of 10 cm diameter and 20 cm height. After demolding, the concrete specimen was coated in the circumferential surface in order to make sure chloride ions can only be removed while they pass through the bottom surface. In addition, the area of steel rebar exposed in the environment was coated and taped with 3M tape to ensure that the corrosion measurements represents the electrochemical property of the steel embedded inside the concrete. Another type of specimen was a beam specimen with the dimension of 10 cm × 10 cm × 20 cm. The cover thickness was 2.5 and 5 cm. The treatments on the concrete surface and exposed area of rebar as mentioned previously were conducted. The purpose of this beam-type specimen is to avoid the dimension effects of the cylindrical specimen while conducting the four-probe electrical resistivity measurement. The specimens were cast and cured for 28 days. After curing, specimens were connected to the DC power supply to perform the ECR process as shown in Fig. 2. The ECR electrolyte was the 0.1 M NaOH solution and the concentration was readjusted everyday according to the result of chemical titration. The adjustable resistor was used to ensure the applied ECR current density.

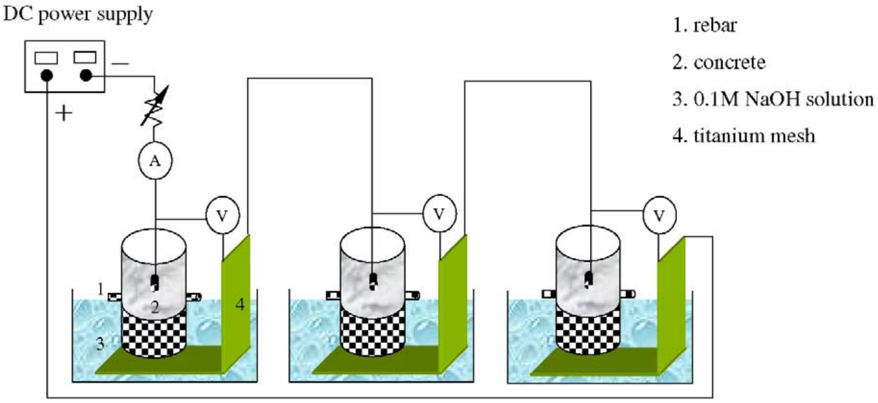

Fig. 2. The set-up for the electrochemical desalination process.

When the designated ECR time arrived, concrete specimens were disconnected from the ECR apparatus. Several experiments then were conducted as mentioned in the following. Extraction of the concrete in the region adjacent to the two reinforcements as shown in Fig. 3 was conducted. The scale of extracting region is 3 cm such that roughly a 1 cm sampling height along the cover thickness direction was adopted. The remaining chloride ion concentration and pH value within this region was measured. Unlike the traditional ECR concept that desires to completely remove free chloride inside the concrete to a degree, a pushing-away concept introduced in [11] was adopted in order to have a compromise between corrosion prevention and bond degradation. In this concept, the focus is to push chloride ions away from rebar in a degree such that the corrosion will not happen within a period (depending on how far the chloride ions were pushed away and the diffusivity of them inside the concrete) and the bond degradation is still acceptable. The main purpose of this research is to explore the design parameter of ECR from the first point, i.e., corrosion prevention. We hope to know how large the ECR current density should be as well as how long should one perform the ECR. The pH value and chloride ion content definitely are two important indices to check. The water-soluble chloride ion concentration was determined by potentiometric titration with 0.01 N silver nitrate ($AgNO_3$) solution. The pH value was determined by the following procedure. The concrete nearby the rebar was dry-cut and made into powder. The concrete powder was then placed into pure water with a known volume. Next, the concentration of OH^- ions was determined similar to the procedure for determining the water-soluble chloride ion concentration using the potentiometric titration instrument.

Other experiments performed on the cylindrical type specimen were the corrosion measurements, the half-cell potential and the DC corrosion rate. After the ECR, specimens were first placed in air for 2 months. The reason for doing so is to ensure that the specimens are completely depolarized. It can be seen from Fig. 4 that the half-cell potentials for various ECR specimens showed a very active nature of rebar just after ECR. This may lead us to make a wrong conclusion that ECR increases the corrosion tendency. However, as depolarization continued, the half-cell potential gradually became nobler and reached a stable value as shown in Fig. 4. The result shown in Fig. 4 indicates that a 2-month depolarization was appropriate. The four-probe electrical resistivity was measured on the beam-type specimens using the Wenner four-probe electrical resistivity meter.

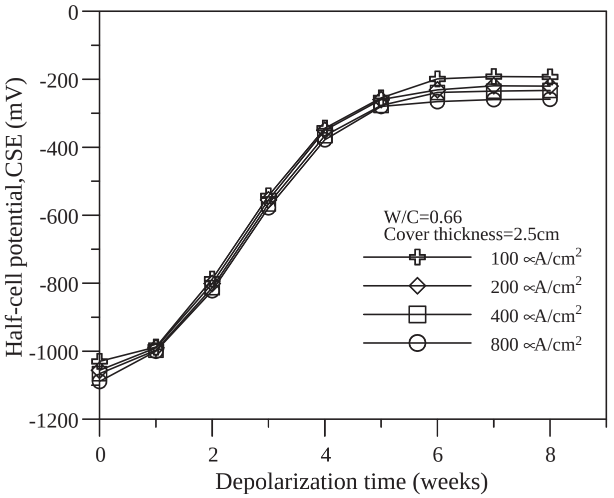

Fig. 4. Changes in half-cell potential after cutting off the desalination current.

3. Results and discussion

3.1. Remaining chloride ions

Generally speaking, the remaining chloride ion concentration decreased as the ECR current density and/or ECR time increased. To combine the effects of ECR current density and ECR time together, a parameter similar to our previous suggestions [24,25] was defined as the following:

$$\phi = i_\mathrm{d} \times t_\mathrm{d}$$

where i_d is the ECR current density and t_d is the ECR time. The results were shown in Fig. 5(a) and (b). In these figures, the dashed line represents the initial chloride ion concentration. It can be found in these figures that a unified regression curve

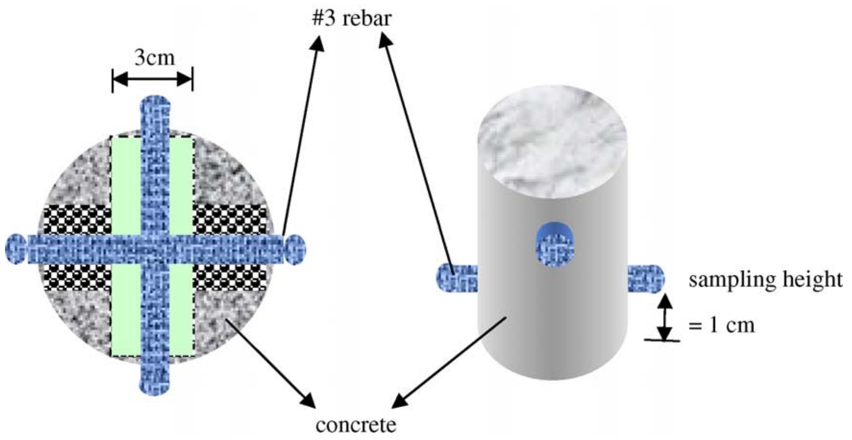

Fig. 3. Sampling position for chloride ions nearby the rebar.

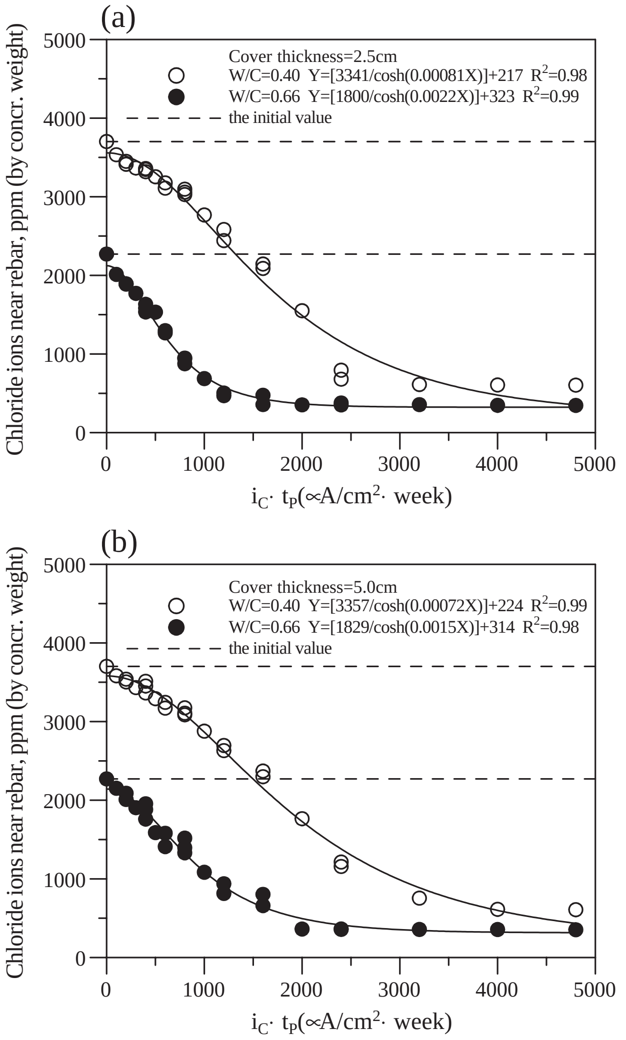

Fig. 5. Remaining chloride ion concentration nearby the rebar vs. the desalination parameter: (a) cover thickness of 2.5 cm and (b) cover thickness of 5.0 cm.

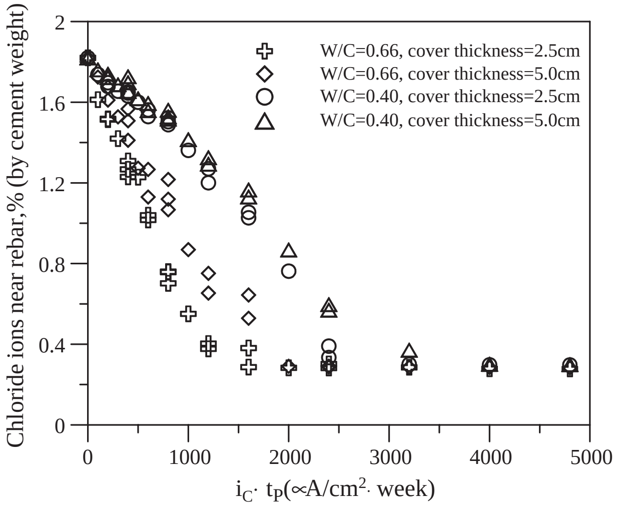

Fig. 6. Remaining chloride ion contents for various concrete specimens.

could be found despite of different combinations of ECR current density and ECR time. In addition, it is also found that after a period the ECR current cannot further push chloride ions away from the adjacent region we define. It is shown in Fig. 6 that ECR process can at most remove chloride ions from concentration of 1.82% to 0.3% by cement weight. It means that about 83% chloride ions were removed. In Table 2, the required minimum ECR parameters (ϕ) for different w/c ratio and cover thickness can be found if a 0.3% remaining chloride ion concentration (by cement weight) was the goal to achieve. It can be found that a higher value for the minimum ECR parameter is required when the concrete cover is thicker and the w/c ratio is a constant. A concrete with a thicker cover has a higher electrical resistance and thus requires more input energy to expel chloride ions.

In addition, comparing specimens with the same concrete cover thickness, it can be found that a larger minimum ECR parameter is required for a specimen with a lower w/c ratio. This phenomenon is due to the fact that a sound concrete with a lower w/c ratio has a denser microstructure and thus the diffusivity of ions becomes more difficult.

3.2. pH value

As the ECR process continues, the hydroxide ions accumulate due to the electrochemical reactions shown in Eqs. (1) and (2). The results for different conditions can be found in Fig. 7(a) and (b). Once again, it can be found that the ECR parameter can unify the data using different ECR current densities and different ECR periods. The pH value became higher when the ECR parameter was larger. The reason comes from the electrochemical reactions at the cathode as explained before. The physical meaning of ECR parameter can be understood as the total charge transferred through a unit metal surface. The larger the ECR parameter is, the more charges transferred that are being yielded by the electrochemical reactions should be counted.

In Ref. [24], it is mentioned that, for a stable growth of the passive film to overcoat the rebar, an environment with a pH value of 11.5 is suggested. In Table 2, the required minimum ECR parameters for various conditions to achieve pH = 11.5 are tabulated. It can be found that a less ECR parameter is required for achieving pH = 11.5 than expelling chloride ions to 0.3% by weight of cement. In addition, a thinner concrete cover and/or a

Table 2
Required desalination parameters under various conditions

W/C	Cover thickness (cm)	Required desalination parameter ($\mu A/cm^2 \times week$)			
		A*	B*	C*	D*
0.66	2.5	1935	221	117	1241
	5.0	2740	266	155	1915
0.40	2.5	3494	336	850	2205
	5.0	3962	461	2186	3185

A*: required chloride concentration is 0.3% by cement weight.
B*: pH value required is 11.5.
C*: half cell potential is −200 mV (CSE).
D*: corrosion rate is 0.08 mpy.

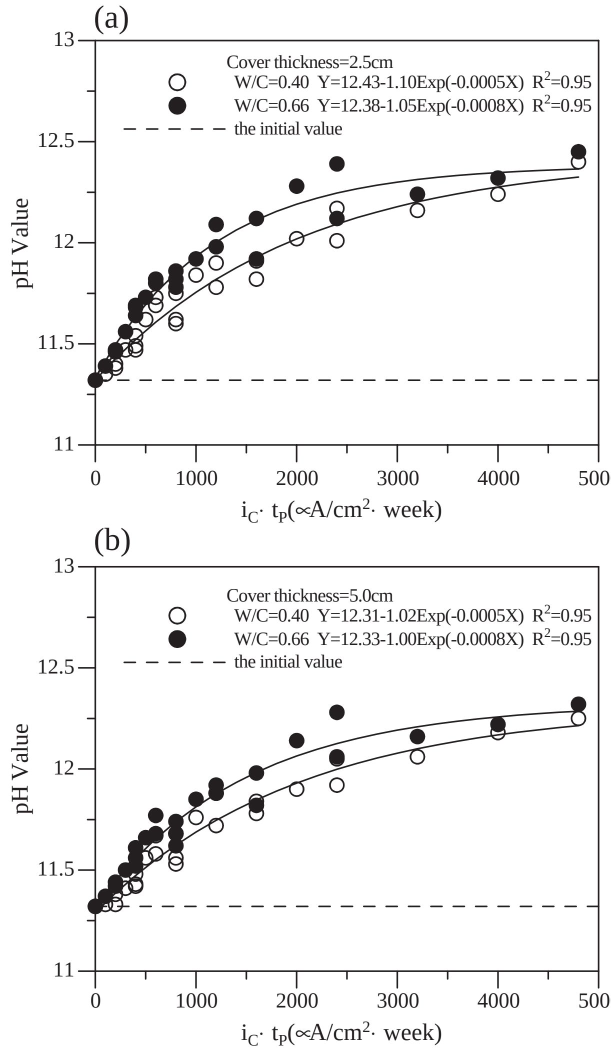

Fig. 7. pH values vs. the desalination parameter: (a) cover thickness of 2.5 cm and (b) cover thickness of 5.0 cm.

higher w/c ratio requires a less ECR parameter to achieve pH = 11.5, which is very similar to the result shown in the remaining chloride ions.

3.3. The half-cell potential

The half-cell potential represents the thermodynamic trend of corrosion. It cannot be used to represent the instantaneous corrosion rate. In ASTM C-876, it is suggested that, when the measured open circuit potential is lower than −350 mV (CSE), the rebar has a corrosion possibility higher than 90%. When the measured potential is higher than −200 mV (CSE), the corrosion possibility is lower than 10%. Using the half-cell potential as an indicator, −200 mV (CSE) was chosen as the goal we should achieve. As mentioned previously, measuring half-cell potential for specimens treated by the ECR current requires depolarization. The depolarization process is required; otherwise, the meaningless data might mislead us to wrongly make a judgement that ECR process cannot prevent corrosion at all. A 2-month depolarization time is chosen as mentioned earlier. Results are shown in Fig. 8(a) and (b). In these figures, the dashed line is used to represent the goal to achieve, i.e., a half-cell potential of −200 mV (CSE). As the ECR parameter increases, the half-cell potential moves toward a nobler value. It means that the corrosion risk dramatically decreases after the ECR. In Table 2, it can be found that a lower w/c ratio and/or a thicker concrete cover requires a larger value of ECR parameter to achieve −200 mV potential (CSE).

3.4. The DC corrosion rate

In order to measure the instantaneous corrosion rate of metal, two electrochemical methods are usually adopted: the DC method and the AC impedance method. It is understood now that the DC method contains an IR drop effect due to the resistance of concrete; thus, it will underestimate the corrosion rate. However, the DC method still can reflect the corrosion rates for various specimens relatively when their resistances are close. The DC corrosion rate is obtained by first measuring the Tafel slope for cathode and anode, then the Stern-Geary formula is used. Song

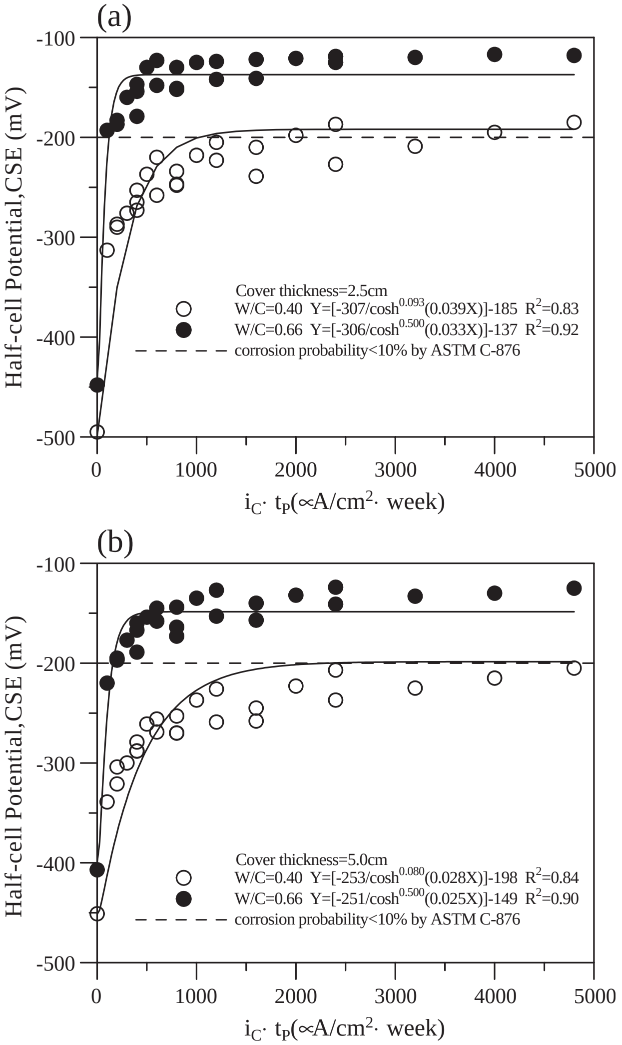

Fig. 8. Half-cell potential vs. the desalination parameter: (a) cover thickness of 2.5 cm and (b) cover thickness of 5.0 cm.

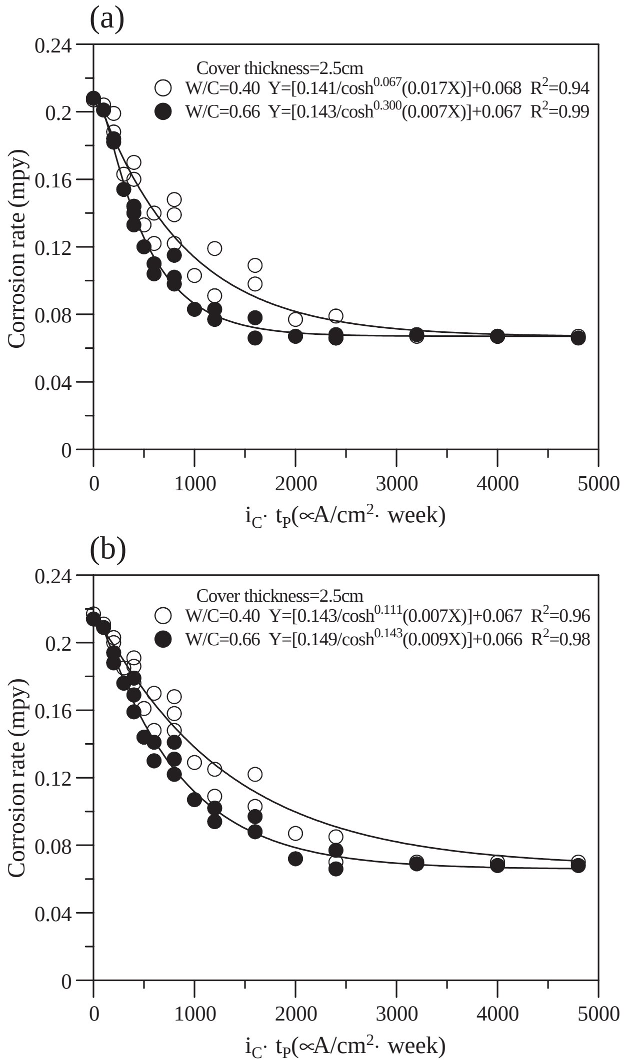

Fig. 9. Corrosion rate vs. the desalination parameter: (a) cover thickness of 2.5 cm and (b) cover thickness of 5.0 cm.

[25] has ever mentioned that the Tafel slope can be obtained correctly when the depolarization is considered. Based on this point and previously mentioned result, a 2-month depolarization time is used. According to a previous study [26], four ranges of the corrosion rate are proposed. When the corrosion current density is lower than 0.1 μA/cm^2 (or corrosion rate is lower than 0.046 mpy), the rebar corrosion rate is negligible. When the corrosion current density is within 0.1 to 0.5 μA/cm^2 (or corrosion rate is within 0.046 to 0.23 mpy), the rebar corrosion rate is slow. When the corrosion current density is within 0.5 to 1 μA/cm^2 (or corrosion rate is within 0.23 to 0.46 mpy), the rebar corrosion rate is mediated. When the corrosion current density is higher than 1 μA/cm^2 (or corrosion rate is faster than 0.46 mpy), the rebar corrosion rate is high. It was found that the corrosion rate for a concrete specimen without ECR increased from 0.21 mpy to 0.48 mpy after 6 weeks, which indicated that the rebar encountered a serious corrosion condition. However, when the ECR process is adopted, the corrosion rate for the rebar decreased from 0.21 mpy to a very low value as a result. The results for various conditions are shown in Fig. 9(a) and (b). From these results, it can be found that it requires a larger ECR parameter when the concrete cover thickness becomes thicker and/or the w/c ratio of concrete is lower. Furthermore, it can be found that the ECR process could not reduce the corrosion rate to a negligible margin (0.046 mpy) within our selected range. Observing these graphs, it can be found that the ECR reduced the corrosion rate efficiently before the corrosion rate reached 0.08 mpy. After the corrosion rate reached 0.08 mpy, increasing ECR parameter did not reduce the corrosion rate much. Therefore, a safety margin of corrosion rate ought to be achieved in the ECR process is selected as 0.08 mpy, which encounters a slow corroded condition. In Table 2, the required ECR parameters to achieve 0.08 mpy margin for various conditions are listed.

3.5. The resistivity

Wenner's four-probe resistivity measurements were performed. It should be noticed that the water content inside the concrete affects the resistivity. Therefore, the experiments

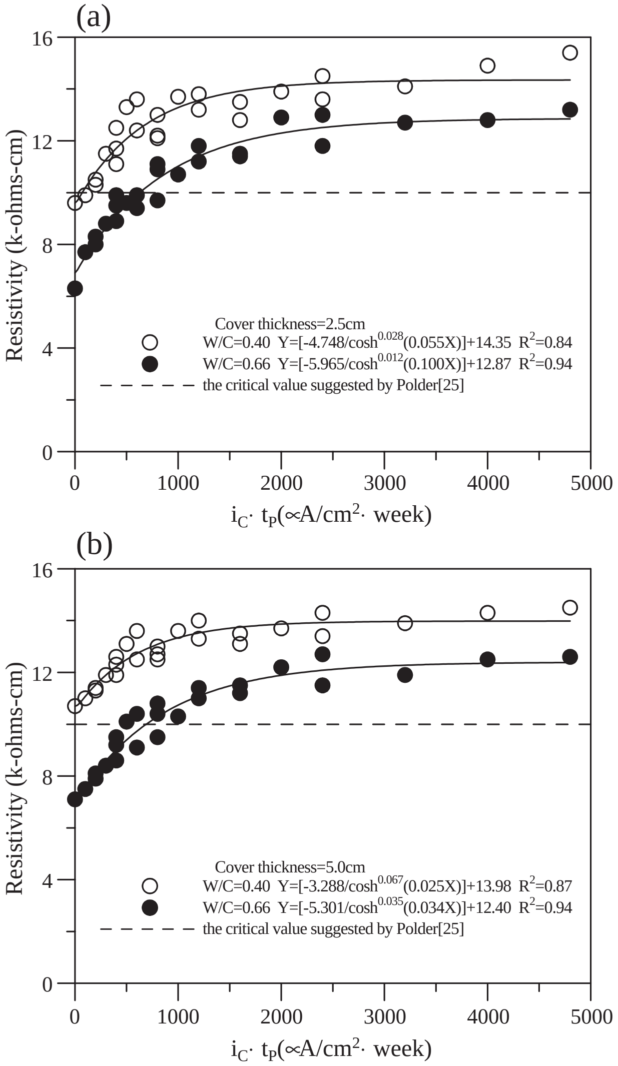

Fig. 10. Resistivity vs. the desalination parameter: (a) cover thickness of 2.5cm and (b) cover thickness of 5.0 cm.

Table 3
Relative change in resistivity after desalination ($\phi = 1000\ \mu\text{A/cm}^2 \times \text{week}$)

W/C	Cover thickness (cm)	Initial resistivity, R_i (kΩ cm)	Resistivity after polarization, R_f (kΩ cm)	$\frac{R_f - R_i}{R_i} \times 100\%$ (%)
0.66	2.5	6.3	11.1	76
	5.0	7.1	10.8	52
0.40	2.5	9.6	13.3	39
	5.0	10.7	13.3	24

should be conducted on specimens carefully. In this study, a SSD (saturated, surface dried) condition of concrete is ensured. The results of resistivity are shown in Fig. 10(a) and (b). A larger ECR parameter increased the resistivity due to a removal of free chloride ions such that the conductivity decreased. The conductivity depended on the free chloride ions inside the concrete; therefore, a more efficient ECR condition will increase the resistivity more. It then can be concluded both from experiment facts and our deduction that a concrete with a lower w/c ratio has a higher resistivity using the same ECR parameter. Polder [27] has mentioned that, when the resistivity of concrete is lower than 10 kΩ cm, the corrosion probability of rebar is very high. Therefore, a 10 kΩ cm resistivity is selected to represent the goal to be achieved. From our results, it can be found that the 10 kΩ cm resistivity can be achieved very quickly using ECR as shown in Fig. 10 or Table 3. To consider the efficiency and economic reasons, an ECR parameter of 1000 $\mu\text{A/cm}^2 \times$ week is selected. The resistivity after ECR (R_f) can then be obtained from Fig. 10. In addition, the relative change in resistivity (p) can be calculated as

$$p = \frac{R_f - R_i}{R_i} \times 100\%$$

in which R_i is the initial resistivity before ECR. It can be found that, for concrete with a lower w/c ratio, the relative change in resistivity is smaller.

3.6. Remarks

In previous results, we use a ECR parameter to combine the effect of ECR current density and ECR time. To use these results, a demonstration is given as follows. Let us assume that the concrete cover thickness is 5.0 cm, the w/c ratio of concrete is 0.66 and the initial chloride content is the same as the experiments. If the goal of ECR is to make the pH value near the rebar reach 11.5, the required ECR parameter is 266 $\mu\text{A/cm}^2 \times$ week from Fig. 7(b) or Table 2. If the ECR current density is 133 $\mu\text{A/cm}^2$, it means that the ECR time is 2 weeks.

In Tables 2 and 3, the required ECR parameters for achieving various safety margins suggested in the previous literatures or our selection are tabulated. We can conservatively select the maximum ECR parameter to guarantee that each safety criterion is satisfied. The safety ECR parameters for various conditions are selected based on this principle and are listed in the last column in Table 2. One may ask a question: can we use a larger ECR parameter than the safety ECR parameter? Maybe it will lower down the corrosion risk; however, a larger ECR parameter reduces the bond quality [10,11] and requires more energy.

It should be mentioned here that the concretes with different initial chloride ion contents and w/c ratios require conducting experiments in order to build up the database. It is a fruitful virgin land left for further exploring. Furthermore, the bond degradation is a major side effect of ECR process and should be taken into account beforehand.

4. Conclusions

In this study, how to select an appropriate ECR parameter (ECR current density and ECR time) is presented based on the evaluation from the remaining chloride ions near the rebar, the pH value near the rebar, the half-cell potential, the DC corrosion rate and the resistivity. It is found that concrete with a lower w/c ratio and/or a thicker cover thickness requires a larger ECR parameter to achieve the same condition. Using this concept, an ECR process can be selected by satisfying in two folds: saving energy and safety.

Acknowledgements

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