

## EFFECT OF ELECTROCHEMICAL PENETRATION OF LITHIUM IONS ON CONCRETE EXPANSION DUE TO ASR

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### Abstract

An electrochemical technique to accelerate the penetration of the lithium ions ( $\text{Li}^+$ ) in a lithium-based electrolyte solution into concrete has been developed for the purpose of suppressing ASR-induced expansion due to  $\text{Li}^+$ . From the results of past research work, the penetration area of  $\text{Li}^+$  is limited around the concrete surface and it is difficult to make  $\text{Li}^+$  penetrate into the deeper part of concrete. In this study, experimental investigation was carried out aiming to grasp the influence of the kinds of lithium salts and the temperature of the electrolyte solution on the migration properties of ions in concrete and ASR-induced expansion of concrete. The electrochemical migration of  $\text{Li}^+$  was found to accelerate with rises in temperature and the effective diffusion coefficient of  $\text{Li}^+$  increased three times with changes in temperature from 20°C to 40°C in the case of a  $\text{Li}_2\text{CO}_3$  electrolyte solution. Moreover, ASR-induced expansion of concrete after this treatment was suppressed compared with the case of non-treated specimens.

**Keywords:** electrochemical corrosion control technique, ASR, alkali, lithium

### 1 INTRODUCTION

It has been confirmed by many researchers that lithium salts have the effect of suppressing ASR-induced expansion of concrete [1], [2], [3]. In order to achieve this suppression effect, ASR-affected concrete needs to contain a sufficient amount of lithium salts. Supposing the application of the lithium salts as a repair additive for concrete structures deteriorated by ASR, sufficient amounts of  $\text{Li}^+$  must be driven into the ASR-affected concrete [4], [5], [6]. For supplying  $\text{Li}^+$  efficiently into concrete, an electrochemical technique has been investigated [7], [8], [9]. In this method,  $\text{Li}^+$  ions contained in the lithium-based electrolyte solution on the concrete surface migrate into concrete due to the electrophoresis that occurs by applying direct electric potential for a constant current between the anode immersed in the electrolyte solution and the steel bar serving as the cathode embedded in the concrete.

Past investigations [10], [11], [12] found that the ASR-induced expansion of concrete after the electrochemical treatment tended to be suppressed compared with the non-treated case mainly around the exposed surface. From this, it can be said that this technique has potential as a repair method aiming for the suppression of ASR-induced expansion. However, the penetration depth of  $\text{Li}^+$  supplied from the electrolyte solution was limited around the concrete surface [10], [11], and deeper penetration of  $\text{Li}^+$  into concrete was required.

Against this background, this study was conducted as an experimental investigation for promoting the electrochemical penetration of  $\text{Li}^+$  and suppressing ASR-induced expansion by testing various kinds of lithium salt and using different electrolyte solution temperatures during treatment. This investigation was

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aimed at clarifying the effective conditions of electrochemical treatment for the accelerated penetration of  $\text{Li}^+$  and suppression of ASR-induced expansion of concrete.

## 2 MATERIALS AND METHODS

### 2.1 Materials and preparation of specimen

The specimens prepared for this study were concrete prisms (100×100×300 mm) and mortar cylinders ( $\varphi$ 100×200 mm). In the concrete specimen, a deformed steel bar  $\varphi$ 13 SR235 (JIS number) was embedded at the center of the square section (refer to Figure 1). The mix proportion of the concrete used for the specimens is shown in Table 1. The W/C ratio of concrete was 0.6. Ordinary portland cement and non-reactive fine aggregates (density: 2.61 g/cm<sup>3</sup>) were used, and as the coarse aggregate, reactive aggregate (G1, density: 2.60 g/cm<sup>3</sup>) and non-reactive aggregate (G2, density: 2.60 g/cm<sup>3</sup>) were mixed at the pessimum weight ratio of 6:4. The rock type of reactive aggregate was andesite and the reactivity of this aggregate was confirmed by the chemical method (JIS A 1145). Moreover, the pessimum ratio was tested by the mortar-bar method (JIS A 1146). In order to adjust the total alkali contents as  $\text{R}_2\text{O} = 8.0 \text{ kg/m}^3$  for the acceleration of ASR in each concrete specimen, a corresponding amount of NaCl was dissolved in the mixing water, supposing the combined deterioration of ASR and chloride attack. All specimens were cured for 28 days in the wet condition. After curing, five faces of each specimen were insulated with epoxy resin coating, leaving one exposed surface (100×300 mm) as shown in Figure 1.

### 2.2 Electrochemical treatment

Electrochemical treatment was applied to the concrete specimens after epoxy resin coating. As the electric current density, 2.0 A/m<sup>2</sup> to the concrete surface was selected considering the results of past studies [10], [11]. The period for treatment was 8 weeks. As the electrolyte solutions, 5N LiOH solution, 0.5N Li<sub>2</sub>CO<sub>3</sub> solution and each solution with 5% surface-activate agent were selected. The surface-activate agent is a copolymer of ethylene oxide and propylene oxide, which is generally used to reduce drying shrinkage of concrete as the result of reduction of the surface tension in pore solution. The surface-activate agent was added to promote the penetration of the lithium-based electrolyte solution into the concrete by the reduction of the surface tension, which may impede the migration of the solution. As the temperature of the electrolyte solution during treatment, 20°C and 40°C were selected. According to a previous study by the authors' [11], the higher temperature can accelerate the electrochemical migration. Some of the concrete specimens were subjected to the conditions for ASR acceleration (40°C, R.H. 95%) for two months before the start of the electrochemical treatment. The list of concrete specimens used in this research is shown in Table 2. The number of specimens for each experimental factor was three.

Ten millimeter thick disks were sliced from the mortar cylinders after the 28-day curing period for the electrophoresis test of  $\text{Li}^+$ . The test was performed basically according to “Test method for effective diffusion coefficient of chloride ion in concrete by migration (JSCE-G571-2003),” but the electrolyte solution in the anode cell was a lithium-based electrolyte solution (5N LiOH solution or 0.5N Li<sub>2</sub>CO<sub>3</sub> solution) and the electrolyte solution in the cathode cell was a saturated Ca(OH)<sub>2</sub> solution (refer to Figure 2). The temperature of the electrolyte was controlled at 20, 30 or 40°C using a rod type heater.

### 2.3 Measurements after electrochemical treatment

Concrete specimens that underwent the electrochemical treatment were kept in the environment for the acceleration of ASR (40°C, R.H. 95%) and the expansion rate of concrete was measured regularly by means of a contact gauge. Measured points in each concrete specimen were 1) near the exposed surface, 2) above the steel bar, 3) opposite side of the exposed surface and 4) vertical to the longitudinal direction of the

steel bar, as shown in Figure 1. Moreover the distribution profiles of ion content ( $\text{Cl}^-$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Li}^+$ ) in the concrete specimens were measured immediately following the treatment using powder samples grinded from the cut out seven plates in a specimen, as shown in Figure 3. The  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Li}^+$  contents were measured by means of atomic absorption spectrometry using a crashed fine powder sample solved by  $\text{HNO}_3$  solution.  $\text{Cl}^-$  content was measured according to JIS A 1154. Moreover, electron probe micro analyzer (EPMA) mapping of the cross section of the concrete specimens (N, SC, SC40) was conducted to grasp the two dimensional distributions of  $\text{Cl}^-$  and  $\text{Na}^+$  formed by the electrochemical treatment. In the electrophoresis test for mortar specimens,  $\text{Li}^+$  content in the cathode cell was measured twice a week during the period of electric current supply.

### 3 RESULTS AND DISCUSSION

#### 3.1 Effective diffusion coefficient of $\text{Li}^+$

The effective diffusion coefficients of  $\text{Li}^+$  in the mortar specimens were calculated by the following equation referring the equation for  $\text{Cl}^-$ .

$$D_e = \frac{J_{\text{Li}}RTL}{|Z_{\text{Li}}|FC_{\text{Li}}\Delta E} \quad (1)$$

where  $D_e$  is the effective diffusion coefficients of  $\text{Li}^+$ ,  $J_{\text{Li}}$  is the flux of  $\text{Li}^+$  ( $\text{mol}/(\text{cm}^2 \cdot \text{year})$ ),  $R$  is the gas constant ( $8.31 \text{ J}/(\text{mol} \cdot \text{K})$ ),  $T$  is the absolute temperature,  $Z_{\text{Li}}$  is the charge number of  $\text{Li}^+$  ( $=1$ ),  $F$  is the Faraday constant ( $96500 \text{ C}/\text{mol}$ ),  $C_{\text{Li}}$  is  $\text{Li}^+$  content in anode cell ( $\text{mol}/\text{l}$ ),  $\Delta E$  is the potential difference between electrodes (V), and  $L$  is the thickness of mortar disk specimen (mm).

The calculated  $D_e$  values are shown in Figure 4. From Figure 4, higher temperature of the electrolyte solution promotes the migration of  $\text{Li}^+$  and results in larger effective diffusion coefficient. Such temperature effect may be related to the Arrhenius equation, which indicates that higher temperature activates the migration of ions. As for the kind of lithium salts, the  $\text{Li}_2\text{CO}_3$  solution promotes larger effective diffusion coefficient than the  $\text{LiOH}$  solution. In the case of the  $\text{Li}_2\text{CO}_3$  electrolyte solution, the effective diffusion coefficient of  $\text{Li}^+$  increased three times with the change of temperature from  $20^\circ\text{C}$  to  $40^\circ\text{C}$ .  $\text{LiOH}$  is highly soluble in water and a large amount of  $\text{Li}^+$  in the  $\text{LiOH}$  solution may migrate through electrophoresis under an electric field. In that case,  $\text{Li}^+$  should be affected by the effect of the electric double layer on the wall of micro pores against penetration into mortar. On the other hand,  $\text{Li}_2\text{CO}_3$  is of low solubility in water and the penetration of the  $\text{Li}_2\text{CO}_3$  solution into the mortar will be mainly achieved by the principle of electroosmosis through the driving force of an electric field. The electric double layer would have almost no impact on the migration of the  $\text{Li}_2\text{CO}_3$  solution due to electroosmosis, resulting in the larger values of effective diffusion coefficient.

#### 3.2 Distribution of $\text{Cl}^-$ content in concrete

The distributions of  $\text{Cl}^-$  content in the specimens just after completing treatment are shown in Figure 5. The names of the specimens are defined in Table 2. The origin of the horizontal axis in Figure 5 corresponds to the exposed surface and the steel bar is located at the center (5 cm from the exposed surface) of each specimen. Although this technique is not for extracting  $\text{Cl}^-$  from concrete,  $\text{Cl}^-$  content around the steel bar located at 5 cm from the exposed surface is decreased by the electrochemical treatment compared with the non-treated case (N), because this technique is based on the same principle as that of desalination. Since such a side effect is expectable, this technique could be applied with good effect on concrete structures deteriorated by the combined mechanism of chloride attack and ASR.

The amount of extracted chlorides increases remarkably with the rise in the electrolyte temperature from 20°C to 40°C. Such a tendency can be clearly seen in Figure 6 showing EPMA maps of Cl<sup>-</sup> in specimens SC and SC40. These photos show that a rise in the electrolyte temperature contributes not only to the increase of the extracted amount of Cl<sup>-</sup> but also the spread of the low chloride content area around the steel bar. The reason for this is considered to be that the migration of Cl<sup>-</sup> due to electrophoresis was accelerated by the rise in temperature, considering the similar phenomenon of Li<sup>+</sup> observed in Figure 4. In this study, the electrolyte temperature of 40°C was selected in order to accelerate the penetration of Li<sup>+</sup> but as a secondary effect, the chloride removal effect was improved. According to Figure 5, the effect of the addition of surface-activate agent is negligible.

### 3.3 Distribution of R<sub>2</sub>O content in concrete

The distributions of R<sub>2</sub>O content in concrete specimens immediately after completing treatment are shown in Figure 7. The amount of R<sub>2</sub>O was calculated by the following formula.

$$R_2O = Na_2O + 0.658K_2O \text{ (kg/m}^3\text{)} \quad (2)$$

According to Fig. 7, in the case of non-treated specimens, approximately the premixed amount of R<sub>2</sub>O of 8.0 kg/m<sup>3</sup> is distributed uniformly, while in the case of the specimens treated with the electrolyte of 20°C, a great amount of R<sub>2</sub>O accumulates around the steel bar in concrete. It is considered that such an accumulation of alkali is caused by the electrophoresis of cations (Na<sup>+</sup>, K<sup>+</sup>) contained in the concrete toward the steel bar as the cathode.

Like the case of Cl<sup>-</sup> distribution, the rise in electrolyte temperature from 20°C to 40°C might accelerate the electrophoresis of Na<sup>+</sup> and K<sup>+</sup>. Then, it can be expected that the rise in temperature would result in a larger amount of R<sub>2</sub>O accumulation around the steel bar. However, in Figure 7, when 0.5 N Li<sub>3</sub>CO<sub>3</sub> is used as the electrolyte solution, the accumulated amount of R<sub>2</sub>O is much reduced with the rise in the electrolyte temperature from 20°C (the case of SC) to 40°C (the case of SC40). Such a tendency can be clearly seen in Figure 8 showing EPMA maps of Na<sup>+</sup> in the specimens SC and SC40. From these photos, the rise in the electrolyte temperature realizes the cancellation of the concentrated alkali around the steel bar, which can be observed in the case of SC. Such a moderation of the alkali accumulation at the steel bar may be caused by the decrease of the transference numbers of Na<sup>+</sup> and K<sup>+</sup> in concrete with the migration of lithium-based electrolyte solution. The reduction of the concentrated alkali at the steel bar due to the rise in the electrolyte temperature would mean the reduction of the risk of ASR or ASR-induced expansion of the concrete around the steel bar.

### 3.4 Distribution of Li<sup>+</sup> content and Li/Na molar ratio in concrete

Distributions of Li<sup>+</sup> content and Li/Na molar ratio in the concrete immediately after treatment are shown in Figure 9 and Figure 10, respectively. The authors reported that it was difficult to drive a sufficient amount of Li<sup>+</sup> into the concrete at the depth of the steel bar by the electrochemical treatment with the LiOH electrolyte solution at room temperature [10], [11]. However, from Figure 9, it can be found that both the amount and the depth of Li<sup>+</sup> penetration into concrete are accelerated by the rise in the electrolyte temperature from 20°C to 40°C. Such a result would be caused by the acceleration of the electroosmosis of the Li<sub>2</sub>CO<sub>3</sub> electrolyte solution by the rise in temperature, as shown in Figure 4. The effect of the addition of surface-activate agent is negligible like the tendency in the cases of Cl<sup>-</sup> and Na<sup>+</sup>.

Among many theories, it has been reported that a Li/Na molar ratio over 1.0 is necessary for the suppression of ASR-induced expansion of concrete [8], [13]. From Figure 10, in the cases of specimens treated with the electrolyte of 20°C, the concrete part where the Li/Na molar ratio exceeds 1.0 is limited near the exposed surface and the Li/Na molar ratio measured around the steel bar where Na<sup>+</sup> ions are

accumulated is far less than 1.0. On the other hand, when the electrolyte temperature is 40°C, the Li/Na molar ratio reaches about 1.0, including the part around the steel bar, through the combined effect of the promoted Li<sup>+</sup> penetration and suppressed Na<sup>+</sup> accumulation.

### 3.5 Variation of expansion rate of concrete after treatment

Variation curves of the concrete expansion rate with time are shown in Figure 11. The horizontal axis of these figures expresses the total period after the end of the curing. Figure 11 shows the expansion rate of concrete measured near the exposed surface, above the steel bar, on the opposite side of the exposed surface and in the vertical direction to the longitudinal way of the steel bar, respectively, as shown in Figure 1. The curves of specimens treated with the electrolyte mixing the surface-activate agent are not shown in Figure 11 because there was almost no effect of the agent on the expansion rate.

Non-treated specimens (N) were moved to the environment for accelerating ASR immediately at the end of the curing period of 28 days and the values of the concrete expansion rate were measured regularly, while the treated specimens were subjected to the conditions for ASR acceleration after the completion of the electrochemical treatment, at which time expansion measurement started, but the original length data were measured before the treatment.

From Figure 11, although the non-treated specimen kept in the environment for ASR acceleration shows relatively large expansion exceeding 0.16%, all the treated specimens show a small expansion rate below 0.05% after the treatment. Such a suppression of concrete expansion would be achieved by a large amount of Li<sup>+</sup> contained in the concrete around the concrete surface, as shown in Figure 11. When the temperature of the electrolyte is 40°C, the concrete expansion during the electrochemical treatment becomes large but almost no increase of the concrete expansion after the treatment is observed. It can be considered that the high temperature treatment holds the risk of accelerating of concrete expansion during the treatment but it also has the effect of promoting the lithium penetration that could suppress the concrete expansion after the treatment. As for the specimens subjected to ASR acceleration for 2 months before starting the electrochemical treatment, the increase in concrete expansion after the treatment is relatively small.

As shown in Figure 7, a large amount of alkali was accumulated around the steel bar by the low temperature treatment and such alkali could accelerate ASR-induced expansion of concrete. However, according to Figure 11, concrete expansion rates above the steel bar are also suppressed by the electrochemical treatment compared with the non-treated cases. It was reported that excessive supply of electricity exceeding the pessimum value did not accelerate the ASR-induced expansion of the concrete [7]. In this study, the amount of accumulated alkali around the steel bar due to the treatment may exceed the pessimum value and this may result in relatively small ASR-induced expansion. Further investigation is necessary to clarify the suppression mechanism of concrete expansion near the steel bar where a sufficient amount of Li<sup>+</sup> is not detected.

From Figure 11, at the opposite side of the exposed surface, the expansion rates of treated concrete are larger than the case of other measuring points. As this part is farthest from the exposed surface, the suppression effect of Li<sup>+</sup> would be small. However, compared with the non-treated case (N), the expansion rate of concrete is also suppressed by the treatment. It is considered that such suppression may be caused by the migration of Na<sup>+</sup> or K<sup>+</sup> contained in the concrete backside toward the steel bar as the cathode electrode with the influence of the electrochemical treatment. In this case, higher temperature treatment (SC40) shows the greatest suppression effect. Since the vertical concrete expansion is not confined by the steel bar, the values of the expansion rate shown in Figure 11 are generally larger than the other cases. However, even in this case, the suppression effect due to the electrochemical treatment is confirmed especially when the temperature of the electrolyte is 40°C.

### 3.6 Analysis of white deposits observed in treated concrete specimens

After completing the ASR acceleration test, the concrete specimens were split and the fractured surface was observed. White deposits were found in some air voids of the treated concrete around the steel bar as shown in Figures 12 and 13. A chemical analysis of the white product was carried out by using Energy Dispersion X-ray Spectrometry (SEM-EDS). As the conditions of analysis, quantitative analysis with standards was conducted while the data were corrected by the ZAF method. The results of the EDS analysis are shown in Table 3. According to this table, the electrochemical treatment forms gels that are of lower alkali content and higher CaO/SiO<sub>2</sub> ratio than the ASR gels formed in the non-treated specimens. Ramyar et al. also reported that the CaO/SiO<sub>2</sub> ratio of ASR products increased with the addition of lithium compounds [14]. However, other researchers reported the opposite, namely that the addition of lithium compounds lowered the CaO/SiO<sub>2</sub> ratio of the gels [6]. Further investigation of this point is necessary.

## 4 CONCLUSIONS

This study carried out an experimental investigation to grasp the electrophoresis property of Li<sup>+</sup> and the expansion behavior of treated concrete under various conditions of electrochemical treatment. The results obtained from this study can be summarized as follows.

- (1) As the results of the electrophoresis test using mortar specimens, the effective diffusion coefficient of Li<sup>+</sup> with the Li<sub>2</sub>CO<sub>3</sub> electrolyte solution was larger than that with the LiOH electrolyte solution. Moreover, in the case of the Li<sub>2</sub>CO<sub>3</sub> electrolyte solution, the effective diffusion coefficient of Li<sup>+</sup> increased three times with the change of temperature from 20°C to 40°C.
- (2) The higher temperature (40°C) of the Li<sub>2</sub>CO<sub>3</sub> electrolyte solution remarkably promoted the electrochemical migration of Cl<sup>-</sup> and Li<sup>+</sup> in concrete compared with the case of the lower temperature (20°C). As a result, the Li/Na molar ratio exceeded about 1.0 including the part around the steel bar owing to the high temperature treatment.
- (3) Regardless of the conditions for electrochemical treatment, the concrete expansion rate after the treatment was suppressed compared with the non-treated case. It can be considered that the high temperature treatment has the risk of accelerating concrete expansion during the treatment but it also has the effect of promoting lithium penetration, which could suppress concrete expansion after the treatment.
- (4) Chemical analysis of the white deposits observed in the treated concrete specimens suggests that the electrochemical treatment forms lower alkali content and higher CaO/SiO<sub>2</sub> ratio gels compared with the ASR gels made in the non-treated concrete.

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Table 1: Mix proportion of concrete.

R <sub>2</sub> O (kg/m <sup>3</sup> )	W/C (%)	s/a (%)	Gmax (mm)	Content (kg/m <sup>3</sup> )								
				C	W	S	G1	G2	WRA*	AEA**	NaCl	
8.0	60	45	15	300	180	781	579	386	0.6	0.02	11.9	

\*WRA: Water reducing agent \*\*AEA: Air entraining agent

Table 2: List of concrete specimens.

Name	Period of ASR acceleration before treatment (Month)	Current Density (A/m <sup>2</sup> )	Electrolyte solution	Temperature (°C)
N	0	0	-	20
SH		2.0	LiOH	20
SHA			LiOH+SA*	20
SC			Li <sub>2</sub> CO <sub>3</sub>	20
SC40				40
SCA			Li <sub>2</sub> CO <sub>3</sub> +SA*	20
SCA40				40
MH		2	2.0	LiOH
MC	Li <sub>2</sub> CO <sub>3</sub>			20

\*SA: surface-activate agent

Table 3: Chemical composition of white deposits or ASR gel.

Name	Composition (%)			
	R <sub>2</sub> O	CaO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
N	4.42	54.87	3.21	37.13
SC	3.82	58.53	3.69	32.5
SC40	2.65	64.77	3.12	26.71

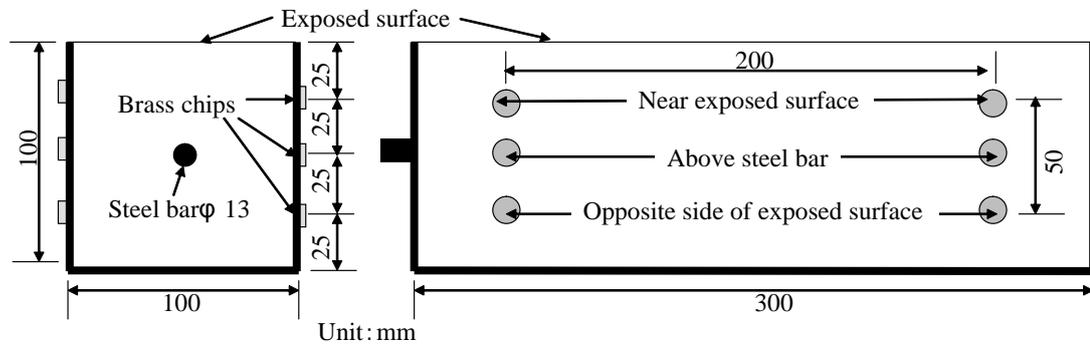


Figure 1: Outline of concrete specimen.

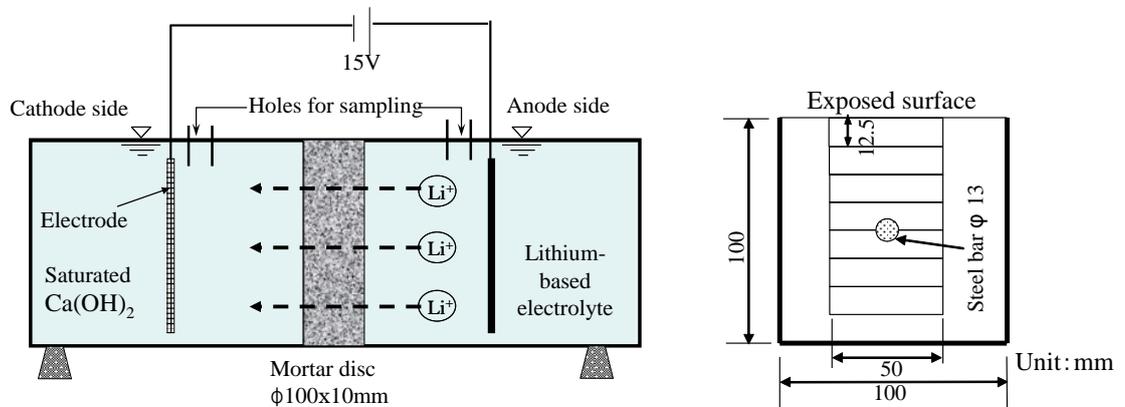


Figure 2: Outline of electrophoresis test for mortar specimen. Figure 3: Cutting of concrete specimen for chemical analysis.

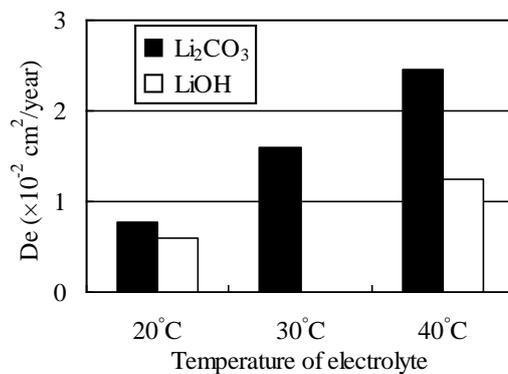


Figure 4: Effective diffusion coefficients of Li<sup>+</sup>.

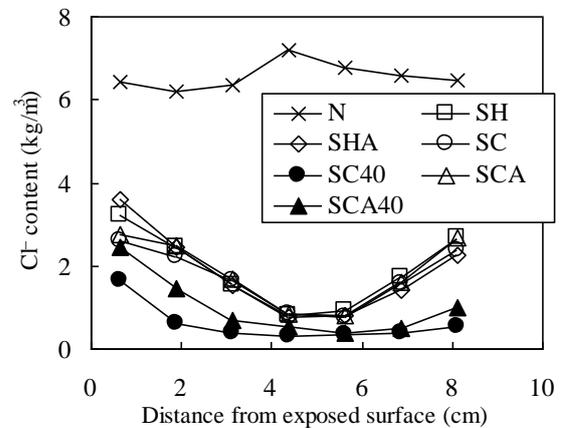


Figure 5: Distributions of Cl<sup>-</sup> in concrete

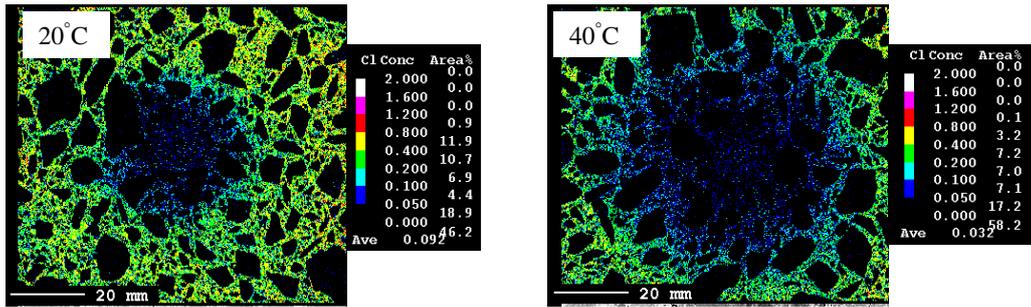


Figure 6: EPMA map of Cl<sup>-</sup> around steel bar in specimens SC and SC40.

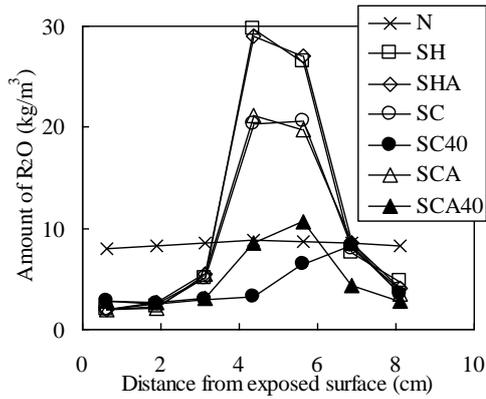


Figure 7: Distributions of R<sub>2</sub>O in concrete.

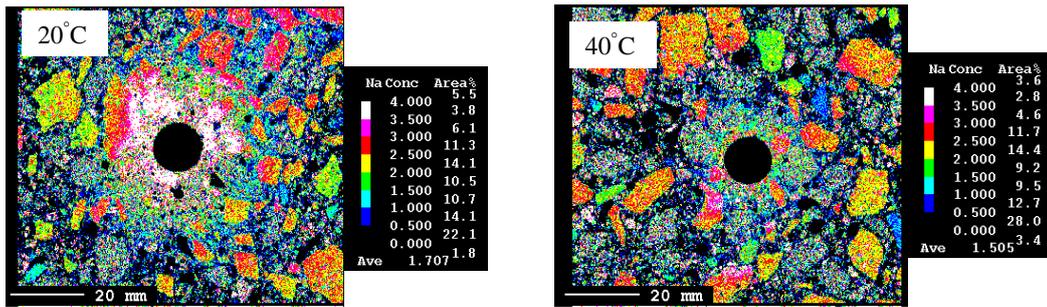


Figure 8: EPMA map of Na<sup>+</sup> around steel bar in specimens SC and SC40.

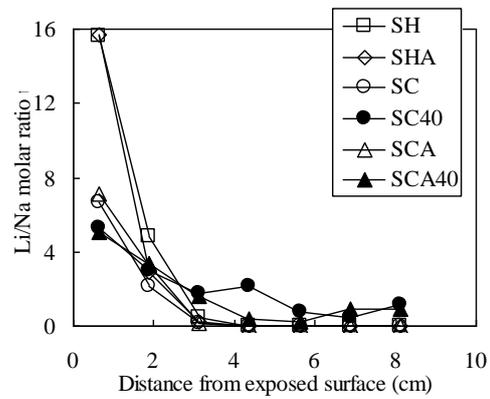
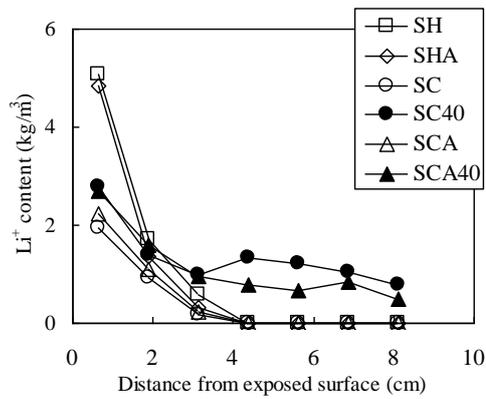


Figure 9: Distributions of Li<sup>+</sup> content in concrete. Figure 10: Distributions of Li/Na molar ratio in concrete.

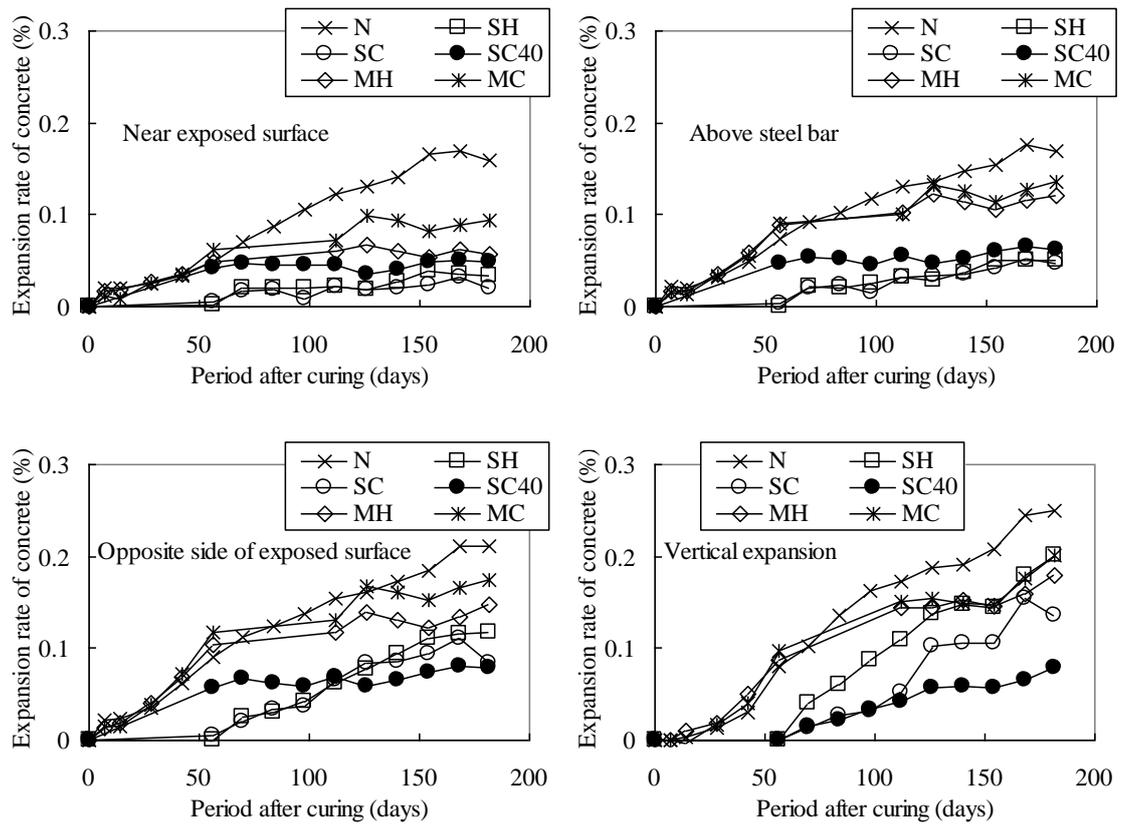


Figure 11: Variations of concrete expansion rate with time after curing.



Figure 12: White deposits around steel in specimen. (SC40)



Figure 13: White deposits observed by microscope.