

Redistribution of chloride after electrochemical chloride removal from reinforced concrete prisms

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Electrochemical chloride removal (ECR) of reinforced concrete is an innovative repair technique gaining widespread acceptance. By removal of 1/3 to 2/3 of the chloride penetrated into the concrete cover it is possible to suppress chloride-induced corrosion of the reinforcement. A non-stationary distribution of the remaining chloride will remain in the concrete cover. This chloride will diffuse back to the reinforcement, which may reinitiate corrosion. In this paper the chloride redistribution inside concrete specimens over one year after removal is described. Based on these findings the redistribution of chloride over a period of 10 years was assessed by numerical calculations. It was found that in concrete with a low total initial chloride content and low chloride mobility (Blast Furnace Slag Cement, low water cement ratio), chloride removal treatment will suppress corrosion of the reinforcement over an extended period of time. In concrete with a relatively high average initial chloride content and high chloride mobility (Ordinary Portland Cement, high water cement ratio), reinforcement corrosion may be reactivated due to redistribution of chloride within a ten year period.

Keywords: reinforcement corrosion, durability, electrochemical chloride removal, desalination, chloride redistribution, numerical simulation, concrete

1 Introduction

Ingress of chloride into the outer layers of concrete structures may cause corrosion of the reinforcement and subsequent damage to the concrete in the form of cracking and spalling. Traditional repair procedures involve breaking out of the chloride-contaminated layer, even if it is structurally sound and application of new, chloride free concrete. This involves high labour costs and may cause considerable disruption due to the noise and dust produced, for example in inhabited buildings or cause serious traffic disturbance on bridge decks.

In order to overcome these drawbacks of conventional repair methods, innovative and less invasive techniques have been proposed and subsequently become operative in practice. They are based on electrochemical principles and the main advantage is the reduction of the amount of old concrete that has to be removed. Cathodic protection is such a technique, which has been successfully applied for the last 20 years on a large scale, see for example (Polder 1998). Another electrochemical technique used in this area is electrochemical chloride removal, also indicated as chloride extraction

or desalination. This technique aims to reduce the chloride level near the reinforcement down to the level at which the risk of corrosion becomes negligible. The selection of various methods for concrete repair and protection was discussed in (COST 1997).

Electrochemical chloride removal (ECR) has proven to be capable of extracting penetrated chloride from the cover of reinforced concrete, both in laboratory experiments as well as in field trials. Examples have been described in (Polder 1994, 1996), (Elsener et al. 1997) and (Tritthart 1996). The state of the art was recently described in (Mietz 1998). The chemical and small scale mechanical effects in cement pastes have been studied (Bertolini et al. 1996) and the effects on bond between steel and concrete have been studied (Vennesland et al. 1996). Some questions have remained. For example, experience shows that it is not possible to bring the chloride content down to zero. Some chloride remains, usually with a high chloride level near the outer surface of the structure and a low chloride level near the reinforcement. This remaining chloride may migrate back to the reinforcement and again eventually result in (re)initiation of corrosion. This redistribution of chloride remaining after removal is the subject of the present paper. Before going into detail, the principles of electrochemical chloride removal will be discussed briefly.

2 Principles of electrochemical chloride removal from concrete

Normally steel in concrete is protected against corrosion by the formation of an atomically thin layer of iron oxides on the steel due to the high pH in the concrete pore solution. This condition is called passivation. Passivation may be lost due to the presence of chloride ions, either from external sources such as de-icing salts or sea water, or from mixing in chloride into the fresh concrete mix as a set accelerator. The chloride ions break down the passive film and corrosion pits may be formed. The corrosion products are more voluminous than the steel and the expanding forces crack the concrete. Electrochemical protection techniques aim at reducing the corrosion activity by passing an electrical direct current. Cathodic protection involves a permanent current to the steel reinforcement aimed at changing the potential of the steel into a less corrosive region. Under cathodic protection conditions, the presence of chloride does no longer cause corrosion. Electrochemical chloride removal has another objective, namely to reduce the amount of chloride inside the concrete. A direct current is passed between the steel and a temporary external counter electrode, called the anode (usually an activated titanium mesh). The steel is made negative and the anode positive, which forces the negatively charged chloride ions to move out of the concrete into the electrolyte surrounding the anode. When the chloride content of the concrete has been reduced sufficiently, the treatment is stopped and the electrolyte and the anode are removed. The treatment usually takes several weeks to months. The currents involved are relatively high, namely corresponding to about 1 A/m^2 of steel surface area (compare to 10 mA/m^2 for cathodic protection). The principle of electrochemical chloride removal is schematically shown in Figure 1.

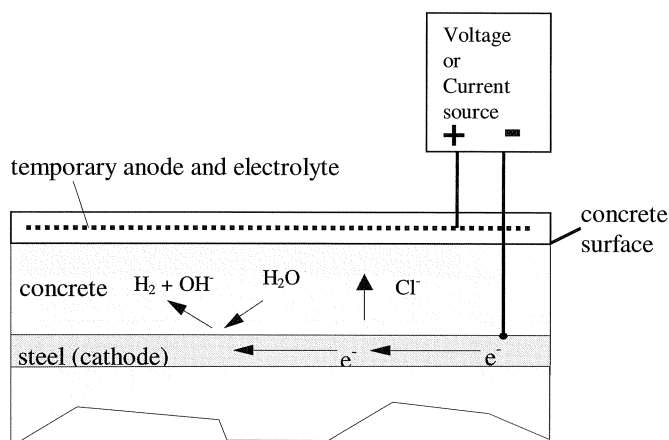


Fig. 1. Principle of electrochemical chloride removal

In several tests, it has been shown to be rather difficult to remove chloride that was mixed in during preparation of concrete. The efficiency of removal of mixed in chloride is low and impractically long treatment times would be necessary. Most researchers have come to the conclusion that extraction of mixed in chloride is not feasible (Mietz 1998). For various reasons, extraction of chloride ions that have penetrated into the concrete cover from external sources is easier.

In all cases, some chloride remains in the concrete after the treatment, usually in a non-equilibrium distribution with lower contents near the reinforcement and higher contents near the concrete surface. Even if further ingress of chloride into the structure is excluded, e.g. by applying a coating or hydrophobic treatment, the remaining chloride may diffuse to the reinforcement and re-initiate corrosion. Such re-initiation would mark the end of the durability of the chloride removal result. It may be assumed that for sufficiently wet concrete, the redistribution of chloride is governed by diffusion, commonly described by Fick's second law.

In this article the redistribution of chloride after electrochemical chloride removal is studied. Experiments were carried out on concrete specimens that had been exposed to a marine environment. Subsequently the chloride was removed by chloride removal. After removal the specimens were stored in outdoor conditions for one year. Based on chloride migration during this exposure period, the long term effectiveness of chloride removal was extrapolated by numerical analysis.

3 Specimen history

The specimen history is outlined schematically in Table 1.

Table 1. Specimen history.

Activity	Year	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Production			■																	
North Sea exposure				■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	
Chloride removal																			■	
Outdoor exposure																				■

3.1 Production (1975)

The materials studied in this investigation were specimens of three concrete compositions made in 1975:

- OPC (CEM I) and a w/c ratio of 0.4 (420 kg cement per m^3);
- OPC and w/c 0.54 (300 kg/ m^3) and
- BSFC (Portland blast furnace slag cement, with about 70% slag, CEM III/B LH HS) and w/c 0.4 (420 kg/ m^3).

Originally, the concrete contained a negligible quantity of chloride. Siliceous river sand and gravel (32 mm) were used as aggregate. The specimens were prisms with dimensions of $500 \times 100 \times 100 \text{ mm}^3$, containing three plain 8 mm rebars at 15, 30 and 46 mm cover depth.

3.2 North Sea Exposure (1976-1993)

The characteristics of the concrete after 16 years submersion in the North Sea can be summarised as follows. Except for strong chloride penetration, the concrete showed relatively few changes as a result of the exposure. Chemical analyses showed that penetration of sulphate and magnesium was limited to the outer few millimetres. Compressive strengths at 16 years were found to be between 40 and 80 N/ mm^2 . Chloride penetration was strong: OPC concrete contained a high chloride content throughout the complete cross-section, BSFC contained a high chloride content in the outer 30 mm (see Table 2 for average chloride content). More details have been given in (Polder & Larbi 1995).

Table 2. Main experimental details for Electrochemical Chloride Removal.

	Specimen code	Composition (cement type) (water/cement ratio)			Treatment (39 days)					Average chloride Content (By mass % of cement)	
		OPC 0.4	BFSC 0.4	OPC 0.54	1 A/m ² lime	4 A/m ² lime	1 A/m ² water	0 A/m ² lime	0 A/m ² 20°C 80%RH	before treat- ment	after treat- ment
treated	1	x			x					4.1	2.3
	2		x		x					1.0	0.7
	3			x	x					4.4	2.6
	4	x				x				4.1	1.2
	5		x			x				2.8	0.9
	6			x		x				2.7	0.8
	7	x					x			2.7	1.8
	8		x					x		1.9	1.0
	9			x				x		3.2	1.8
controls	10	x						x		2.5	-
	11		x					x		1.0	-
	12			x				x		2.7	-
	13	x							x	2.5	-
	14		x						x	1.9	-
	15			x					x	3.2	-

- not determined

3.3 Chloride Removal (1993)

After cutting off the top 100 mm of the prisms, electrical contacts were fixed to each of the rebars and the top and bottom surfaces were epoxy coated. Nine prisms were placed in PVC cylinders containing an activated titanium anode mesh and an electrolyte solution (nrs 1 to 9). Three prisms placed in saturated lime but receiving no current served as "wet controls" (nrs 10 to 12). Three prisms were kept in a climate room at 20°C and 80% RH as "dry controls" (nrs 13 to 15). A constant current was passed from galvanostats between the reinforcement bars and the anode of nine prisms for 39 days. The applied current densities were either 1 or 4 A/m² steel surface in saturated calcium hydroxide solution or 1 A/m² in tap water. Table 2 shows the main experimental details. After treatment another 100 mm was cut off and analysed for chloride content. Average chloride contents were found to have been reduced to between 40 and 70 % of the initial contents depending on the amount of charge passed. More details have been given in Polder (1996).

3.4 Outdoor Exposure (1993-1994)

The remaining parts of each prism, with dimensions of about $100 \times 100 \times 200 \text{ mm}^3$, were used for the corrosion investigations. Electrical contacts were fixed to the rebars. The specimens were then exposed outside the TNO laboratory for a period of one year, not sheltered from rain. During this period measurements of steel potential and concrete resistivity were made frequently.

4 Electrochemical measurements

Steel potentials were measured shortly after ECR (November 1993) and several times during the outdoor exposure (until November 1994) using a Ag / AgCl (saturated KCl) reference electrode. The concrete resistivity was measured with a Wenner four electrode probe (spacing 30 mm) using 108 Hz AC. The results are given in Table 3. Compared to non-treated samples (nrs 10 to 15) the corrosion potential has shifted towards less negative values.

Table 3. Steel potential (E_{steel}) vs. Ag / AgCl reference electrode and resistivity (ρ) of reinforced concrete prisms after chloride removal treatment.

	Specimen code	November 1993			November 1994			
		E_{steel} (mV)* Rebar cover		ρ ($\Omega \text{ m}$)	E_{steel} (mV)* Rebar cover			ρ ($\Omega \text{ m}$)
		30 mm	46 mm		15 mm	30 mm	46 mm	
treated	1	35	-130	110	60	-35	50	120
	2	10	-5	990	60	-10	10	1095
	3	0	-25	90	-175	0	-110	105
	4	25	80	110	80	30	40	95
	5	75	5	430	115	45	-45	545
	6	145	140	160	150	165	190	400
	7	75	80	155	140	110	150	170
	8	-70	-125	610	10	-15	-55	1005
	9	55	25	120	45	105	75	125
controls	10	-345	-315	200	-465	-435	-225	155
	11	-330	-330	990	-350	-375	-230	990
	12	-295	-420	160	-455	-470	-560	155
	13	-205	-250	200	-140	-30	-165	385
	14	-110	-330	610	-240	-55	-195	1130
	15	-335	-310	120	-230	-305	-315	160

* 15 mm not measured

5 Chloride redistribution

During the 16 year period of North Sea exposure chloride was able to penetrate into the concrete. The chloride penetration profile after ECR was determined by analysing samples taken from the locations shown in Figure 2. It was found that the local chloride contents had been altered by the chloride removal treatment, in that part of the chloride had been removed from the concrete and part of the chloride had been displaced. As a result, significant chloride concentration gradients were present after removal. In particular, the chloride content adjacent to the rebars was low. This non-equilibrium situation was expected to change during the one year outdoor exposure period due to redistribution of chloride. This could lead to an increase in chloride content at the rebars, which would influence the corrosion state of the rebar. In total, the local chloride content was measured three times:

- (a) after 16 years of North Sea exposure;
- (b) after chloride removal;
- (c) after one year of outdoor exposure.

The effect of chloride removal was inferred from comparing profiles (a) and (b). For redistribution (b) and (c) are compared. Since measurement of the chloride content is inherently destructive, measurements need to be carried out on slices taken from different cross-sections of the same specimen. In the discussion of these results it is assumed that the initial chloride profile and material properties of the three cross-sections taken from the same specimen are identical.

6 Local chloride content determined experimentally

After one year of outdoor exposure, samples were taken for local chloride determination as was done after ECR. In Figure 3 for one prism the ten local samples in the cross section are shown in each of three situations: before ECR, immediately after ECR and after one year of outdoor exposure. The figure shows the general decrease in chloride as a result of ECR (cf. grey bars vs. black bars); furthermore during one year of outdoor exposure redistribution of chloride is seen to take place (cf. white vs. grey bars). Chloride redistribution during outdoor exposure will influence the corrosion state of the reinforcement. The change in local chloride content at the three rebars with respect to the local chloride content immediately after chloride removal treatment is summarised in Table 4.

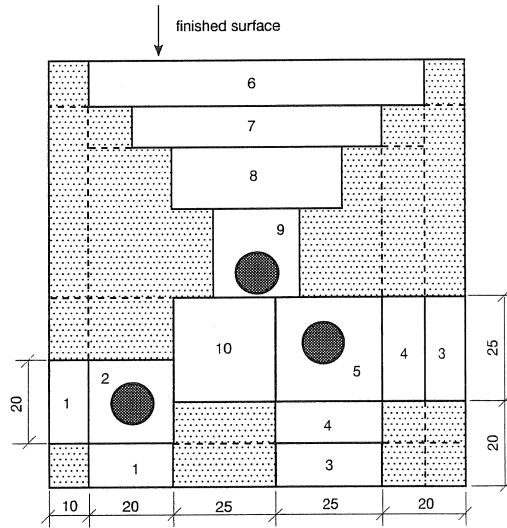


Fig. 2. Layout of sampling for local chloride analysis (cross-section). Dimensions are given in mm.

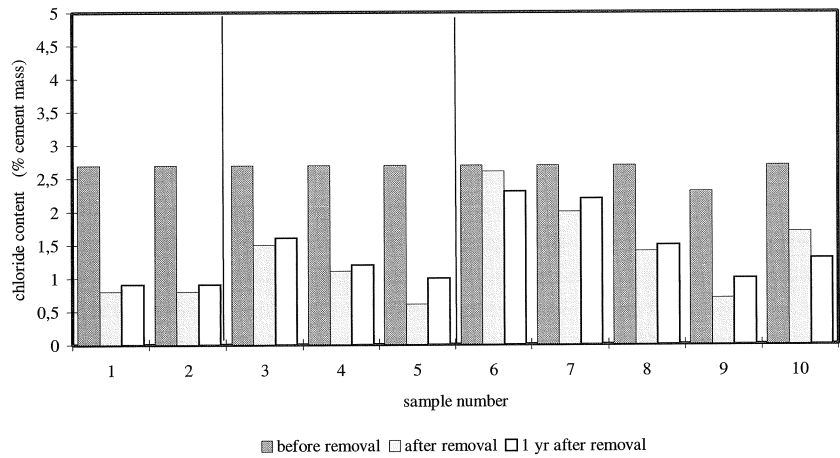


Fig. 3. Local concrete chloride contents in Specimen 7, before, shortly after and one year after ECR; rebars were located at positions 2, 5 and 9.

Table 4. Change of local chloride content due to redistribution during one year outdoor exposure, measured at the location of the rebars.

Specimen code	Apparent change in local chloride content (% by mass of cement) at rebar with cover of		
	15 mm	30 mm	46 mm
1	0.1	0.1	-0.6
2	0.0	0.0	0.9
3	0.3	0.4	0.5
4	0.2	0.2	-0.1
5	0.0	0.0	0.0
6	0.1	0.2	0.1
7	0.1	0.4	0.3
8	0.2	-0.1	-0.2
9	0.1	0.1	0.2

7 Calculated chloride redistribution

In order to predict the development of chloride redistribution over a long period of time from the local chloride content in the specimen after chloride removal treatment the redistribution of chloride was analysed using the finite element package DIANA. The concrete was modelled by the grid shown in Figure 4, which consists of 10×10 elements having four nodes each. It was assumed that the chloride transport could be described by pure diffusion with a constant diffusion coefficient as determined for these materials after North Sea exposure (Polder & Larbi 1995):

- BSFC 0.4 $D = 0.3 \cdot 10^{-12} \text{ m}^2/\text{s}$
- OPC 0.4 $D = 2.0 \cdot 10^{-12} \text{ m}^2/\text{s}$
- OPC 0.54 $D = 3.0 \cdot 10^{-12} \text{ m}^2/\text{s}$

In order to check the sensitivity of the model to an incorrect value of the diffusion coefficient, control calculations were carried out with diffusion coefficients twice as large and twice as small as the values specified above. These resulted in poor matches with the actual chloride distribution after one year.

For the calculations the following initial and boundary conditions were used:

- (1) all external fluxes are 0;
- (2) at $t = 0$ an initial chloride distribution is present; this chloride concentration is dependent on the location in the grid and is derived from the local chloride concentrations determined experimentally in 1993 shortly after extraction;
- (3) the presence of rebars in the concrete is not taken into account (i.e. in the calculations the specimen was assumed to consist only of concrete to which homogeneous transport properties were assigned).

Based on the initial chloride distribution after ECR, a transient analysis was carried out with time steps of 3.15×10^6 s (i.e. 1/10th of a year). Calculated chloride levels were obtained by taking 10 steps for a one year, or 100 steps for a ten year redistribution period. The calculated change in chloride distribution after one year is given for specimen No 2 (bsfc 0.4) in Figure 5 and 6 and for specimen No 3 (opc 0.54) in Figure 7 and 8.

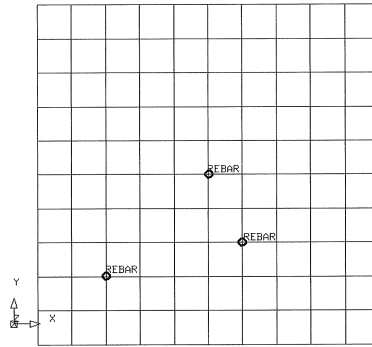


Fig. 4. Finite element grid used for calculation of chloride redistribution.

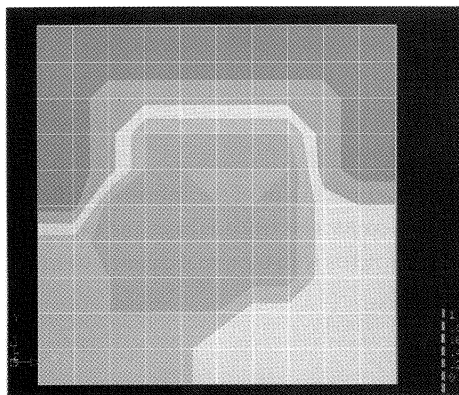


Fig. 5.

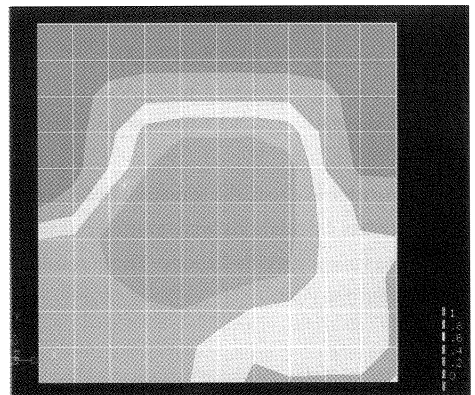


Fig. 6.

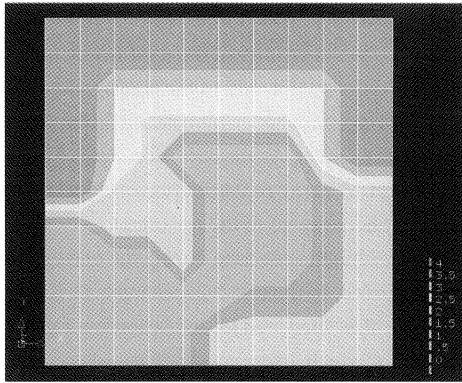


Fig. 7.

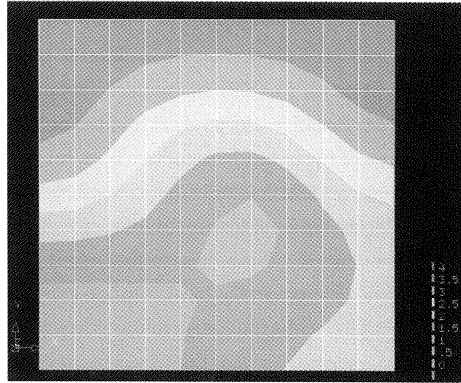


Fig. 8.

8 Discussion

8.1 Corrosion Related Measurements

From the steel potential measurements it is clear that chloride removal treatment is an effective means of shifting the corrosion potential to more positive values. Both dry and wet control specimens (nrs 10 to 15) maintain their negative corrosion potentials. For example in specimen 2, which had an average chloride content of 1.0 % before and 0.7 % after treatment, the steel potential shifted from around -300 mV vs. Ag/AgCl to close to 0 mV (cf. control specimen 11). Even larger shifts were recorded in the case of specimen 6 in which the average chloride content was reduced from 2.7 to 0.8 % and potentials were shifted from between -500 and -300 mV to between $+130$ and $+190$ mV (cf. control specimen 12).

The observed steel potentials can be evaluated with regard to the corrosion activity they indicate using the following empirical criteria. A potential more negative than -260 mV vs. Ag/AgCl is taken to mean that the probability of corrosion is > 90 %. Potentials more positive than -110 mV indicate that there is a probability of > 90 % that the steel is not corroding. Within the intervening interval the corrosion activity is uncertain. These results have been confirmed by visual inspection of steel bars after exposure.

8.2 Measured Chloride Redistribution

The three different types of concrete used in the experiments differ with respect to the mobility of the chloride. The mobility of the chloride can be characterised by the diffusion coefficient (Polder & Larbi 1995). The mobility of chloride in blast furnace slag cement concrete is low, expressed in terms of diffusion coefficient $D = 0.3 \times 10^{-12}$ m²/s. Chloride is more mobile in ordinary portland cement concrete with $D = 2.0 \times 10^{-12}$ m²/s for OPC with a low water to cement ratio of 0.4 and $D = 3.0 \times 10^{-12}$ m²/s for OPC with a relatively high w/c of 0.54). After 16 years North Sea exposure, the OPC 0.54 concrete specimen was almost fully saturated with chloride.

The difference in chloride mobility becomes apparent during the outdoor exposure period after chloride removal treatment. From Figure 3, it can be seen that the chloride removal treatment caused the largest changes in chloride content to occur in the vicinity of the reinforcement (locations 2, 5 and 9). In the more distant parts of the cross-section and in the area between the rebars (location 10), a larger fraction of the initial chloride remained. Chloride redistribution causes some of the chloride to diffuse back towards the reinforcement. As the corrosion state of the reinforcement is mainly determined by the local chloride content, redistribution of chloride after the chloride removal treatment influences the long-term effectiveness of the treatment. From Figure 3 it can be seen that at critical locations within the concrete during the one year period subtle changes in the local chloride content were observed. In the BSFC 0.4 specimens (2, 5, 8) negligible chloride redistribution occurred. In one OPC 0.54 specimen (3) redistribution caused the local chloride level at the rebars to increase to a level at which active corrosion could occur (well over 1% by mass of cement). This was confirmed by the potential measurements.

8.3 *Calculated Chloride Redistribution*

The chloride redistribution process will eventually reduce the beneficial effect of chloride removal treatment, but this effect will be limited since some of the chloride was removed from the specimens. The remaining chloride tends to spread out over the whole cross-section. From the outdoor exposure experiments it was found that within a period of one year, redistribution of the chloride did not have a large influence on the corrosion state of the steel. In order to investigate the effect of chloride redistribution on the long-term effectiveness of the chloride removal treatment, finite element calculations were carried out to assess the chloride redistribution. Results were given after one year and ten years redistribution. The best correlation between calculated and measured local chloride levels at the rebar surface after one year outdoor exposure was found in the OPC 0.54 concrete.

The calculations illustrate that after the chloride removal treatment, differences in the local chloride content throughout the cross-section of the specimen level off gradually. In specimens characterized by a high overall chloride content this process will probably lead to loss of rebar passivation within a period of 10 years.

During exposure over a ten year period, further redistribution of chloride takes place. The increase in the chloride level at the rebars depends on the chloride mobility and the amount of chloride stored in the concrete. BSFC 0.4 concrete is characterised by a low chloride mobility and a low chloride content, hence it has the lowest value of 0.08 % increase in the chloride level at the rebars compared to the chloride level after one year of outdoor exposure. In OPC 0.54 the chloride mobility is higher, and more chloride is present. This induces a calculated increase in the chloride level of between 0.15 to 1.0 % compared to the level after one year at the rebar surface, depending on the amount of remaining chloride. Treatment at high current density left about 0.8% chloride (average over specimen 6). Treatment at low current density (specimens 3 and 9) left about 2% chloride in the concrete. It will be clear that the probability of corrosion in ten years time is lower for specimens 6 than for specimens 3 or 9.

The calculated chloride distributions for a ten year exposure period show that in the blast furnace slag cement concrete, chloride levels at the rebars of 0.6–0.9 % by mass of cement could be expected. In view of the dense microstructure of this concrete, reflected in the high electrical resistivity, these

chloride levels will most probably not cause active corrosion. In the ordinary Portland cement concrete specimens, in which a large amount of chloride remained after treatment, chloride levels at the rebars of up to 2.2 % were calculated. Such chloride levels have a high probability of causing active corrosion, particularly in view of the low concrete resistivities.

9 Conclusions

In previous papers it was shown that electrochemical removal of chloride is an effective means of re-installing corrosion protection of reinforcing steel in concrete structures with penetrated chloride. The treatment may remove 1/3 to 2/3 of the chloride present, depending mainly on the process parameters. After the removal, some chloride remains in the concrete. In our experiments a low chloride content was found at the rebars, whereas near the surface the chloride level was much higher. The durability of chloride removal treatment depends on the rate of redistribution of the remaining chloride and in particular on the chloride content near the rebars in the future.

This paper has shown that chloride redistribution occurs during one year outdoor exposure, causing small but detectable changes in the chloride level at the rebars. These changes were most noticeable in the case of OPC 0.54 concrete. It was assumed that the chloride transport was determined by diffusion. Previously determined chloride diffusion coefficients of the concrete mixes involved were used to simulate chloride redistribution in one year. A reasonable fit was obtained between simulated and experimental redistribution data. Subsequently the chloride redistribution over a ten year period was calculated in order to evaluate the long term effect of the chloride removal process.

The calculations suggest that in BFSC (*w/c*) 0.4 concrete redistribution of chloride will most probably not cause loss of passivation for a period of at least ten years. On the other hand in the OPC concrete corrosion probably will be reactivated due to redistribution of chloride in a ten year period.

The Dutch research committee CUR B62 "Chloride removal and realkalisation of concrete" has adopted the approach described here for determining the durability of chloride removal treatment. Further work was published in (Polder & Van den Hondel 1998).

Acknowledgement

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