

Steel Activation in Concrete Following Interruption of Long Term Cathodic Polarization

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ABSTRACT

A set of partially immersed reinforced concrete columns with mature corrosion patterns was used to demonstrate that corrosion macrocell currents provided “cathodic prevention” to parts of the steel assembly that remained passive even though exposed to high chloride (~ 4.7% of the cement weight) concrete. Depassivation of that steel was observed upon macrocell decoupling.

Keywords: Cathodic Prevention, Polarization, Rebar, Concrete

INTRODUCTION

Reinforcing steel is initially passive in concrete because of the high pH (typically >13) of the pore water [1]. Passivity breaks down when chloride ions penetrate the concrete and reach a critical concentration value C_T (usually expressed as mass of chloride per unit volume of concrete, or as a fraction of the cement content of the concrete) in the immediate surroundings of the steel [2-4]. As in other passivity breakdown phenomena, the value of C_T is expected to be higher if the passive steel is cathodically polarized with respect to the open circuit potential [5,6]. There is ample evidence to that effect from experiments in liquid solutions simulating concrete pore water [5,7-9], but much less data has been reported for actual concrete environments [10-13] where numerous complicating factors may be present (e.g. chloride binding [14,15], occluded cell conditions [16], buffering effects [17], extrapolation to long service times [18]). Direct information regarding passivity breakdown behavior in concrete is important to validate otherwise promising corrosion control procedures such as cathodic prevention, based on cathodically polarizing the steel while still in the passive stage to hinder the initiation of corrosion [19,20].

This paper presents quantitative evidence of substantial hindering of steel depassivation by cathodic polarization imposed over a period of years in a realistic concrete environment with high levels of chloride contamination. The polarization was provided by galvanic coupling to a separate active portion of the steel assembly, in laboratory columns exposed for nine years

to conditions representing marine bridge substructure service. Depassivation of the previously coupled passive steel took place shortly after interruption of the macrocell current. Analysis and implications are presented.

EXPERIMENTAL

Experimental Arrangement and Prior History

Four concrete columns from an ongoing investigation [21] were used in the experiment reported here. (Figure 1). Each column had eleven segments of regular “black” rebar, placed horizontally. Each rebar segment had an exposed surface area of 166 cm². The rebar segments were numbered from #1 (at the top of the column) to #11 (at the bottom).

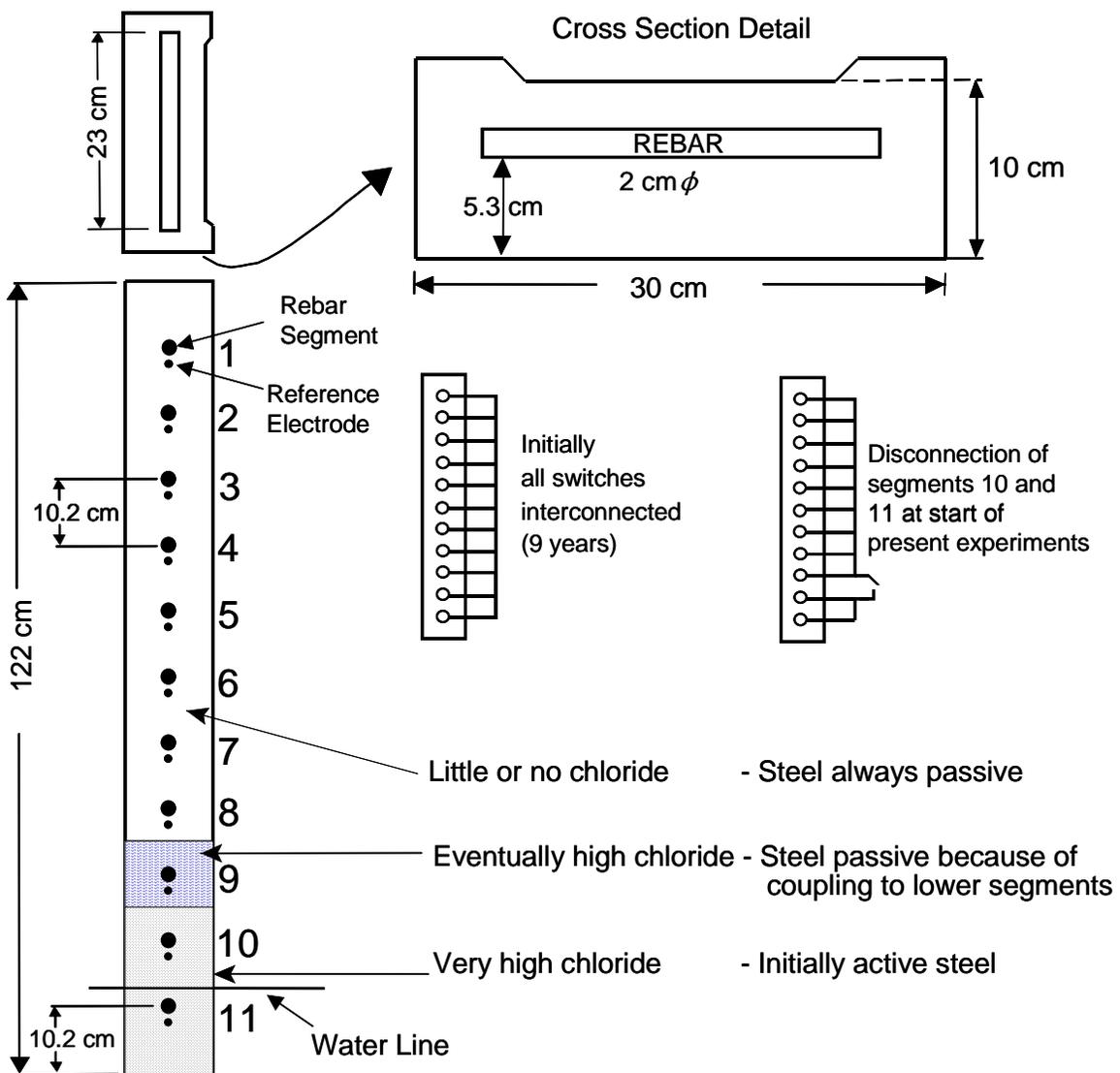


Figure 1. Column Configuration

The as-received steel (0.23 % C) had a high temperature mill scale. The rebars were intentionally pre-rusted by dipping in a 3.5% NaCl solution. An activated titanium reference electrode [22] was placed horizontally about 1.3 cm below the bottom of each rebar. These electrodes were numbered same as the rebar segments, from #1 (at the top of the column) to #11 (at the bottom). Electrical connections to all rebars and reference electrodes were routed to a switch box. During the first nine years of exposure, all rebars were normally electrically interconnected by toggle switches. The switches were numbered from #1 (between rebar segments #1 and #2) to #10 (between rebar segments #10 and #11).

The concrete used to construct the columns had a water-cement ratio of 0.45, 2200 kg/m³ unit weight, 360 kg/m³ Type I Portland cement, and sand as fine aggregate. The coarse aggregate was limestone with maximum size of 1 cm. To accelerate the onset of corrosion at the lower rebar segments, the concrete mix for the lower 25.4 cm of each column had 11.9 Kg/m³ Cl⁻ added, by including the appropriate amount of NaCl. Rebar segments #10 and #11 were in this high chloride zone.

The test columns were moist-cured for five days, and then were exposed to laboratory air for six more days. All the columns were then placed in an exposure tank so that the lower 12.7 cm of the columns were immersed in 5% NaCl solution, placing rebar segment #11 just below the water line. The rebar segments were wired and electrically interconnected by closing the switches shortly afterwards; exposure time was counted from the nominal moment of interconnection. Over the next nine years fresh water and NaCl were added periodically to the tank as needed to maintain the water level and Cl⁻ concentration constant.

All tests were performed at ambient room temperature (typically 22° C ± 2° C) and ambient relative humidity (typically 60% ± 15%). After one year of exposure to room conditions the above water portion of two of the columns (labeled W1 and W2 hereafter) were subject to controlled applications of fresh water leading to water saturation (details in Reference [21]) during the following two years. A plastic sheet covered these columns. Afterward no further wetting was applied to the above-water portion of these two columns but the plastic sheets were left in place for 4 additional years after which the covers were removed. The other two columns (hereafter labeled columns D1 and D2) were directly exposed to room conditions. The columns were nine years old when the experiments described here were started and subsequently conducted over a period of 220 days.

Present Experiments

Test Sequence. The present tests began by interrupting in all columns the connection between the lower two segments (corroding because of the high Cl concentration in the concrete surrounding these two segments) and the rest of the assembly (which was in the passive state), at a moment designated as Day 0. This was achieved by opening Switch #9 while leaving all the other switches closed. The lower 2-segment and the upper 9-segment groups were afterwards allowed to evolve as separately interconnected assemblies. As shown below, spontaneous activation events took place after some time in the lowest segment (#9) of the upper 9-segment group of both W columns (15 and 75 days on W2 and W1 respectively). Shortly after each of those events, Segment # 9 in those columns was disconnected from the rest by opening Switch #8, allowing for independent evolution of the recently activated segment and the remainder of the upper group. No other activation events were observed in any of the four columns through the conclusion of the experiment at Day 220.

Potentials. Static potential (E_s) measurements were regularly performed (using a 200 M Ω input impedance voltmeter) by measuring the potential difference between each rebar segment and the activated titanium reference electrode immediately below. All reported potential values are referred to the saturated calomel electrode (SCE) scale. At periodic intervals any switches closed between segments at the time were momentarily open, and the potential of each internal reference electrode (IRE) was measured with respect to a SCE placed in contact with the solution of the tank. This procedure calibrated the internal reference electrodes while minimizing errors from macrocell-induced IR drops in the concrete. The switches were then returned to their previous settings.

Inter-rebar Segment Current Measurements and Net current densities. Electronic currents circulating between rebar segments joined by a closed switch were measured by momentarily opening the switches one at a time and inserting a low resistance ammeter. These current values are defined here as the macrocell currents (I_{m_j}), where j represents the switch between segments j and $j+1$. I_{m_j} was defined as positive if the electronic current was flowing from the segment(s) below to the segment(s) above the switch.

The net currents associated with each rebar segment were calculated from the I_{m_j} currents obtained at the switches. Thus

$$I_{s_j} = I_{m_j} - I_{m_{j-1}} \quad (1 < j < LS) \quad (1)$$

$$I_{s_1} = I_{m_1} \quad (2)$$

$$I_{s_{LS}} = -I_{m_{(LS-1)}} \quad (3)$$

Where I_{m_j} was the macrocell measured at switch j , I_{s_j} was the net current associated with rebar Segment j , and LS was the number for the lowest rebar segment in an interconnected group. With the sign convention being used, these I_{s_j} currents identify each segment as a net anode when negative or a net cathode when positive. These net currents were converted to net current densities (i_{s_j}) by dividing each I_{s_j} value by the surface area of the rebar segment.

Inter-rebar Segment Concrete Resistance Measurements. Concrete resistance measurements were performed by opening all switches and first connecting the current leads of a Nilsson® Model 400 soil resistance meter to rebar segments #1 and #11, through which the instrument impressed a 97 Hz square wave alternating current. The potential leads were then sequentially connected to pairs of consecutive reference electrodes, and the instrument measured the resistance (R_{ac}) in ohms between these electrodes. The apparent average resistivity, ρ , of the concrete located between any two reference electrodes was calculated by

$$\rho = \frac{R_{ac} * A_{cs}}{d} \quad (4)$$

Where R_{ac} is the resistance between the two electrodes, A_{cs} is the cross-sectional area of the column, and d is the vertical distance between electrodes ($A_{cs}/d = 30$ cm was used).

Chloride Analysis. Columns W1, D1 and D2 were temporarily removed from the tank at Days 90, 600, 600 respectively for chloride analyses at various positions surrounding the rebar (Figure 2). Concrete powder was extracted by drilling. Total chloride content was determined by potentiometric titration.

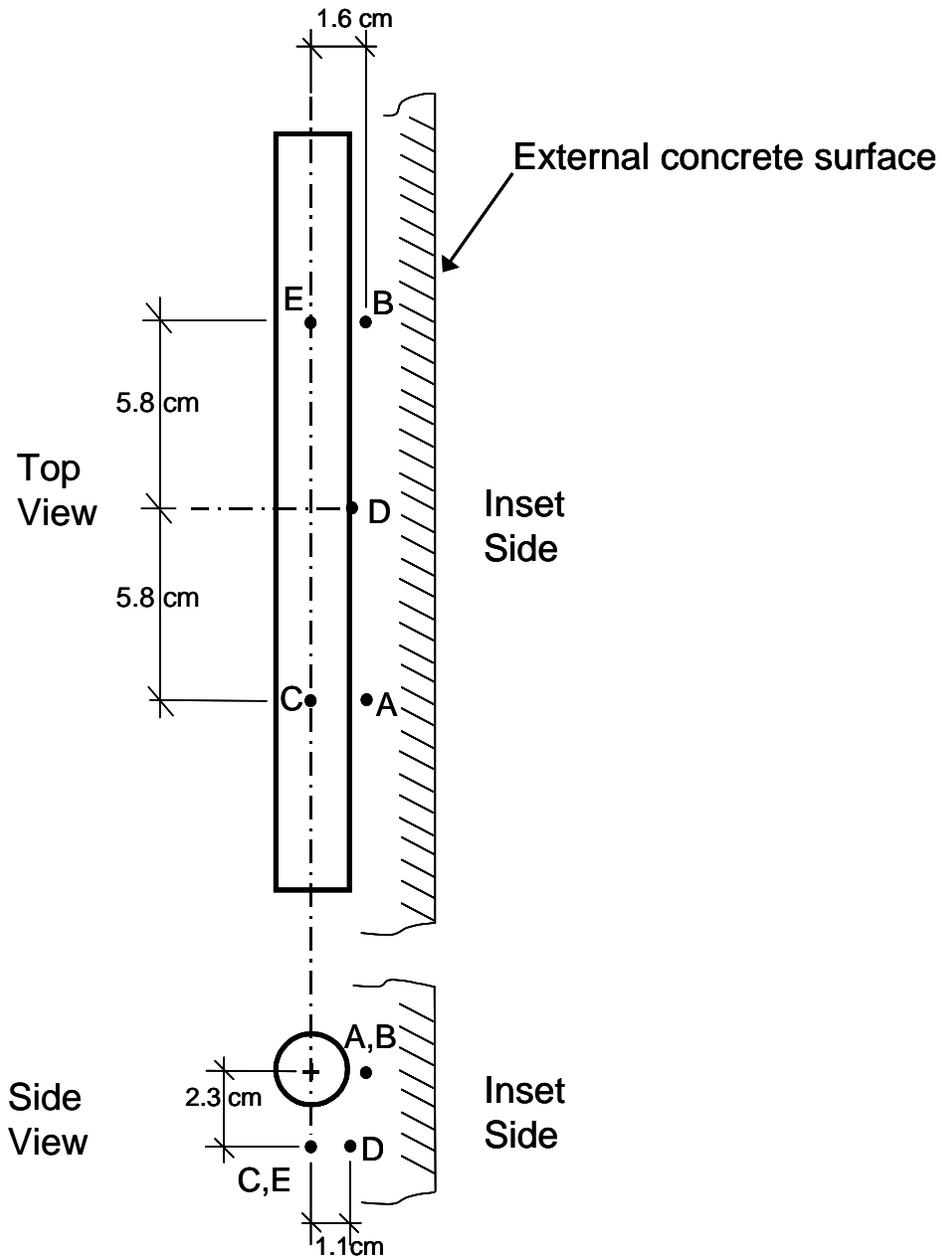


Figure 2. Positions (centers of gravity of the volume extracted) of the concrete powder samples extracted for chloride analysis around rebar Segment #9 (distances referred to the rebar axes).

RESULTS

Chloride Content

The chloride analyses revealed a high concentration close to the surface of rebar segment #9 (Table 1). These values are greatly in excess of the $\sim 1\text{-}2 \text{ kg/m}^3$ typically reported for the critical chloride threshold for steel in concrete under open circuit conditions [4]. As positions A and B were closer to the rebar, the average chloride content at these positions was used to estimate environment severity in the following.

Table 1. Chloride analyses results from the concrete extracted at positions shown in Figure 2.

Position around #9	Column (Cl^- in Kg/m^3)		
	W1	D1	D2
A	9.7	18.6	17.1
B	16.2	20.5	17.7
C	19.0	26.1	
D	21.0		
E	22.7	23.3	20.7

Resistivity

Figure 3 shows typical resistivity values at the different elevations per column set during the time that the experiment was conducted. The resistivity at the position of segment #9 was between $\sim 3 \text{ k}\Omega\text{-cm}$ and $\sim 5 \text{ k}\Omega\text{-cm}$ for the W columns, and between $\sim 3 \text{ k}\Omega\text{-cm}$ and $\sim 20 \text{ k}\Omega\text{-cm}$ for the D columns.

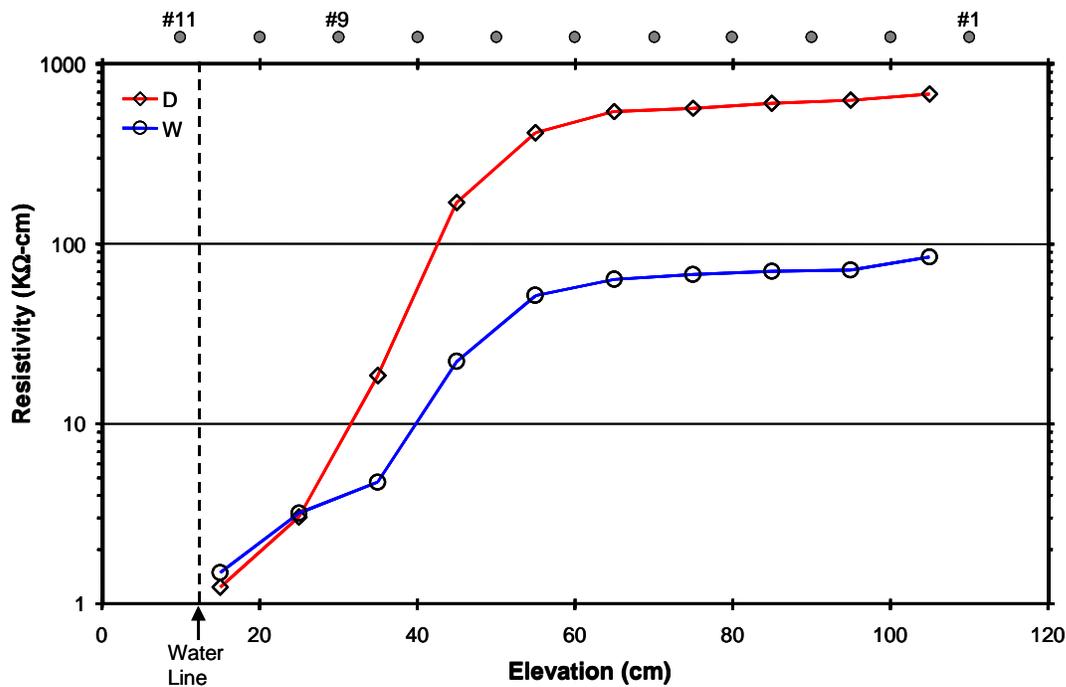


Figure 3. Typical resistivities at the different levels measured during this experiment, averaged by column type. Rebar positions shown at top.

Potential Evolution

Just before Day 0, when all the rebar segments were still interconnected, the potential of rebar segment #9 was ~ -400 mV and ~ -500 mV in column sets W and D respectively. After the disconnection at switch #9 all the upper 9-rebar groups began depolarizing toward more positive potential values which ranged from -80 mV to 10 mV. Spontaneous sharp potential drops, indicative of activation, were observed in both W columns at Day 15 (W2) and Day 75 (W1). Figure 4 illustrates this for Column W1. Seven days after the spontaneous activation was observed, Switch #8 was opened separating rebar segments #1 to #8 from rebar segment #9. The subsequent potential evolution revealed that only segment #9 had experienced activation, drifting over time toward ~ -430 mV and ~ -480 mV in columns W1 and W2 respectively. Segments #1 to #8, interconnected but separated from the rest, continued depolarizing in passive fashion toward terminal values of ~ -35 mV. Neither of the D columns showed signs of spontaneous activation during the 220 day test period, as illustrated for column D2 in Figure 5.

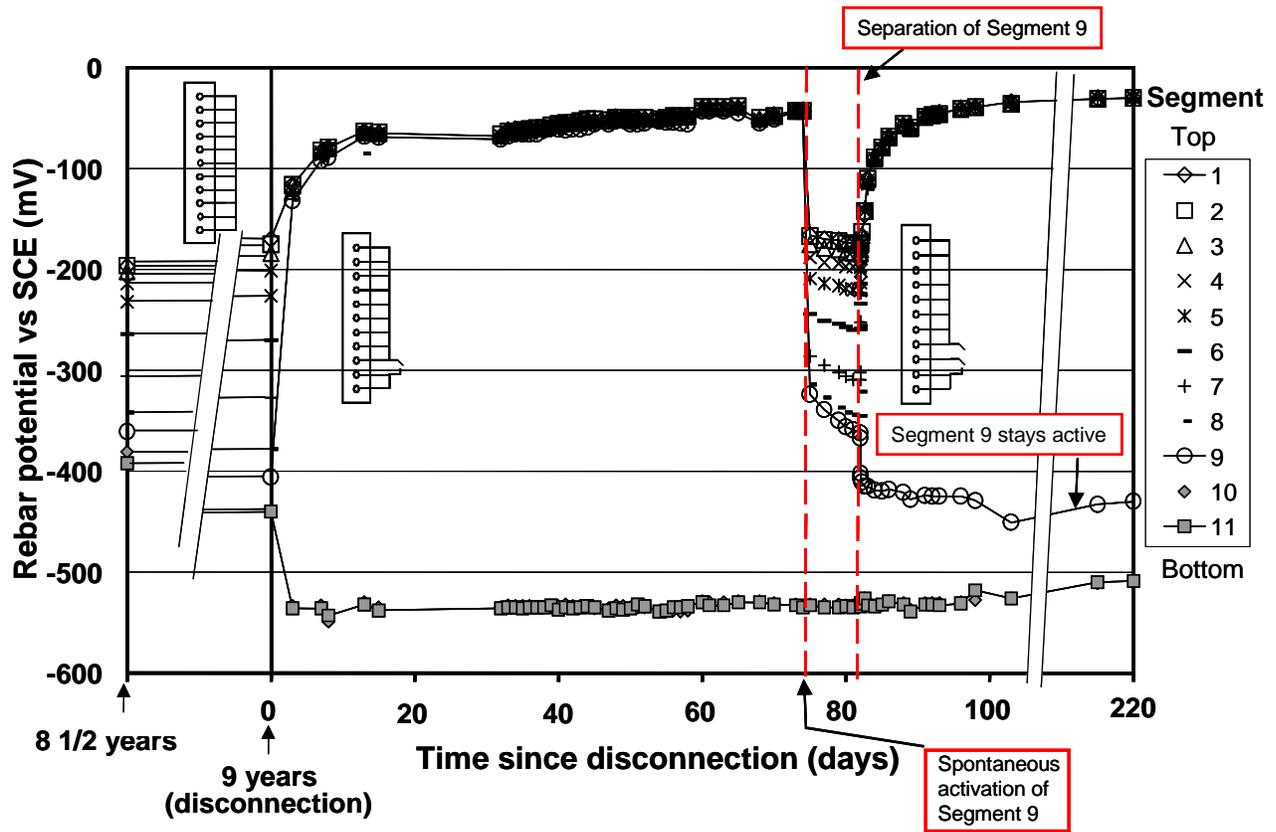


Figure 4. Potential evolution in Column W1, revealing sustained active conditions in segments #10 and #11, initial passive behavior in segments #1 to #9, and spontaneous activation of segment #9 after 75 days.

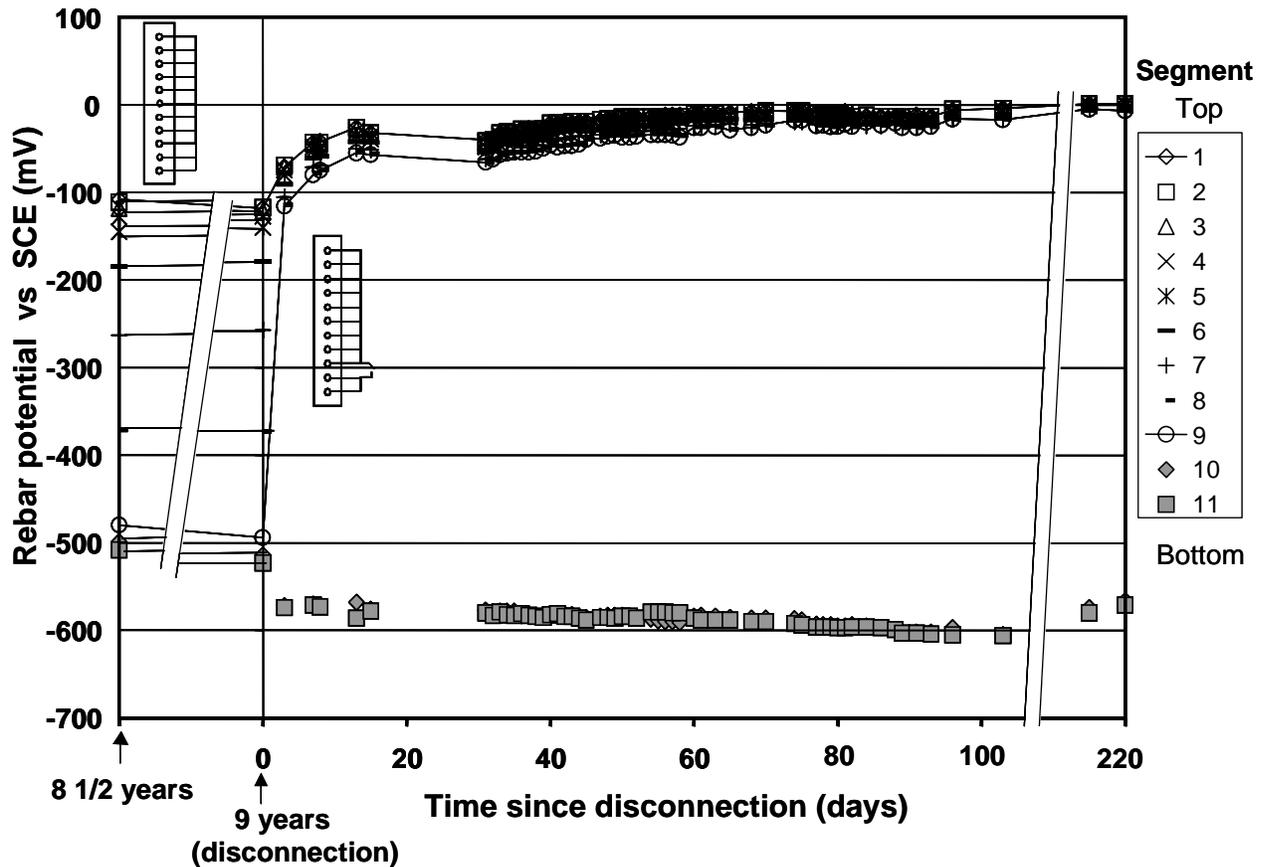


Figure 5. Potential evolution in Column D2, revealing sustained active conditions in segments #10 and #11, passive behavior in segments #1 to #9 during the duration of the test.

Net Currents Densities (i_{s_j})

Just before the beginning of the test, the lowest two segments in all the columns (with the exception of segment #10 in column D1) were net anodes, with i_{s_j} values varying from $-1.5 \mu\text{A}/\text{cm}^2$ to $-0.1 \mu\text{A}/\text{cm}^2$, indicative that most of these segments were corroding appreciably. The remaining segments were net cathodes with current densities that decreased as the distance from the anodes increased. The magnitude of these cathodic current densities explains the significant polarization observed at rebar segment #9 and above as a result of the coupling with the two active rebar segments. This current pattern is illustrated in Figure 6a for all the columns.

After a few days following opening switch #9, the value of i_{s_j} at all passive rebar segments decreased dramatically to absolute values $< 0.002 \mu\text{A}/\text{cm}^2$ and remained so until a spontaneous activation event (if any) took place. The pattern change upon spontaneous activation is illustrated in Figure 6b for the i_{s_j} values measured on column W1 just after rebar Segment #9 became active (and therefore anodic to the rest of the assembly). A short time after isolating rebar segment #9 in columns W1 and W2, the i_{s_j} values returned to values similar or smaller than those registered shortly before this segment became active. At Day 220 (the end of the experiment), the largest i_{s_j} measured was $\sim 0.0009 \mu\text{A}/\text{cm}^2$ at the passive segment groups.

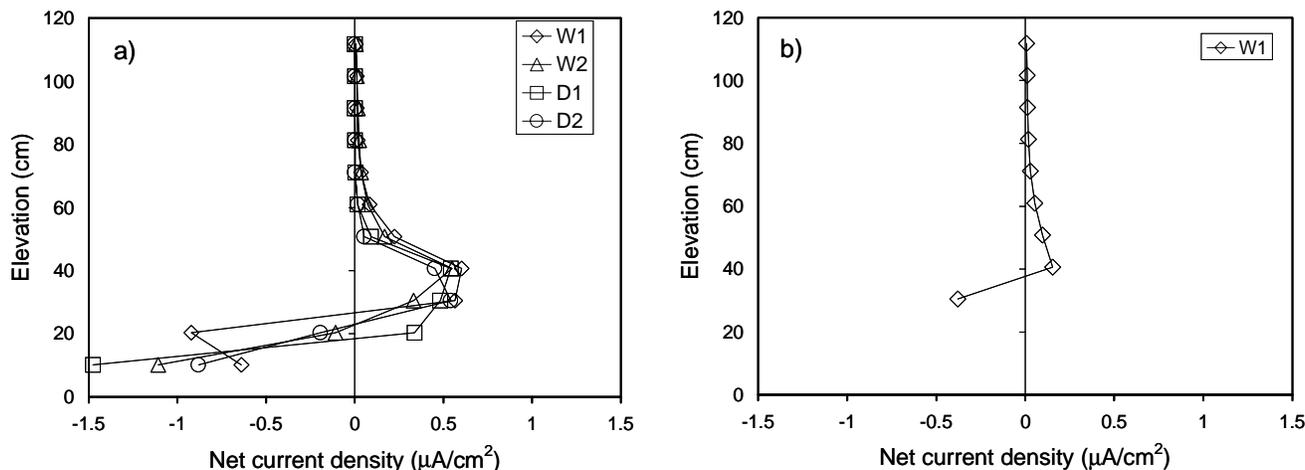


Figure 6. Net current densities (i_{sj}) a) Before starting the test, b) 75 days after disconnection, just after segment #9 was observed to have suffered spontaneous activation on column W1. At that moment Segment #9 was a net anode while segments #1 to #8 were cathodes.

DISCUSSION

Protection and depassivation behavior

The experiments presented here show evidence of protective action, by cathodic polarization of passive steel, in concrete columns that had developed a mature state of polarization over many years. This observation is of particular interest since much currently reported evidence of the expected increase in corrosion threshold by cathodic polarization is derived from short term experiments, often only in liquid test solutions.

The protective condition is inferred from the passive behavior displayed by cathodically polarized (to ~ -350 mV ~ -500 mV) rebar segment #9 in all four columns, in spite of indications of severe chloride contamination of the surrounding concrete. The concrete in the rebar close vicinity (sampling zones A and B, centered at about 6 mm from the rebar surface) had a measured chloride concentration, averaged over the three columns examined, in the order of $17 \text{ kg}/\text{m}^3$ or 4.7 % of the cement content. That amount is greatly in excess of the values (typically $<1\%$ of cement content) commonly quoted in the literature as chloride concentration thresholds (C_T) for steel in concrete at the usual open circuit potential of passive steel (~ 0 mV to -100 mV) [4,23,24]. The initial passive behavior of rebar segment #9 in all four columns was evidenced by drift toward such range of noble potentials upon disconnection of the cathodically polarizing source, and also by the marked reduction in macrocell current between segment #9

and the rest of the passive rebar assembly ^a. The later spontaneous activation of segment #9 in two of the four columns confirmed the aggressive character of the surrounding concrete. In all cases, the cathodic polarization levels of the rebars concerned were achieved with modest current densities in the order of $0.5 \mu\text{A}/\text{cm}^2$.

Cathodic polarization could act to avert the initiation of active corrosion by increasing the actual critical chloride threshold, as commonly observed in localized corrosion phenomena [5,6], but also by beneficially altering the composition of the concrete immediately next to the rebar surface as a result of ionic migration induced by the electric field in the concrete. While segment #9 was still coupled to the active lower segments, the average electric field in the concrete around the rebar surface was in the order of a few mV/cm, as estimated by multiplying the macrocell current density (Figure 6a) by the resistivity at the segment level (Figure 3). The direction of the current indicates that the field pointed toward the rebar surface. There is some evidence [25] that sustained application of a field of this magnitude and direction may fractionally reduce the Cl^- content, and increase the OH^- content, of the pore water at the rebar surface compared with the values expected in the absence of migration effects.

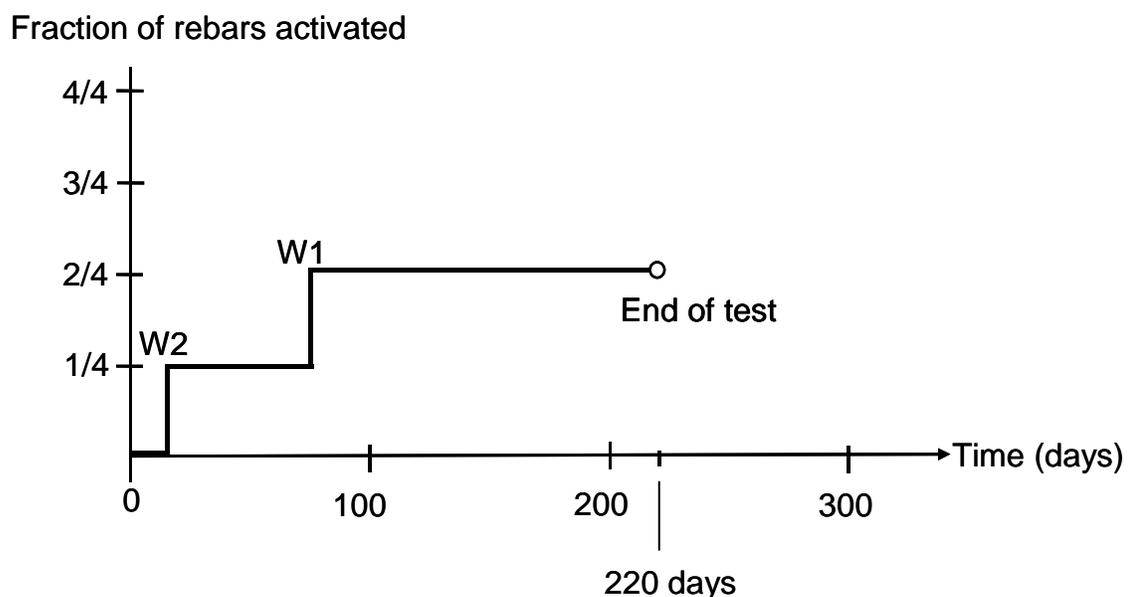


Figure 7. Spontaneous activation sequence of the #9 segments.

^a It should be pointed out that for the pore water pH (~ 13) expected in the concrete used here, iron oxides such as those reported to be responsible for steel passivity (e.g. Fe_2O_3 [26]) are still thermodynamically stable at potentials as low as $\sim -0.8\text{V}$ (SCE). Thus, exposure of the steel to the modest polarized potentials encountered here is not expected by itself to compromise passivity.

Figure 7 summarizes the timing for spontaneous activation events. The results suggest that the #9 segments in the D columns could have also spontaneously depassivated, given enough time beyond the 220 days allotted to this test. The scatter observed may have simply resulted from the stochastic nature of localized corrosion initiation, as documented in many other systems [27]. However, some systematic factors were likely at work. Some of the delay probably reflected the time needed for recovery of the open circuit potential toward noble values (Figures 4 and 5). If the Cl^- and OH^- concentrations at the rebar surface were significantly altered by the applied field as suggested above, some time would pass also while the concentration profiles of those species readjusted toward a more aggressive condition. Such mechanism could account for a greater delay in the initiation of corrosion in the D columns, since the #9 segments there had applied cathodic current densities comparable to those in the W columns (Figure 6) but with greater resistivity in the surrounding concrete (Figure 3) with a consequently larger electric field. Calculations are in progress to estimate the possible extent of this effect.

An additional mitigating factor in the D columns may have been a dryer concrete condition (manifested in the greater resistivity), which is speculated to raise the pH of the pore water [24 pp 22] thus reducing the Cl^-/OH^- ratio compared with the wetter W columns at the same elevation.

Comparison with other systems

A limited number of investigations have studied the relationship between the potential the rebar had shortly before becoming active (E_p) and the C_T value in concrete or mortar. A summary of data reported in the literature together with the present results is shown in Figure 8 in a similar manner as in References [13] and [29]. The specimens in all the cited experiments were carbon steel embedded in mortar or concrete, either polarized (potentiostatically, galvanostatically or from a macrocell) or left free at open circuit potential (OCP), and exposed to a chloride environment in various ways (immersion, cyclic ponding, splash zone or simulated splash zone). The Cl^- and cement contents in each case were either directly reported or inferred from the experimental conditions given by each investigator. For the present experiments, a single average value of 4.7% was used to represent the results of all four columns. The arrow pointing right of some of the symbols indicates that, as in the present experiments, the C_T value at the given potential is a lower bound as the steel never depassivated under those conditions.

There is considerable scatter in the results, partly reflecting the inadequacy of using the chloride/cement ratio as the only measure of environmental aggressivity [28]. As indicated elsewhere, phenomena such as chloride binding prevent observing a unique correlation between free chloride in the pore water and total chloride content [14]. Furthermore, pore water pH is not a function of cement content only [5], and even in simpler water solutions corrosion initiation at a given potential is not dictated by the $[\text{Cl}^-]/[\text{OH}^-]$ ratio alone [5]. These data should be interpreted with caution as the C_T values given usually represent estimated bulk chloride concentrations near, but not immediately at, the steel surface. If migration caused Cl^- depletion and OH^- enrichment at the steel surface, the local C_T value at the moment of activation may have been lower than thought. Such occurrence could create an important distinction between data for open circuit as opposed to polarized conditions; only the

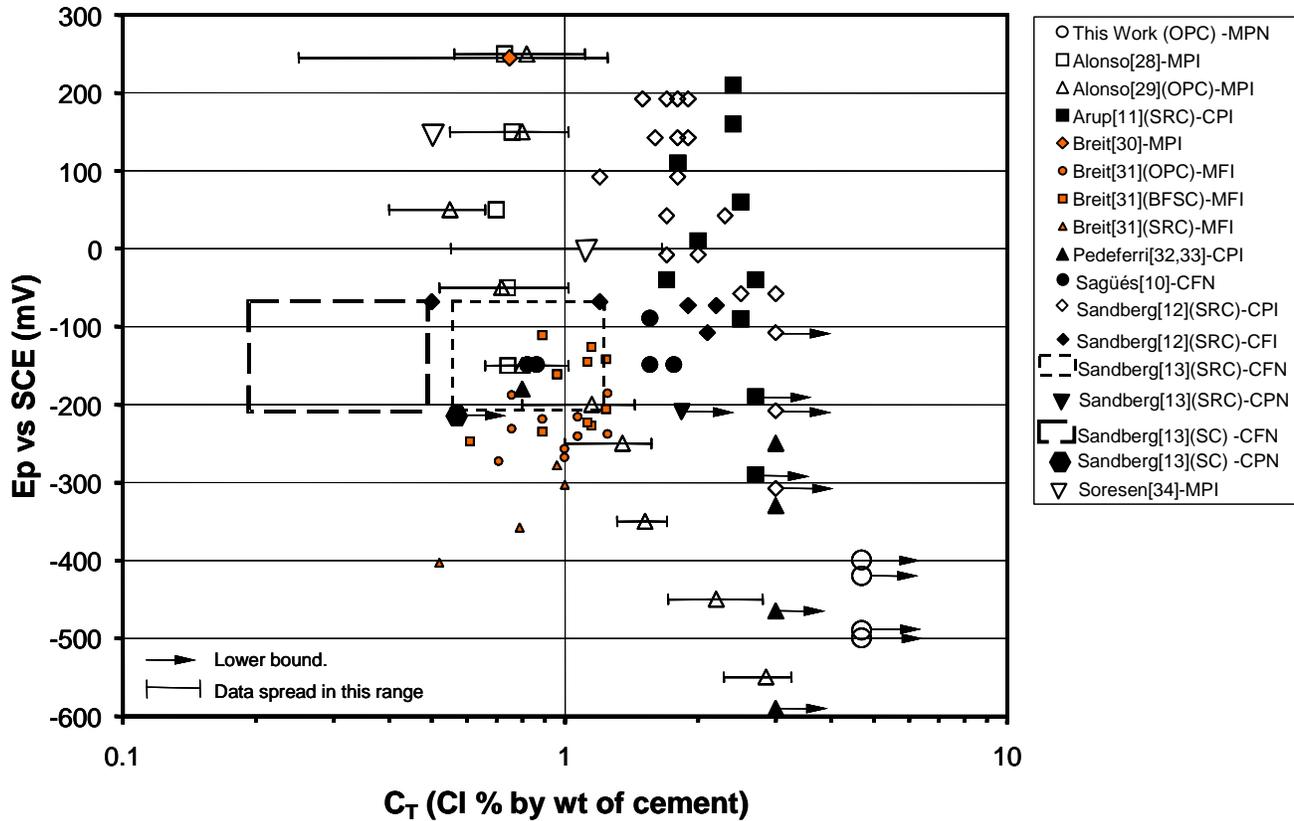


Figure 8. Values of E_p vs. C_T obtained here and reported by other investigators. OPC: Ordinary Portland cement; SRC: Sulfate resistant cement; BFSC : Blast furnace slag cement; SC: slag cement; M: mortar or C: concrete; P : Polarized or F : free open circuit potential; I : Immersed or N : Not Immersed (cyclic or permanent ponding [10], splash zone[13] or simulated splash zone[this work]). Missing designations indicate data not available or multiple conditions.

latter would have net electric fields in the concrete surrounding the specimens. Nevertheless, despite the scatter, the plot suggests that as expected, increasingly greater values of C_T are required to sustain active corrosion at more negative potentials. The present results correspond to the largest C_T value bound, and among the largest cathodic polarization levels, of the reported data set, suggesting that considerable protection (or corrosion prevention used the terminology proposed by Pedferri and others [19,20]) could be achieved by adequate polarization in the passive conditions. The actual extent of polarization from normal open circuit conditions required for a given factor increase in the chloride threshold is still subject to much uncertainty, as evidenced in Figure 8 by the varying trends from different investigators.

CONCLUSIONS

1. Experiments were conducted on columns with mature corrosion patterns. Rebar segments that were cathodically polarized by macrocell currents to potentials of $\sim -350\text{mV}$ to $\sim -500\text{ mV}$ (SCE) for 9 years remained in the passive condition even though the surrounding concrete had reached a chloride content in the order of 17 kg/m^3 ($\sim 4.7\%$ of the cement weight). This level of polarization was produced by a cathodic current density in the order of $0.5\ \mu\text{A/cm}^2$.
2. Spontaneous activation of some of the segments took place after a time delay following interruption of the polarizing macrocell current, confirming the origin of the protective action. The results are supportive of the applicability of the cathodic prevention concept to a mature corrosion system.

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