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Accelerated Chloride Threshold Testing: Part I—ASTM A 615 and A 706 Reinforcement

by David Trejo and Radhakrishna G. Pillai

To evaluate the service-life and life-cycle costs of reinforced concrete structures susceptible to corrosion, quantitative measures of key material properties and parameters must be known. For chloride-induced corrosion, key material properties include the transport rate of the chloride ions (such as diffusivity and sorptivity) in the cementitious material and the critical chloride threshold level of the steel reinforcement in the cementitious material. Critical chloride threshold values for conventional steel reinforcement types have been reported throughout the literature, but no standardized short-term method for evaluating this parameter is currently available. Thus, this paper presents a new methodology for evaluating the critical chloride threshold of steel reinforcement in concrete. Results from tests conducted using the proposed methodology with conventional steel reinforcement embedded in mortar indicate that the method provides a relatively good estimate of the critical chloride threshold level over a relatively short test period.

Keywords: acceleration; corrosion; gradient.

INTRODUCTION

Corrosion of steel reinforcement in reinforced concrete (RC) structures is a significant problem (Koch et al. 2002). Rehabilitation of corroded steel reinforcement in these structures constitutes a major portion of the overall repair needs in the U.S. Thus, owners, designers, material producers, and contractors are considering the potential use of building materials that minimize corrosion of the reinforcement, maximize service life, and optimize life-cycle costs. Both mineral and chemical admixtures in the concrete can delay the onset of corrosion and have proven to be an effective approach in minimizing the impact of corrosion (Maslehuddin et al. 1987; Thomas and Matthews 1993; Ozyildirim 1994). In addition, several reinforcing steels have been developed to resist corrosion when embedded in concrete and exposed to chlorides and other aggressive chemicals.

The implementation of these corrosion-resistant steel reinforcement products has been relatively limited due to the lack of specific quantitative data on the corrosion performance and lack of information on the cost justification and benefits of these products. In addition, realistic corrosion testing in cementitious materials often takes several years to evaluate, thereby further slowing the implementation of these products. A simple, short-term procedure is needed to evaluate the performance of the steel reinforcement embedded in cementitious materials.

Current methods for evaluating the corrosion performance of steel embedded in concrete in the laboratory include ASTM C 876-91 (1999), "Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete," and ASTM G 109-99 (1999), "Test Method for Determining the Effects of Chemical Admixtures on the Corrosion of Embedded Steel Reinforcement in Concrete Exposed to

Chloride Environments." ASTM C 876 (1999) provides guidance on evaluating the half-cell potential of uncoated reinforcement embedded in concrete and provides direction, using the numeric magnitude or potential difference techniques, to identify areas of probable corrosion or time of corrosion activation. This test method, by itself, provides neither direct quantitative data for evaluating the service life of uncracked RC structures nor guidance on making samples for evaluating the corrosion performance. ASTM G 109 (1999) was developed to determine the effect of chemical admixtures on the corrosion rate of reinforcing steel embedded in concrete. This test method by itself can provide quantitative data only on the corrosion rate of steel and directly provides no information on critical chloride threshold values of steel reinforcement in cementitious materials.

To determine the time to first repair of RC structures exposed to chlorides, the time to initiation of corrosion and time of propagation of corrosion must be determined. The time to initiation of corrosion is defined as the time when a RC structure is placed into service until the time when active corrosion of the steel reinforcement begins. This time can be estimated using the transport rate of chloride ions into the concrete towards the steel reinforcement and the minimum chloride ion concentration to change the steel reinforcement from a passive to active corrosion state. This chloride ion concentration is defined as the critical chloride threshold value.

It should be noted that the critical chloride threshold level is not considered to be a unique value for all conditions. This value is dependent on concrete mixture proportions, cement type and constituents, presence of admixtures, environmental factors, steel reinforcement surface conditions, and other factors. Hansson and Sorensen (1988) reported critical chloride threshold values for plain carbon steel based on total chlorides from 0.6 to 1.4% by weight of cement. Hope and Ip (1987) reported critical chloride threshold values based on total chlorides from 0.097 to 0.19% by weight of cement for plain carbon steel. Others have reported different values. Although the critical chloride threshold value is expected to vary when using different materials under different exposure conditions, the variability in these values could be decreased if standardized testing is developed.

The time to initiation of corrosion is dependent on this critical chloride threshold value and other variables, including whether the concrete is cracked or sound. For cracked concrete, the transport rate of the chloride ions is dependent

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ACI member **David Trejo** is an assistant professor of civil engineering at Texas A&M University, College Station, Tex. He is a member of ACI Committees 222, Corrosion of Metals in Concrete; 229, Controlled Low-Strength Materials; 236, Material Science of Concrete; and 365, Service Life Prediction. His research interests include corrosion of steel in cementitious materials, corrosion mechanisms, concrete durability, service-life prediction, and life-cycle costing.

Radhakrishna G. Pillai is a graduate student in the Construction, Geotechnical, and Structures Division of the Civil Engineering Department at Texas A&M University. His research interests include corrosion of reinforcement in concrete and quantifying parameters for predicting service life of reinforced concrete structures.

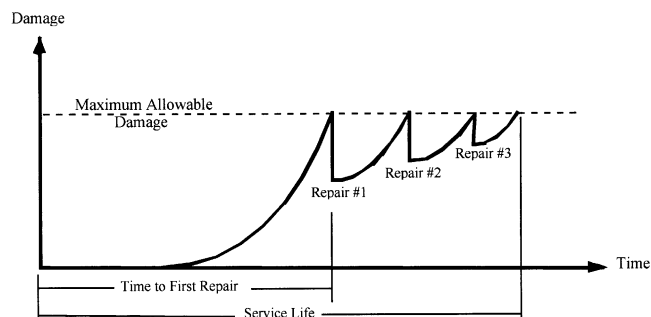


Fig. 1—Time to first repair and service life as function of maximum allowable damage.

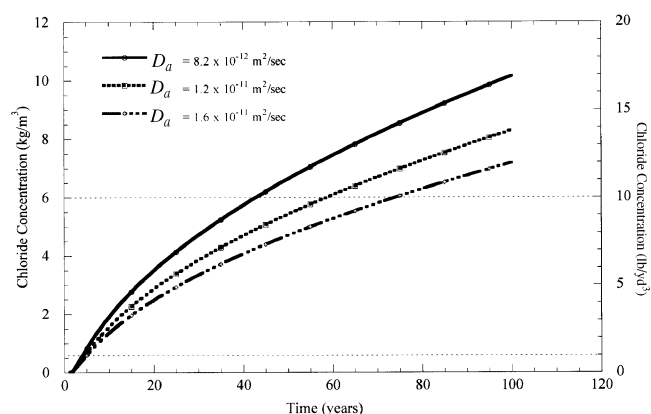


Fig. 2—Critical chloride threshold level versus time to corrosion initiation for uncracked concrete.

on the concrete crack characteristics, the chloride concentration in the solution being applied to the RC structure surface, the number of applications, the temperature, the critical chloride threshold level of the reinforcement, and other parameters. For sound concrete, the transport rate of the chloride ions is dependent on the cementitious material in which the chloride ions are being transported through, the temperature, the chloride ion concentration at the surface, the concrete cover, the critical chloride threshold level of the reinforcement, and other parameters.

In simple terms, the propagation period is defined as the time it takes for the corroding reinforcement to crack and spall the concrete cover. This time period can be estimated with the average corrosion rate of the steel reinforcement and the amount of corrosion required to crack or spall the concrete cover. In general, because the overall service life of an RC structure includes repairs, the time between a repair and the time when the structure degrades to the maximum allowable damage is needed to determine the service life. Figure 1 shows a typical damage versus time curve for an RC structure exposed to chlorides.

The objective of this paper is not to present all methodologies for predicting the transport rate of chloride ions into cementitious materials, but rather to show that the critical chloride threshold value is a key parameter for estimating the service life of RC structures and how this parameter can be evaluated in a relatively short time period. As such, a basic model for predicting the transport rate of chlorides will be presented to show that the chloride threshold is a key parameter. Although many equations are available on chloride migration in cementitious materials, a simple diffusion model will be used to show the importance of the critical chloride threshold value.

If the surface chloride concentration is a function of the square root of time, the following equation can be used to predict the initiation time period

$$C(x, t_i) = k\sqrt{t_i} \left\{ \exp\left(\frac{-x^2}{4D_a t_i}\right) - \left(\frac{x\sqrt{\pi}}{\sqrt{4D_a t_i}}\right) \left(1 - \operatorname{erf}\left(\frac{x}{\sqrt{4D_a t_i}}\right)\right) \right\} \quad (1)$$

If $C(x, t_i)$ is the critical chloride threshold level of the steel reinforcement, D_a is the apparent diffusion coefficient of the cementitious material, x is the cover depth, and k is a constant, the time to initiation of corrosion t_i can be determined using trial and error or by plotting the equation in a dimensionless form.

If it is assumed that the cover x is 50 mm (2 in.), the diffusion coefficient ranges from 8.2×10^{-12} to 1.6×10^{-11} m²/s (1.3×10^{-8} to 2.5×10^{-8} in.²/s), and k is 2, then the concentration $C(x, t_i)$ can be plotted as a function of time t_i as shown in Fig. 2. The figure shows that by changing the critical chloride threshold level from 0.6 to 5.9 kg/m³ (1.0 to 10 lb/yd³), the time to initiation of corrosion in an uncracked RC structure can be increased from approximately 4 to 42 years for an apparent diffusion coefficient of 8.2×10^{-12} m²/s (1.3×10^{-8} in.²/s) and from approximately 5 to 75 years for an apparent diffusion coefficient of 1.6×10^{-11} m²/s (2.5×10^{-8} in.²/s). Figure 2 clearly shows that there can be significant extension in the corrosion initiation phase by using reinforcing steel with higher critical chloride threshold value.

Miller and Darwin (2002) reported that the initiation period for cracked concrete bridge decks in Kansas can be determined using the critical chloride threshold level of the steel reinforcement and the rate of chloride transport into the crack. Modifying their equation to determine time increments in years and converting to SI units, the following equation for the initiation time for cracked concrete was reported

$$\text{Time to Initiation of Corrosion (years)} = \quad (2)$$

$$\frac{\text{Critical Chloride Threshold (kg/m}^3\text{)}}{0.3 \text{ kg/m}^3/\text{yr}}$$

Assuming this relationship is valid for other locations, the increase in the time to initiation of corrosion for two different reinforcing steels is simply the difference in the times to initiation of corrosion. Using the example of the two reinforcing steels with critical chloride threshold levels of

0.6 to 5.9 kg/m³ (1.0 to 10 lb/yd³), the time to initiation of corrosion could be extended from approximately 2 years to 20 years. Thus, increasing the critical chloride threshold level of the steel reinforcement can significantly increase the time to initiation of corrosion for cracked concrete also.

The propagation period can be estimated by determining the average corrosion rate of the steel reinforcement embedded in the cementitious material and the amount of corrosion required to crack or spall the concrete cover. Pfeifer (2000) reported that cracking or spalling will occur when 25 μm (1 mil) of the steel surface corrodes. Using this value and the average corrosion rate, the propagation period can be estimated as follows

$$\text{Propagation Time (years)} = \frac{1 \mu\text{m}}{\text{corrosion rate}(\mu\text{m/yr})} \quad (3)$$

The time to first repair can then be determined by taking the sum of the initiation time and propagation time. The overall service life can then be determined by summing the time to first repair and the times between repairs. Because the critical chloride threshold value of the reinforcement can have a significant influence on the initiation phase and the initiation phase can be a significant portion of the overall service life, a simple, short-term test method should be developed to evaluate this parameter.

RESEARCH SIGNIFICANCE

To optimize the design and construction of RC structures exposed to aggressive corrosive environments, owners, designers, and contractors need methodologies for determining the lowest life-cycle costs. To determine these life-cycle costs, the service life of these structures must be estimated. To estimate the service life, quantitative measures of the transport rate of chloride ions, critical chloride threshold, and corrosion rates are needed. Standardized short-term test methods are not available for determining the critical chloride threshold level of the steel reinforcement embedded in cementitious materials. This paper proposes a new, short-term method for determining the critical chloride threshold of steel reinforcement in cementitious materials.

OVERVIEW OF EXPERIMENTAL PROGRAM

The overall experimental program was performed in three general phases. After the original concept of the test procedure was developed, specific research investigations were performed to define certain variables in the test procedure. Some of these variables investigated were the rate of migration of chloride ions and variability of chloride concentrations at defined depth. After sufficient information was developed to perform the test, a preliminary test program with a limited number of samples was implemented to define the total number of samples needed to meet certain confidence levels in the results. The last phase of the experimental program was to perform the overall test program and procedure defined from the first two phases.

TEST LAYOUT AND PROCEDURE

A new test procedure, the accelerated chloride threshold (ACT) test, has been developed to determine the critical chloride threshold of steel reinforcement in cementitious materials (Trejo and Miller 2002). Because the time to initiate corrosion of the steel reinforcement is dependent on

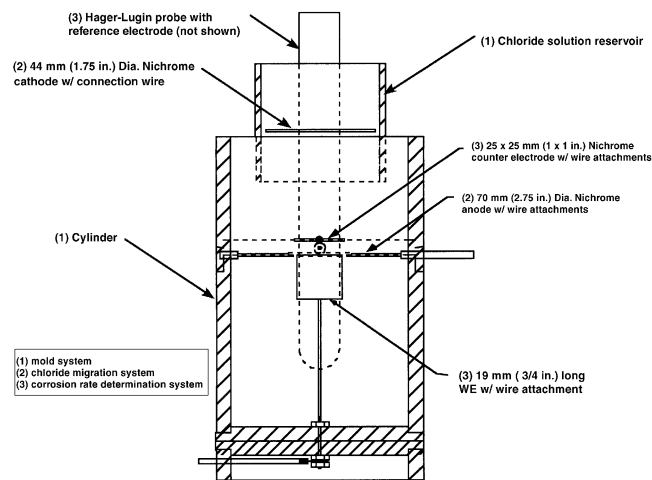


Fig. 3—Layout of ACT test setup.

the rate at which the chloride ions penetrate into the cementitious material, this rate is typically very slow and the time to initiate corrosion of the steel reinforcement embedded in cementitious materials in the field usually takes many years or decades. The ACT test method accelerates the transport of chloride ions towards the steel reinforcement by applying a potential gradient across two electrodes; an anode embedded in the cementitious material at the steel reinforcement level; and a cathode placed in a 50.8 mm (2 in.) diameter reservoir on top of the ACT canister containing a 3.5% chloride ion solution. By applying a potential gradient across these electrodes, negatively charged ions (Cl^- , OH^-) are attracted to the anode where electrons are being liberated. Positively charged ions (Ca^{++} , Na^+) are drawn to the cathode at the top of the ACT canister. By applying this potential gradient, the time required for determining the critical chloride threshold level can be reduced from years to weeks.

It should be noted that in the ACT test method, the unwanted polarization of steel reinforcement during the application of the external potential gradient is minimized by connecting the anode to the ground terminal of the voltage source and by embedding the anode at the same level as the exposed surface of the steel reinforcement. The cathode is connected to the negative terminal of the voltage source. By minimizing the shift in the potential from the open circuit potential (OCP) of the steel reinforcement, changes in the steel surface that could alter the corrosion characteristics of the sample are minimized. Because the pore solution of cementitious materials have high concentrations of hydroxyl ions and these hydroxyl ions can significantly affect the corrosion performance of the steel reinforcement, care must be taken to minimize the influence of the applied potential gradient on the pH of the pore solution.

Figure 3 shows the typical layout for the ACT test canister. The test sample is composed of an acrylonitrile-butylene-styrene (ABS) cylinder used for molding the cementitious material, a chloride transport system for accelerating the chloride ion transport into the cementitious material, and a system for determining the polarization resistance R_p of the steel reinforcement embedded in the cementitious material.

The ABS cylinder is fabricated such that the cementitious material can be placed in the canisters in three separate lifts, allowing each lift to be easily placed and consolidated while at the same time making it easy to install the necessary electrical and electrochemical components of the test system. The

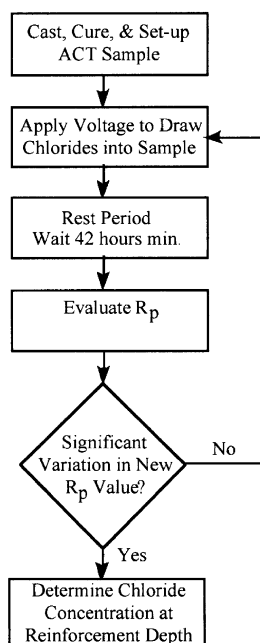


Fig. 4—ACT test procedure.

chloride transport system is composed of the anode, a cathode, and an external time controlled voltage source. The anode is a 69 mm (2.8 in.) diameter nichrome mesh with a 25 x 25 mm (1 x 1 in.) section removed from the center. The cathode is a 44 mm (1.8 in.) diameter nichrome mesh. The corrosion rate system consists of a standard three-electrode corrosion cell system, a working electrode (the sample being evaluated), a 25 x 25 mm (1 x 1 in.) counter electrode, and a saturated calomel reference electrode. Because the resistivity of the cementitious material is typically high and can affect the corrosion rate readings, a Luggin probe was used. The tip of the probe is placed within 2 mm (0.079 in.) of the steel reinforcing bar surface. A frit was attached to the end of the Luggin probe to prevent leakage of the fill solution (0.1% chloride solution) into the cementitious material.

After curing (discussed later), samples are removed from the curing room and placed in the laboratory. Initially, a potential gradient of 20 volts is applied between the anode and cathode for 12 h each day. A 20 V voltage source was used for this purpose. Two steel reinforcing bar types meeting ASTM A 615 and ASTM A 706 specifications were assessed in this research program. The ACT samples with ASTM A 615 steel reinforcement had one 12 h potential gradient applied each day for 5 days. Because the ASTM A 706 exhibited a lower critical chloride threshold value in preliminary tests, the ACT samples containing this reinforcement type had a potential gradient applied for 12 h each day for 4 days. After the final 12 h potential gradient was applied, the samples were allowed to rest for 42 h. The rest period was immediately followed by evaluating the R_p of the embedded steel reinforcement using a potentiostat at a scan rate of 0.0167 mV/s. The scan started approximately -20 mV from the measured open circuit potential OCP and proceeded to approximately +15 mV from the OCP.

It is common to use the Stern-Geary equation to determine the corrosion rate I_{corr} from the R_p as follows

$$I_{corr} = B \left(\frac{I}{R_p} \right) \quad (4)$$

where $R_p = \Delta E / \Delta I$ and $B = (\beta_a \cdot \beta_b) / (2.3 \cdot [\beta_a + \beta_b])$. The values of ΔE and ΔI are obtained from the R_p plot and β_a and β_b represent the slope of the polarization curve near the OCP. Andrade et al. (1986), Liu and Weyers (1997), Yalcyn and Ergun (1996), and Baronio et al. (1996) reported various values for the proportionality constant B . As a result of this, the corrosion initiation can be more reliably identified by only evaluating the inverse R_p values that are directly proportional to the corrosion rate.

Preliminary tests indicated that the inverse of the R_p increased significantly when the steel reinforcing bar shifted from a passive to active corrosion state. A statistical analysis procedure—which involves predicting the inverse R_p value from previous observations, calculating the standard error of the prediction, and determining if the actual value differs significantly from the predicted value—was used to identify if the corrosion rate transferred from a passive to active state. For this research, a t -score of at least 3 is considered to differ significantly. A linear prediction method was used for this study. This procedure for detecting the corrosion initiation avoids the use of assumed proportionality constants B . It should be noted that half cell potential readings may also be applicable for evaluating the onset of corrosion in the ACT samples.

To ensure that the chloride ion concentration at the steel-concrete interface did not excessively exceed the minimum concentration of chloride ions required to initiate corrosion of the steel sample, the test procedure was developed such that the potential gradient between the anode and cathode was applied for only 6 h, followed by at least a 42 h rest period. Prior to applying an additional 6 h of potential gradient to the ACT sample, the R_p of the working electrode is evaluated and, using the inverse of this value, the statistical analysis is conducted. If the inverse of the R_p value deviates significantly from the past corrosion rates, the sample would be considered to be actively corroding and the cementitious material adjacent to the steel reinforcement would be evaluated for chloride ion concentration. A flow diagram for the ACT test procedure is shown in Fig. 4.

After the inverse R_p value indicated that the reinforcement was actively corroding, the specimens are disconnected from the voltage source and the potentiostat, and the chloride ion concentration of the cementitious material directly adjacent to the exposed surface of steel reinforcement is determined. The ACT setup was designed such that the anode is placed at the top level of the steel reinforcement. By striking the sample against a solid surface at the anode level, the sample easily shears along the plane of the anode, which is perpendicular to the longitudinal axis of the canister. Because the anode has a 25 x 25 mm (1 x 1 in.) cutout at the center, the failure plane passes through the transition zone of the steel reinforcement and the cementitious material. An indentation from the steel reinforcement remains in the top section of the cementitious material. This top section is then used for evaluating the chloride ion concentration directly adjacent to the steel reinforcement.

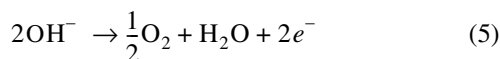
The mortar at the indentation area on the top section is ground using a profile grinder to an average depth of 1 mm (0.04 in.) and a typical diameter of approximately 38 mm (1.21 in.). Note that this mortar is the mortar directly adjacent to the steel sample. This provides a mortar dust sample of approximately 2 g (0.004 lb) of cementitious material. This material is then evaluated for chloride ions using the modified test method for determining total chloride ion content in

concrete using a specific ion probe as outlined in SHRP-S/FR-92-110 (1992). Modifications to the test method were reported by Khan (1998) and later by Peterson (1998), Sohaghpurwala et al. (1998), and Weyers (1998). Because this chloride ion concentration is the chloride ion concentration that initiates corrosion of the steel reinforcement, for this research, it is defined as the critical chloride threshold value of the steel reinforcement.

PRELIMINARY CHLORIDE TRANSPORT RATE AND pH TESTING

The general methodology of the test method is to apply a potential gradient between a cathode and the anode for a set time, stop the test, and at some time later before applying another potential gradient, evaluating the R_p of the steel reinforcement sample. As long as sufficient chloride ions are not available at the steel reinforcement surface, the steel reinforcement will remain passive. As the process of applying a potential gradient continues, more and more chloride ions will be drawn into the cementitious material towards the steel reinforcement. When the chloride ion concentration at the steel-cementitious material interface reaches a certain level, corrosion will initiate. The sample is then removed from further applied potential gradients and the cementitious material directly adjacent to the steel reinforcement surface is evaluated for the chloride ion concentration.

As already noted, because the pore solution has a high concentration of hydroxyl ions, these ions will be drawn towards the anode with the chloride ions. Unlike chloride ions, where oxidation typically does not occur because the half-cell reaction is very noble, the hydroxide ions will oxidize by generating oxygen as follows



Thus, the pH of the pore solution will be a function of the rate at which hydroxyl ions are drawn to the anode and the rate at which they are being oxidized. If the transport rate towards the anode exceeds the rate at which the hydroxyl ions are being oxidized, the pH should increase. If the rate of oxidation exceeds the rate of transport, the pH should decrease. At the same time the hydroxyl ions are being consumed at the anode, cations are being drawn from the anode towards the cathode. This research did not investigate the change in cation concentration at the anode surface. But because the corrosion performance of steel is very dependent on the pH of the electrolyte solution, studies were completed on pore solution pH. It should be noted that the steel reinforcement sample is not being directly polarized and these reactions most likely only occur at the anode-mortar interface.

Because one of the objectives of this work is to develop a short-term test, preliminary investigations were used to determine the rate of chloride ion transport as a function of applied potential gradient. In addition, a preliminary study was performed to investigate the influence of applied potential gradient on the pH of the pore solution at the anode-mortar interface. Figure 5 shows the upper portion of the ACT sample and the expected potential and current lines anticipated when a potential gradient is being applied to the sample. Figure 6 and 7 show the influence of applying 1, 5, 10, 20, and 40 volts in 48-h increments between an anode and a cathode on the chloride concentration and pH of the cemen-

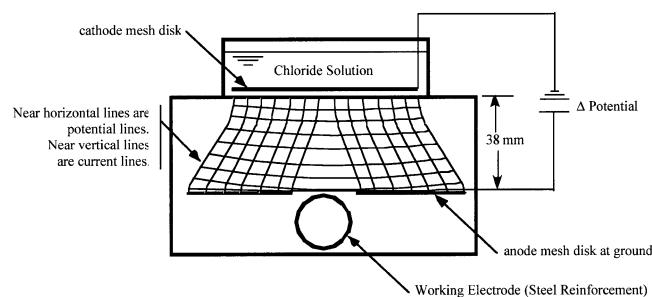


Fig. 5—Potential and current lines in ACT sample.

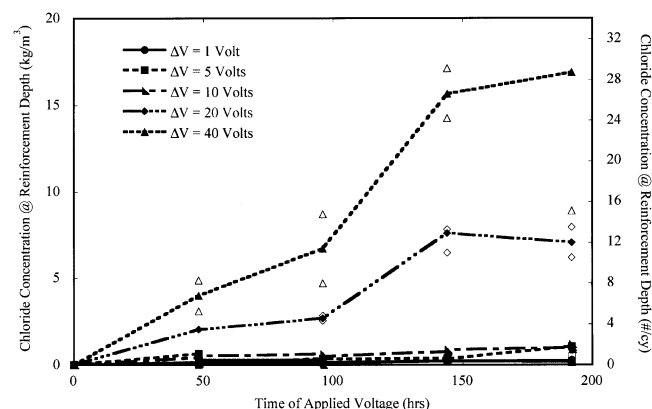


Fig. 6—Chloride concentrations near steel reinforcement surface for different levels of applied voltages.

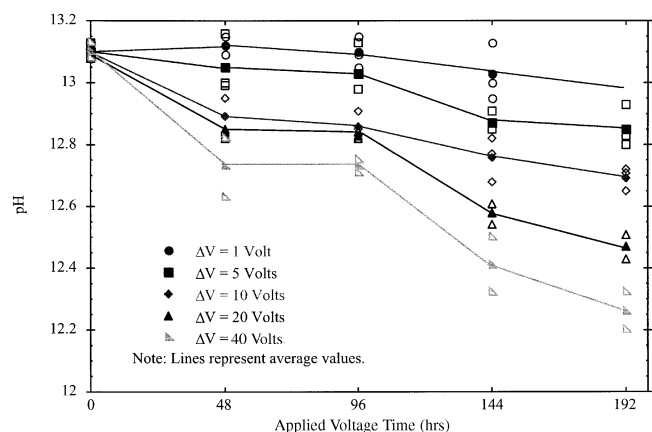


Fig. 7—pH values adjacent steel reinforcement surface for different levels of applied voltages.

titious material within 1 mm (0.04 in.) of the embedded steel reinforcement surface. The chloride and pH samples were obtained from the same ACT samples. The samples for evaluating the chloride ion concentration were taken from the mortar adjacent to the reinforcement. The samples for evaluating the pH were obtained from the mortar adjacent to the anode.

From Figure 6, it is clear that the potential gradients of 1, 5, and 10 volts are not sufficient to significantly increase the rate of chloride ion transport into the cementitious material and decrease the test time period. Figure 7 shows that the pH of the pore solution adjacent to the anode decreases as a function of applied potential gradient level and time, indicating that the oxidation rate of the hydroxyl ions exceeds the rate of transport of hydroxyl ions to the anode. From these data, the

Table 1—Chemical composition of cement (Type I) used

Loss on ignition	SO ₃	SiO ₂	Fe ₂ O ₃	MgO	Al ₂ O ₃	Na ₂ O	CaO	C ₃ S	C ₃ A	Voltage
1.68	2.99	20.78	1.96	1.27	5.24	0.49	64.45	57.95	10.58	0.18

Table 2—Reinforcing steel composition, wt %

Sample	C	Mn	Si	S	P	Cu	Cr	Ni	Mo	V	Fe
ASTM A 615	0.32	0.91	0.23	0.039	0.034	0.51	0.55	0.30	0.09	0.014	Remaining
ASTM A 706	0.23	0.89	0.24	0.040	0.012	0.41	0.18	0.18	0.07	0.032	Remaining

maximum reduction at the reinforcement-mortar interface can be estimated as follows

$$\text{pH} = 13.07 - m \cdot (\text{Total Time of Applied Potential Gradient}) \quad (6)$$

where m is 0.0032 and 0.0042 for applied potential gradients of 20 and 40 volts, respectively. The applied potential gradient of 20 volts would provide a reasonable time for completing the testing and at the same time would reduce the rate of pH reduction by approximately 30% when compared with the results using 40 volts.

MATERIALS AND EXPERIMENTAL DESIGN

The mortar mixture used in the study had a water:cement:sand ratio of 1:2:4.5 and contained distilled water (resistivity = $0.33\text{M}\Omega\text{-cm}^2$), Type I ordinary portland cement (meeting specification ASTM C 150), and Ottawa sand (meeting specification ASTM C 778). The chemical composition of the cement used for testing is shown in Table 1. Samples were cured for 7 days at $32^\circ\text{C} \pm 3^\circ\text{C}$ ($90^\circ\text{F} \pm 5^\circ\text{F}$). The compressive strength of the 75 x 150 mm (3 x 6 in.) cylinders was 11.3 MPa (1640 psi) at 1 day, 33.2 MPa (4800 psi) at 7 days, 37.7 MPa (5475 psi) at 14 days, and 38.6 MPa (5600 psi) at 28 days. The average 28-day permeability of three test samples using ASTM C 1202-97 (1998) was determined to be very high (> 4000 coulombs), as testing was terminated for all samples due to high temperatures.

Two reinforcing steel types were evaluated in the test program: ASTM A 615 and ASTM A 706. Each reinforcing steel type was obtained from the same production lot. The composition of each reinforcing steel is shown in Table 2. The 19 mm (3/4 in.) diameter steel reinforcing samples were cut to 19 mm (3/4 in.) lengths using a lathe. The samples were then drilled and tapped with a 5-40 thread on the circumference at the center (9.5 mm in from the cut end). Care was taken not to disturb the as-received surface conditions. All reinforcing steel samples were then cleaned using ethyl alcohol and all surface area, except 150 mm^2 (0.23 in.^2) opposite the drilled and tapped hole, was coated with two coats of a low viscosity epoxy.

To determine the number of samples that needed to be tested, a statistical experimental design was developed. Because the number of samples required for testing is a function of the standard deviation, mean value, normality of the data, confidence level, and other parameters, three samples of each steel type were initially cast and tested. The mean of these three samples \bar{X}_3 and the standard deviation SD_3 were determined for each steel type and used to provide an initial estimate the number of samples required for the test program. The \bar{X}_3 for the ACT canisters with ASTM A 615 and ASTM A 706 steel reinforcement was 0.74 and 0.33 kg/m^3 (1.24 and 0.55 lb/yd^3), respectively. The SD_3 for the ASTM

A 615 and ASTM A 706 steel reinforcement was 0.53 kg/m^3 (0.9 lb/yd^3) and 0.02 kg/m^3 (0.03 lb/yd^3), respectively.

Because reliable mean and standard deviation values for the critical chloride threshold are not available for ASTM A 615 and A 706 reinforcing steels, the number of samples was first estimated assuming a normal distribution. The research team decided that the maximum allowable half 95% confidence interval length based on the t -distribution should be less than 40% of the \bar{X}_3 values. As such, the first estimate for determining the number of samples based on a normal distribution assumed a half 95% confidence interval length of 35% (to be conservative) of the \bar{X}_3 values and was determined as follows

$$n_o = \left(\frac{(z\text{-value})_{0.025} \cdot SD_3}{0.35 \cdot \bar{X}_3} \right)^2 \quad (7)$$

The z -value is determined from statistical tables. For the ACT canisters with ASTM A 615 steel reinforcement, the first estimate for the number of samples n_o was 17 samples. The n_o for the ACT canisters with ASTM A 706 steel reinforcement was 5. These estimated quantities and degrees of freedom were then used to determine the half 95% confident interval length based on a t -distribution, and the final quantities n were adjusted to obtain a half 95% confident interval length of 40% of the \bar{X}_3 values as follows

$$n = \left(\frac{(t\text{-value})_{(n-1), 0.025} \cdot SD_3}{0.4 \cdot (\bar{X}_3)} \right)^2 \quad (8)$$

Using this equation, it was determined that 20 ACT samples containing ASTM A 615 steel reinforcement and 10 ACT samples containing ASTM A 706 samples should be fabricated and evaluated in the test program.

It should be noted that two of the ACT samples containing ASTM A 615 steel reinforcement were damaged while moving the samples from the curing room to the testing laboratory. A statistical analysis will be performed on the final data in the next section and implications of testing only 18 ACT samples with ASTM A 615 steel reinforcement will be noted.

RESULTS AND DISCUSSION

The R_p values of the steel reinforcing bars embedded in the ACT canisters were determined immediately after curing, 42 h after the last 12-h applied potential gradient increment, and every 48 h thereafter (42 h after the end of the 6-h applied potential gradient increment). The inverse R_p values were plotted as a function of applied potential gradient times as shown in Fig. 8 and 9 for the different types of reinforcement.

The researchers used a statistical analysis of the inverse R_p data to detect when the steel reinforcement transferred from a passive to active corrosion state. This statistical analysis method uses a linear prediction method based on the previously obtained inverse R_p observations to predict a new inverse R_p value. The first predicted value was predicted after four actual inverse R_p values were obtained. If the actual measured inverse R_p value deviates significantly from the predicted inverse R_p value, the sample would be assumed to be actively corroding. Increasing significantly in this case is defined as exhibiting a t -score of at least 3. One sample exhibited a significant increase in the inverse R_p value before four measurements were obtained. This sample was assumed

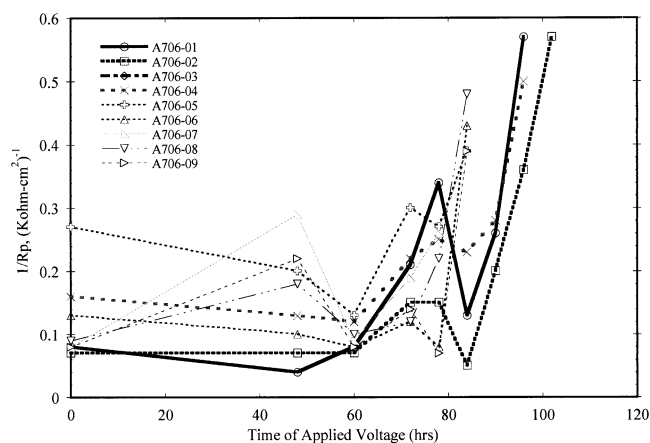


Fig. 8—Inverse R_p values for ASTM A 706 ACT samples as function of applied voltage time.

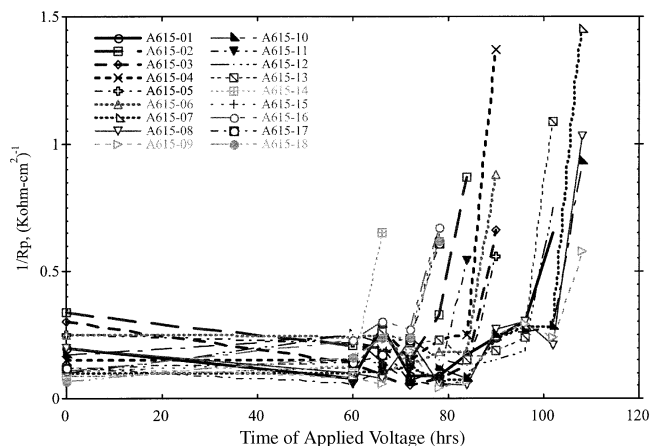
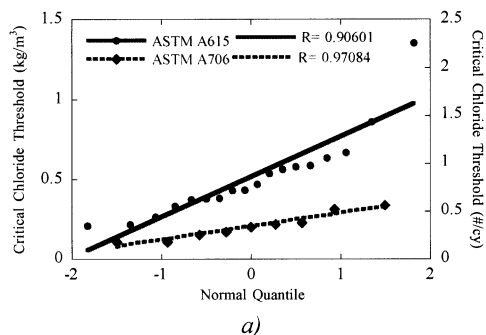


Fig. 9—Inverse R_p values for ASTM A 615 ACT samples as function of applied voltage time.



to be actively corroding. One ACT sample containing A 615 reinforcement showed a significant increase in the inverse R_p value after only 66 h of applied potential gradient.

After it was determined that the steel reinforcement in the ACT canisters was actively corroding, the samples were sheared at the anode level, the mortar adjacent to the reinforcement was ground, and this mortar dust was evaluated for chloride ion concentration. Table 3 and 4 show the critical chloride ion thresholds for the samples. Because the pH of the mortar adjacent to the steel surface may have changed as a result of applying the potential gradient, chloride ion threshold values are reported as both kg/m^3 (lb/yd^3) and $[\text{Cl}^-]/[\text{OH}^-]$.

To perform a statistical analysis of the critical chloride threshold, a normal distribution of the data was assumed. This assumption was then checked using normal quantile plots of the data. Figure 10(a) shows the normal quantile plot used to determine whether the normal distribution is appropriate for the critical chloride threshold values. The quantile plot assuming normal distribution for the ACT canisters containing ASTM A 615 steel reinforcement does not

Table 3—Critical chloride concentration value for ASTM A 615 steel

Sample identification	Critical chloride concentration threshold value			
	kg/m^3 (lb/yd^3)	Weight % cement	$[\text{Cl}^-]/[\text{OH}^-]$	Modified $[\text{Cl}^-]/[\text{OH}^-]^*$
A 615-01	0.5 (0.8)	0.08	0.11	0.24
A 615-02	0.4 (0.6)	0.07	0.09	0.17
A 615-03	1.4 (2.3)	0.24	0.32	0.62
A 615-04	0.6 (0.9)	0.10	0.13	0.26
A 615-05	0.3 (0.4)	0.05	0.06	0.12
A 615-06	0.9 (1.5)	0.15	0.20	0.40
A 615-07	0.6 (1.0)	0.10	0.14	0.30
A 615-08	0.5 (0.9)	0.10	0.13	0.28
A 615-09	0.6 (1.1)	0.11	0.15	0.33
A 615-10	0.3 (0.6)	0.06	0.08	0.17
A 615-11	0.4 (0.7)	0.08	0.10	0.19
A 615-12	0.6 (1.0)	0.10	0.14	0.30
A 615-13	0.4 (0.7)	0.08	0.10	0.22
A 615-14	0.2 (0.4)	0.04	0.05	0.08
A 615-15	0.2 (0.3)	0.04	0.05	0.09
A 615-16	0.4 (0.6)	0.07	0.09	0.16
A 615-17	0.7 (1.1)	0.12	0.16	0.28
A 615-18	0.4 (0.6)	0.07	0.09	0.16
Average	0.5 (0.9)	0.09	0.12	0.24
Standard deviation	0.3 (0.5)	0.05	0.06	0.13

*pH modified per Eq. (6).

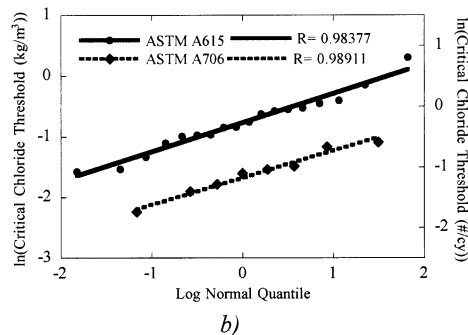


Fig. 10—Normal and lognormal quantile plots.

Table 4—Critical chloride threshold value for ASTM A 706 steel

Sample identification	Critical chloride concentration threshold value			
	kg/m ³ (lb/yd ³)	Weight % cement	[Cl ⁻]/[OH ⁻]	Modified [Cl ⁻]/[OH ⁻]*
A 706-01	0.3 (0.5)	0.06	0.07	0.15
A 706-02	0.3 (0.6)	0.06	0.08	0.17
A 706-03	0.2 (0.3)	0.03	0.04	0.08
A 706-04	0.1 (0.2)	0.02	0.03	0.05
A 706-05	0.2 (0.3)	0.04	0.05	0.09
A 706-06	0.2 (0.4)	0.04	0.05	0.10
A 706-07	0.2 (0.4)	0.04	0.05	0.09
A 706-08	0.1 (0.3)	0.03	0.04	0.07
A 706-09	0.1 (0.2)	0.02	0.03	0.05
Average	0.2 (0.3)	0.04	0.05	0.09
Standard deviation	0.1 (0.1)	0.01	0.02	0.04

* pH modified per Eq. (6).

exhibit a linear trend, and thus the assumption of normal distribution is incorrect. Therefore, a lognormal quantile plot was developed and evaluated for linearity. The lognormal quantile plot shown in Fig. 10(b) shows a linear trend, indicating that data exhibits a lognormal distribution. Transforming the data changed the Shapiro-Wilkinson test for normality *p*-value from 0.005 to 0.836 for the ACT canisters containing ASTM A 615 reinforcement and from 0.450 to 0.574 for the ACT canisters containing ASTM A 706 reinforcement (a value greater than 0.05 indicates that the assumed distribution is good).

Using lognormal distributions, a statistical analysis of all the data was completed. The data from the ACT tests indicate that the steel reinforcement tested in this program meeting ASTM A 615 specifications exhibits a mean critical chloride threshold level of 0.52 kg/m³ (0.87 lb/yd³). Using a 95% confidence level for this steel reinforcement, the critical chloride threshold values would range from 0.21 to 1.36 kg/m³ (0.35 to 2.29 lb/yd³). The ASTM A 706 steel reinforcement evaluated in this research exhibited a mean critical chloride threshold level of 0.20 kg/m³ (0.34 lb/yd³), with a 95% confidence range from 0.11 to 0.34 kg/m³ (0.18 to 0.57 lb/yd³). The range of the lower to upper values indicates a 95% probability that the critical chloride threshold for the reinforcing steels evaluated will be in this range. These critical chloride threshold values are near the lower end of data reported in the literature but the value for the ASTM A 615 reinforcement is similar to that used by state highway agencies.

The results indicate that the mean time of applied potential gradient to activate the ACT samples containing ASTM A 615 reinforcement was 91.3 h with a standard deviation of 13.1 h. The mean time of applied potential gradient for the ASTM A 706 steel reinforcement was 88.7 h with a standard deviation of 7.2 h. Assuming that 60 h of the potential gradient could be applied during the first week (12 h each day for 5 days) and then 12 h could be applied each week (assumes that work will not be done on weekends), the test time for the ACT samples with ASTM A 615 steel reinforcement would take less than 5 weeks. For the ACT samples containing ASTM A 706 steel reinforcement where the potential gradient is applied for 12 h each day for 4 days, the test time would be approximately 5 weeks. Including sample preparation, casting, and curing, the testing could be completed in approximately 7 weeks.

For the materials and test methods used in this study, the ACT results indicate that ASTM A 615 steel reinforcement exhibits a higher critical chloride threshold than the ASTM A 706 steel reinforcement. Additional longer-term data is needed to validate this test procedure. This testing is currently underway. In general, the results of the testing are representative only of the steel from the heats tested and the mortar in which the reinforcements were embedded. But, if the production process and steel composition are similar, these values could be representative of products manufactured at the particular mill where the steel was produced.

It should be noted that changes in steel composition and manufacturing processes can affect the corrosion performance and, in particular, the critical chloride threshold of steel reinforcement in concrete. Different cementitious materials can also influence the critical chloride threshold level. As such, steel reinforcement products produced with different compositions and processes embedded in different types of cementitious materials could be expected to exhibit different critical chloride threshold values than those reported. Further testing of other steel reinforcing products in different cementitious materials is needed to determine the critical chloride threshold values for a wider range of materials. These products should include both the evaluation of different types of steel reinforcement and cementitious products.

CONCLUSIONS

An accelerated standard test methodology has been presented to evaluate the critical chloride threshold value of steel reinforcement embedded in a 1:2:4.5 (water:cement:sand) portland cement mortar. The critical chloride threshold value with a 95% confidence level for ASTM A 615 steel reinforcement in this mortar was determined to range from 0.30 to 0.71 kg/m³ (0.51 to 1.20 lb/yd³) with a mean value of 0.52 kg/m³ (0.87 lb/yd³). Reinforcement meeting the requirements of ASTM A 706 in the same mortar exhibited a mean critical chloride threshold of 0.20 kg/m³ (0.34 lb/yd³) with a 95% confidence range from 0.15 to 0.24 kg/m³ (0.25 to 0.40 lb/yd³). These critical chloride threshold values are near the lower end of data reported in the literature. To determine these values, a total test time, including fabrication and curing of the ACT specimens, would take approximately 7 weeks using the proposed ACT test procedure. It is believed that the proposed test method can be extended to evaluate the critical chloride threshold value of any steel reinforcing type embedded in any cementitious material.

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