See discussions, stats, and author profiles for this publication at: https://www.researchgate.net/publication/287535451

Accelerated chloride threshold testing - Part II: Corrosion-resistant reinforcement

Article in Aci Materials Journal · January 2004

CITATIONS		READS	
47		338	
2 author	z:		
6	David Trejo Oregon State University		Radhakrishna Pillai
			Indian Institute of Technology Madras
	124 PUBLICATIONS 1,008 CITATIONS		125 PUBLICATIONS 457 CITATIONS
	SEE PROFILE		SEE PROFILE
Some of	the authors of this publication are also working on these related projects:		

Project Online NPTEL Course on Maintenance and Repair of Concrete Structures View project

Bond performance of pre-tensioned concrete system View project

Accelerated Chloride Threshold Testing—Part II: **Corrosion-Resistant Reinforcement**

by David Trejo and Radhakrishna G. Pillai

Test methods to evaluate the corrosion performance of reinforcing steel have been typically developed to evaluate the corrosion rate of conventional plain carbon steel in cementitious materials. Although the corrosion rate is a necessary parameter for predicting the service life of reinforced concrete structures, these data alone are not sufficient to compare corrosion performance or to predict service life. Steel reinforcement is now being specifically produced to resist corrosion initiation and this resistance is mainly obtained by increasing the critical chloride threshold levels of the steels. A newly developed test method, the accelerated chloride threshold (ACT) test, was developed and used to evaluate the corrosion performance of three corrosion-resistant reinforcing bars embedded in mortar. Results from this test indicate critical chloride thresholds for microcomposite, 304 stainless steel, and 316LN stainless steel reinforcement is 4.6, 5.0, and 10.8 kg/m³ (7.7, 8.5, and 18.1 lb/yd^3) and based on the mortar unit weight.

Keywords: acceleration; corrosion; gradient; steel.

INTRODUCTION

Preventing and controlling the corrosion of steel reinforcement in reinforced concrete (RC) structures is a challenge facing engineers, designers, and owners. New materials and rehabilitation methods are being developed at a steady rate for structures exposed to aggressive corrosion conditions (Trejo et al. 1994; Darwin et al. 1995; Trejo et al. 2000; Leng, Feng, and Lu 2000; and Castellote, Andrade, and Alonso 2000). The difficulty in implementing these new materials is the lack of standardized, quantitative, corrosion performance data for these systems. This lack of reliable, quantitative, corrosion performance data for steel reinforcement in cementitious materials is a direct result of there being no short-term standardized test methods to realistically evaluate the corrosion performance of these systems.

When comparing the corrosion performance and service-life of different RC systems exposed to chlorides, the transport rate of the chloride ions into the cementitious material, the critical chloride threshold level of the steel reinforcement, and the reinforcement cover are determined by the engineer to optimize the corrosion performance of the RC system. The environment and chloride ion surface loading rate are dependent on where the structure is to be constructed and typically the engineer has limited control of these variables. To predict overall service life, a repair strategy can be assumed. For very aggressive conditions, the engineer should use materials and construction practices that limit cracking of the concrete cover, reduce the diffusion coefficient of the concrete, increase the concrete cover, and use a steel reinforcement with a high critical chloride threshold level.

A significant amount of research has been reported on how mineral and chemical admixtures can affect the corrosion performance and transport rate of chloride ions into concrete and these materials are needed for constructing corrosionresistant structures (Sivasundaram, Carette, and Malhotra 1991; Li, Peng, and Ma 1999; Malhotra, Zhang, and Leaman 2000; Bleszynski et al. 2002). Luping and Nilsson (1993); Ammar, Loche, and Dumargue (1998); and MacDonald and Northwood (2000) all proposed accelerated methods for predicting the transport rate of chloride ions into cementitious materials. These methods can be used with the critical chloride threshold values to estimate the time from when the structure is placed into service until the time when the steel reinforcement begins to corrode.

A significant amount of research has also been reported on the corrosion rates of steel reinforcement in cementitious materials. The effect of cement composition (Rasheeduzzafar et al. 1990), mineral admixtures (Hope and Ip 1987; Al-Amoudi et al. 1993; Pal, Mukherjee, and Pathak 2002; Andióna et al. 2001), material composition (Lorentz and French 1995), loading conditions (Yoon et al. 2000), and environmental conditions (Pech-Canul and Castro 2002, Balabanic, Bicanic, and Durekovic 1996) have been investigated and reported throughout the literature. This information can be used as guidance for designers and engineers to estimate the time from corrosion initiation to the time of cracking or spalling.

Zemajtis and Weyers (1995) completed a comprehensive report on the performance of repair methods and reported that concrete deck patches can last from 4 to 10 years. Chamberlin and Weyers (1991) reported findings from a questionnaire and literature search and concluded that concrete overlays can last from about 10 to 25 years, depending on the strategy used. Thus, information, data, and test methodologies are available for predicting the transport rate of chloride ions into cementitious materials, estimating the corrosion rate of the reinforcement embedded in various materials and exposed to various conditions, and estimating the performance of concrete repairs and overlays.

Information on the critical chloride threshold level is available in the literature, but results are mostly limited to conventional plain carbon reinforcing steel. Because of the long test durations required to determine critical chloride threshold values in cementitious materials, critical chloride threshold data for corrosion-resistant steel reinforcements are mostly only available from solution testing, which typically does not represent realistic values obtained in cementitious materials. Bertolini et al. (1996) evaluated the critical chloride

ACI Materials Journal, V. 101, No. 1, January-February 2004.

ACI Materials Journal, V. 101, No. 1, January-reordary 2004. MS No. 03-011 received January 8, 2003, and reviewed under Institute publication policies. Copyright © 2004, American Concrete Institute. All rights reserved, including the making of copies unless permission is obtained from the copyright proprietors. Pertinent discussion including authors' closure, if any, will be published in the November-December 2004 ACI Materials Journal if the discussion is received by August 1, 2004.

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—Variation in times to corrosion for reinforcing steels with different critical chloride threshold values.

threshold of steel in a simulated concrete pore solution and reported that the critical chloride ion-hydroxyl ratio was greater than 8 at a pH of 13.9. Cui, Sagüés, and Powers (2001) reported that a 316LN stainless steel clad reinforcement was passive up to a chloride concentration of 5 weight % in a saturated Ca(OH)₂ solution with a pH of approximately 12.6 ([Cl⁻]/[OH⁻] ~ 35). Hurley and Scully (2002) evaluated the critical chloride threshold of stainless steel reinforcement in a corrosion cell containing glass beads, Ottawa sand, and a saturated Ca(OH)₂ solution (pH of 12.6) and found that, depending on the test method, the critical chloride ionhydroxyl ratio was 24 and larger, significantly higher than the value reported by Bertolini et al. (1996). Gu et al. (1996) found that after 25 months of exposure, a stainless steel reinforcement (Nitronic) embedded in a 0.5 water-cement ratio (w/c) concrete and exposed to a 3.5% sodium chloride aerated solution exhibited no corrosion activity. Rasheeduzzafar et al. (1992) tested stainless steel samples in chloride-contaminated concrete and found no corrosion activity after 7 years.

Although solution testing may provide an indication of relative performance, in general, these data cannot be used as reliable quantitative data for determining the service life, which is needed for making economic comparisons of the different RC systems. The challenge for evaluating the critical chloride threshold of corrosion-resistant steel reinforcement is clearly the time and costs required to initiate corrosion of the steel reinforcement in the cementitious materials. Because corrosion-resistant reinforcing steels typically have higher critical chloride threshold levels, the testing time required to initiate corrosion can be significantly longer than testing times for conventional plain carbon steel reinforcement.

If it is assumed that the transport of chloride ions occurs only as a result of chloride concentration gradients, the cementitious material has an apparent diffusion coefficient of 9×10^{-12} m²/s, the chloride concentration surface loading increases as a function of the square root of time, and the concrete cover is 0.05 meters (2 in.), the time required for the chloride concentration to reach the critical chloride threshold level can be determined. Figure 1 shows how the critical chloride threshold of the steel reinforcement can affect the time to corrosion. For a steel reinforcement with a critical chloride threshold of 0.59 kg/m³ (1 lb/yd³), the time to corrosion initiation would be approximately 3 years. If the critical chloride threshold could be raised to 5.9 kg/m³ (10 lb/yd³) or 11.8 kg/m³ (20 lb/yd³), the time to corrosion initiation could be extended to 31 and 82 years, respectively, significantly increasing the service life of the RC structure.

The main challenge associated with evaluating the critical chloride threshold of steel reinforcement exhibiting high critical chloride threshold values is the time required to evaluate this threshold level. As already discussed, most tests performed on corrosion-resistant reinforcing steels embedded in cementitious materials have provided limited quantitative data on the critical chloride threshold values. Results from the literature on corrosion-resistant steel reinforcement only provide critical chloride threshold values for these steels in simulated concrete pore solution or indicate that these steels have not activated after some extended time period. This provides no quantitative information to allow owners, designers, and engineers to estimate the service life of the different systems so economic comparisons can be made to optimize the design of the RC structure. Thus, an accelerated test method is needed to obtain quantitative values for the critical chloride threshold of steel reinforcement embedded in cementitious materials.

One approach to accelerate the transport of chloride ions into a cementitious material is to expose RC test samples to elevated temperatures. The change in diffusion coefficient as a function of temperature can be determined as follows

$$D = D_o \cdot \frac{\frac{U}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T}\right)}{e}$$
(1)

where *D* is the diffusion coefficient at some temperature *T* (in Kelvin); D_o is the diffusion coefficient at some reference temperature T_{ref} (in Kelvin); *U* is the activation energy; and *R* is the universal gas constant (8.31 J/mol – K). Berke and Hicks (1993) reported activation energies of approximately 50,000, 45,000, and 32,000 J/mol for concrete with *w/c* of 0.4, 0.5, and 0.6, respectively.

If RC samples are used to evaluate the critical chloride threshold in the laboratory, samples can be exposed to cycles of wetting and drying with a chloride solution to activate corrosion of the reinforcement. If it is assumed that transport will occur via diffusion only (an assumption for simplification), that the concrete has a w/c of 0.5, a cover depth of 0.038 m (1.5 in.), a diffusion coefficient of 9×10^{-12} m²/s at 20 °C (293 °K), and the chloride surface loading increases at a rate dependent on the square root of time, the time for the chloride ion concentration to reach the critical chloride threshold levels at different test temperatures can be determined. If it is assumed that three reinforcing steels having critical chloride threshold values of 0.59, 5.9, and 11.8 kg/m^3 (1, 10, and 20 lb/yd^3), the approximate test time for the different exposure temperatures can be determined as shown in Table 1. By raising the exposure temperature, the test time can be

	Critical chloride threshold value					
Test temperature, C° (°F)	0.59 kg/m ³ (1 lb/yd ³)	5.9 kg/m ³ (10 lb/yd ³)	11.8 kg/m ³ (20 lb/yd ³)			
20 (68)	1.8 years	13 years	28 years			
30 (86)	1.2 years	10 years	24 years			
40 (104)	0.8 years	9 years	21 years			
50 (122)	0.6 years	7 years	19.3 years			

Table 1—Estimated test periods for evaluating critical chloride threshold values of corrosionresistant steel reinforcement embedded in concrete

*Assuming diffusion of chlorides only for different exposure temperatures.

decreased by approximately 30 to 70%, with the lower percentages for the higher critical chloride threshold values.

For reinforcing steels with low critical chloride threshold values, conventional testing of RC samples at elevated temperatures consisting of ponding and drying with chloride solutions can be completed in reasonable test time periods. But, as the critical chloride threshold increases, the duration of the test becomes long, even with high exposure temperatures. These long test durations required to determine the critical chloride threshold level impede the implementation of new materials that resist corrosion because these tests are often not economical to perform. Because critical chloride threshold levels are needed to estimate the service life of RC systems and these service life periods are needed to compare life-cycle costs of the different RC systems so that the most economical system can be identified, selected, and implemented, it is essential that a standardized, short-term test be developed for estimating the critical chloride threshold of both conventional plain carbon reinforcing steel and corrosionresistant reinforcing steels.

RESEARCH SIGNIFICANCE

To economically justify the use of materials that enhance the corrosion resistance of RC structures, life-cycle cost comparisons are needed. To perform life-cycle cost analyses, the service life must be estimated. To estimate the life-cycle costs of RC systems, several parameters are needed. All parameters except the critical chloride threshold value of the steel reinforcement can be determined or evaluated over a relatively short period. This paper proposes the use of an accelerated test method to estimate the critical chloride threshold values of corrosion-resistant steel reinforcement embedded in a cementitious material.

ACCELERATED CHLORIDE THRESHOLD TEST

The accelerated chloride threshold (ACT) test method has been described in Part 1 of this paper series (Trejo and Pillai 2003). The general test methodology includes applying a potential gradient between a cathode immersed in a small reservoir on top of a canister containing chloride solution and an anode embedded in the cementitious material and placed at the depth of the reinforcement for a set time, turning off the applied potential for some time, and before applying another potential gradient, evaluating the polarization resistance R_p of the steel reinforcement sample being evaluated. As long as the chloride concentration in the cementitious material adjacent to the steel reinforcement is lower than the critical chloride threshold value of the steel reinforcement, the steel reinforcement will remain passive. As the process of applying a potential gradient continues, the chloride ion

Table 2—Cement composition

LOI	SO ₃	SiO_2	Fe ₂ O ₃	MgO	Al ₂ O ₃	Na ₂ O	CaO	C ₃ S	C ₃ A	I.R.*
1.68	2.99	20.78	1.96	1.27	5.24	0.49	64.45	57.95	10.58	0.18
** • • •										

*Insoluble residue.

Table 3—Composition as	weight % of reinforcing
steels used in ACT study	

Reinforce- ment type	С	Mn	Si	s	Р	Cu	Cr	Ni	Мо	Fe
SS304	0.025	1.19	0.53	0.009	0.03	0.56	18.43	8.64	0.35	Re.
Micro- composite	0.08	0.45	0.24	0.011	0.014	0.13	10.4	0.11	0.03	Re.
SS316LN	0.018	1.32	0.58	0.026	0.027	0.54	17.57	10.8	2.01	Re.
N										

Note: Re. = Remaining

concentration adjacent to the steel reinforcement will increase. When the chloride ion concentration at the steelcementitious material interface reaches the critical chloride threshold level of the steel reinforcement, corrosion will initiate. When corrosion activation occurs, the ACT sample is removed from further applied potential gradients and the cementitious material directly adjacent to the steel reinforcement surface is evaluated for the chloride ion concentration. This chloride ion concentration is defined as the critical chloride threshold level for the steel reinforcement in the specific cementitious material used in the test.

MATERIALS, TEST PROCEDURE, AND EXPERIMENTAL DESIGN

Materials

A mortar was used to evaluate the critical chloride threshold of three different corrosion-resistant reinforcing steels. The same mortar used to evaluate the critical chloride threshold of the ACT canisters containing the ASTM A 615 and A 706 steel reinforcement and reported in Part 1 of this paper was used in this study. The water:cement:sand ratio of the mortar mixture was 1:2:4.5. The water was distilled and exhibited a resistivity of 0.33 M Ω -cm². Type I ordinary portland cement meeting specification ASTM C 150 and Ottawa sand meeting the specification of ASTM C 778 was used in the mortar mixture. The unit weight of the mortar was obtained following ASTM C 138 and was determined to be 2107 kg/m³ (3548 lb/yd³). All critical chloride threshold values are reported using this unit weight. Using typical concrete unit weights will increase the reported critical chloride threshold values. The chemical composition of the cement is shown in Table 2. Samples were cured for 7 days at $32 \degree C \pm 3 \degree C$ (90 °F \pm 5 °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.

Three steel reinforcing types were evaluated in the test program. Microcomposite steel reinforcing bars, 304 stainless steel reinforcing bars, and 316LN stainless steel reinforcing bars were embedded in the ACT canisters and evaluated for critical chloride threshold values. The compositions of the steel reinforcing bars used in the test program are shown in Table 3. Each reinforcing bar type was obtained from the same steel heat and batch. All steel reinforcing samples were 19 mm in diameter (3/4 in.) and were cut to 19 mm (3/4 in.) lengths using a lathe. The samples were then drilled and



Fig. 2—ACT canister separated into three main components.

tapped with a 5-40 thread on the circumference of the sample at the center (9.5 mm in from the cut end). Care was taken not to disturb the as-received surface conditions. It should be noted that the stainless steels were received without mill scale. The microcomposite steel reinforcement had mill scale present. After cutting, all reinforcing steel samples were cleaned using ethyl alcohol in an ultrasonic cleaner for 5 min and all surface area, except 150 mm² (0.23 in.²) opposite the drilled and tapped hole, was coated with two coatings of low viscosity epoxy. The thickness of the epoxy layers was not measured. The steel reinforcing samples were then secured in the ACT canisters and the canisters were readied for fabrication.

Test procedure

The ACT canister is fabricated and delivered with the anode, counter electrode, and Luggin probe secured. After the steel reinforcement sample is secured in the ACT canister, the sample is ready to be cast. Mortar is mixed following a modified ASTM C 305. Instead of mixing the cement and water first, the sand is mixed with approximately 1/2 of the water for 2 min in a mortar mixer, then the cement and remaining water is added and mixed for an additional 5 min. The ACT canister is separated into the three main components as shown in Fig. 2. Mortar is placed in the bottom section of the ACT canister in one lift and the mortar is rodded approximately 20 times with a 9.5 mm rod. Care is taken not to damage the surface of the exposed steel sample or the epoxy coating. After rodding, the bottom section of the canister is tapped 10 times around the perimeter to release air pockets. After tapping, approximately 3 to 4 mm (0.12 to 0.16 in.) of additional mortar is mounded on the existing mortar in the bottom section. The anode is placed on top of the bottom section. Mounding this small amount of mortar prevents air pockets from forming under the anode. The middle section of the ACT canister is then placed on top of the lower section

and this section is then filled with mortar. The Luggin probe is then inserted in the opening of the canister and the tip is placed within 2 mm (0.08 in.) of the steel reinforcing bar surface. The Luggin probe is secured to the outside of the ACT canister with tape. Because the middle canister section is only 6 mm (0.24 in.) deep and the anode is placed at the interface of the bottom and middle portions of the canister, this section is consolidated only with tapping of the canister sides. After the middle section is filled and consolidated, the upper section of the ACT canister is placed on the filled middle section of the ACT canister. The counter electrode is pre-assembled in the upper portion, and when the upper portion of the ACT canister is placed on the filled middle section of the ACT canister, the counter electrode makes contact with the mortar placed in the middle section. Mortar is then placed in the upper portion of the ACT canister in one lift. The upper portion is then rodded 20 times and tapped 10 times. The top surface is struck even with a small, smooth wooden trowel. The 51 mm-diameter reservoir is then placed into the mortar at the center of the ACT canister approximately 6 to 7 mm (0.24 to 0.28 in.) deep. The ACT canisters are then placed in the curing room for 1 week.

After 7-day curing, samples are removed from the curing room and placed in the laboratory. A potential gradient of 20 V is applied between the anode and cathode for 12 h each day for a total of 60 h (except as noted later). A voltage source was used to apply the potential gradient. After the final 12-h potential gradient is applied, the samples are allowed to rest for 42 h. The rest period is immediately followed by evaluating the R_p of the embedded steel reinforcement using a potentiostat at a scan rate of 0.167 mV/s. The scan starts from approximately -20 mV from the measured open circuit potential (OCP) and proceeds to approximately +15 mV from the OCP. It should be noted that the OCP of the stainless steel samples was unstable for some tests and polarization scans had to be completed twice to generate a potentialcurrent plot that could provide an R_p value for these samples. When a second test was needed, a minimum of 15 min was allotted before retesting.

Experimental design

To determine the number of samples that needed to be evaluated in this phase of the research, a similar statistical approach as presented in Part 1 of this paper was used. Three ACT samples of each steel type were initially cast, cured, and tested. The mean critical chloride threshold value of these three samples \overline{X}_3 and the standard deviation SD_3 were determined for each steel type and these data were used to provide an initial estimate of the minimum number of samples required for the test program. The \overline{X}_3 for the ACT canisters with the microcomposite, SS304, and SS316LN steel reinforcements was 4.4, 4.4, and 10.3 kg/m³ (7.4, 7.3, and 17.4 lb/yd³), respectively. The SD_3 for the microcomposite, SS304, and SS316LN steel reinforcements was 0.7, 0.7, and 1.5 kg/m³ (1.2, 1.1, and 2.6 lb/yd³), respectively.

Because reliable mean and standard deviation values for the critical chloride threshold are not available for these corrosion-resistant reinforcing steels, the number of samples was first estimated assuming a normal distribution. Because the critical chloride threshold values of these corrosion-resistant steels were expected to be higher than the conventional carbon steels, the research team decided that the maximum allowable half 95% confidence interval length based on the *t*-distribution should be less than 15% of the \overline{X}_3 values instead of the 40% used for the conventional carbon steels. With this assumption, the first conservative estimate for determining the number of samples based on a normal distribution assumed a half 95% confidence interval length of 10% of the \overline{X}_3 values. For the ACT canisters containing microcomposite steel reinforcement, the first estimate for the number of samples n_o was 10 samples. The n_o for the ACT canisters with SS304 and SS316LN steel reinforcement was 10 and 9 samples, respectively. These quantities and degrees of freedom were then used to determine the half 95% confidence interval length based on a *t*-distribution and the final quantities *n* were adjusted to obtain a half 95% confidence interval length of 10% of the \overline{X}_3 values. From this analysis, it was determined that a minimum of 9 ACT samples were needed for each of the three steel reinforcement types.

RESULTS AND DISCUSSION

It has been well established that the formation of a stable passivating film at the steel surface contributes to enhanced corrosion resistance. But, the presence of the passive film in conjunction with mill scale has not been thoroughly researched. If the mill scale formed during the production of the reinforcing bars forms a tightly bound, dense physical barrier, the passive film may not form. In addition, if a cracked or a loosely bound mill scale is formed during production, the passive film will most likely form under the mill scale and at the locations of the cracks. Because the plain carbon steel reinforcement contains limited passivating elements such as chromium, silicon, and titanium, and the mill scale does exhibit cracking, these steels would be expected to have limited corrosion resistance to chloride ions.

The corrosion-resistant reinforcing steels all contained chromium in excess of 10% by weight. Because the SS304 and SS316LN reinforcing steels had the mill scale removed prior to shipping and both have chromium contents greater than 17% by weight and nickel contents greater than 8.6% by weight, a stable passive film would be expected over the entire steel reinforcement surface. These stable passive films would be expected to resist breakdown by chloride ions and should exhibit a high critical chloride threshold value. The SS316LN stainless steel reinforcement would be expected to exhibit the highest critical chloride threshold due to its higher chromium and molybdenum contents.

The microcomposite steel reinforcement had the mill scale present when received and all ACT testing including this reinforcement was tested with the mill scale present. Limited observations of this mill scale indicated some longitudinal cracking. The manufacturers of this product indicate that this reinforcing steel is microstructurally designed to eliminate microgalvanic cells that can provide better corrosion resistance and improved mechanical properties. Based on this and the fact that the steel has an elevated chromium content, the corrosion performance of this reinforcement, even with cracks in the mill scale, would be expected to be higher than the plain carbon steel.

Thus, from general scientific principles, it would be expected that the plain carbon steel reinforcing bars would exhibit low critical chloride threshold values, the SS316LN would exhibit high critical chloride threshold values, and the SS304 and microcomposite steel reinforcements would exhibit critical chloride threshold values somewhere between the plain carbon steel and the SS316LN reinforcement.

The same test procedure used to evaluate the conventional carbon steel reinforcing bars was used to evaluate the corrosion-



Fig. 3—Inverse R_p *values for microcomposite steel reinforcement embedded in ACT canisters.*



Fig. 4—Inverse R_p values for SS304 steel reinforcement embedded in ACT canisters.

resistant steel reinforcing bars. The ACT samples were evaluated for R_p values approximately 1 h after being removed from the curing room, immediately after the initial 42-h rest period (60 h after first applying the 20 V potential gradient), and after every 42-h rest period thereafter. Because preliminary testing of the ACT canisters containing SS316LN steel reinforcement exhibited high critical chloride threshold values and earlier testing indicated that the maximum amount of chlorides that could be drawn into the sample after 120 h was approximately 5 kg/m³ (8.4 lb/yd³), the research team decided to accelerate the testing of these samples by applying a potential gradient of 20 V for 12 h each day for 10 days (instead of the 5 days for the plain carbon steels). In addition, because these samples exhibited unstable OCP values at the first test time, values were not obtained for this test time. Also, because of the longer test times required for the ACT canisters containing SS316LN steel reinforcement, some R_p values were not obtained for a select number of scheduled test times. Because the corrosion rate is directly proportional to the inverse R_p values, the inverse R_p values (instead of the R_p) values are plotted as a function of applied potential gradient. These plots are shown in Fig. 3 to 5 for each steel type. Note that the abscissas on these plots have different maximum values and care should be taken when comparing the mean times to activation and the respective standard deviations. The scatter in the magnitude of the inverse R_p values for the SS316LN seemed to be less



Fig. 5—Inverse R_p values for SS316LN steel reinforcement embedded in ACT canisters.

Table 4—Critical chloride threshold value for microcomposite steel

	Criti	Critical chloride threshold value					
Sample identification	kg/m ³ (lb/yd ³)	Weight % cement	[Cl ⁻]/[OH ⁻]	Modified [Cl [_]]/[OH [_]]			
Microcomposite-01	4.7 (7.9)	0.83	1.11	2.81			
Microcomposite-02	3.6 (6.1)	0.65	0.86	1.99			
Microcomposite-03	4.9 (8.3)	0.88	1.17	3.37			
Microcomposite-04	3.4 (5.8)	0.61	0.81	3.35			
Microcomposite-05	6.1 (10.3)	1.09	1.45	4.38			
Microcomposite-06	4.1 (6.9)	0.73	0.98	2.70			
Microcomposite-07	5.0 (8.5)	0.89	1.19	3.01			
Microcomposite-08	4.8 (8.2)	0.86	1.15	2.90			
Microcomposite-09	4.5 (7.5)	0.80	1.06	2.68			
Average	4.6 (7.7)	0.82	1.09	3.02			
Standard deviation	0.8 (1.4)	0.14	0.19	0.65			

than that for both the SS304 and microcomposite. The reason for this is unknown but may be attributed to the stable passive film on the SS316LN, which resulted in more uniform inverse R_p values.

After five R_p measurements were obtained, a statistical analysis of the inverse R_p data was performed to detect when the steel reinforcement transferred from a passive to active corrosion state. This approach was used for several reasons, one being that the type of potentiostat and type of noise suppression system (if used) could affect the magnitude of the R_p . As such, a statistical analysis methodology similar to that used for the convention plain carbon steel reinforcement where a linear prediction method based on the previously obtained inverse R_p observations is used to predict a new inverse R_p value. If the actual measured inverse R_p value exhibits a *t*-score of at least 3, the sample is assumed to be actively corroding.

After it is determined that the steel reinforcement in the ACT canisters is actively corroding, the samples are sheared at the anode level, the mortar adjacent to the reinforcement is ground into dust, and this mortar dust is then evaluated for chloride ion concentration following the modified Strategic Highway Research Program (SHRP) procedure (1992). Table 4 to 6 show the critical chloride ion threshold values for each of the ACT samples containing the different corrosion-resistant reinforcements. Critical chloride ion threshold values

Table 5—Critical chloride threshold value for SS304 steel

	Critical chloride threshold value						
Sample identification	kg/m ³ (lb/yd ³)	Weight % cement	[Cl ⁻]/[OH ⁻]	Modified [Cl ⁻]/[OH ⁻]			
SS304-01	4.3 (7.3)	0.77	1.03	4.42			
SS304-02	5.0 (8.5)	0.90	1.19	5.60			
SS304-03	3.7 (6.2)	0.66	0.88	3.76			
SS304-04	5.8 (9.7)	1.02	1.36	3.77			
SS304-05	5.8 (9.7)	1.03	1.37	4.51			
SS304-06	5.7 (9.6)	1.01	1.35	6.33			
SS304-07	3.3 (5.5)	0.58	0.78	1.80			
SS304-08	6.1 (10.2)	1.08	1.44	6.74			
SS304-09	5.7 (9.5)	1.01	1.34	6.31			
Average	5.0 (8.5)	1.23	1.19	4.81			
Standard deviation	1.0 (1.7)	0.32	0.24	1.60			

Table 6—Critical chloride threshold value for SS316LN steel

	Critical chloride threshold value						
Sample identification	kg/m ³ (lb/yd ³)	Weight % cement	[Cl ⁻]/[OH ⁻]	Modified [Cl ⁻]/[OH ⁻]			
SS316LN-01	10.0 (16.9)	1.78	2.38	22.62			
SS316LN-02	9.0 (15.1)	1.60	2.13	22.15			
SS316LN-03	12.0 (20.2)	2.13	2.84	28.23			
SS316LN-04	12.9 (21.7)	2.29	3.05	30.35			
SS316LN-05*	11.8 (20.5)	2.17	2.88	42.71			
SS316LN-06	9.8 (16.5)	1.74	2.32	22.08			
SS316LN-07	10.3 (17.3)	1.83	2.43	34.49			
SS316LN-08	8.4 (14.1)	1.49	1.98	18.85			
SS316LN-09*	12.2 (19.8)	2.09	2.79	41.25			
SS316LN-10	11.2 (18.9)	2.00	2.66	26.47			
Average	10.8 (18.1)	1.91	2.47	28.92			
Standard deviation	1.5 (2.5)	0.26	0.36	8.27			

*Minimum values-samples did not activate.

are reported as kg/m³ (lb/yd³), as a percentage of cement weight, as a $[Cl^{-}]/[OH^{-}]$, and as a modified $[Cl^{-}]/[OH^{-}]$. The modified $[Cl^{-}]/[OH^{-}]$ makes adjustments for the decrease in pH as a result of applying the potential gradient as shown in Eq. (6) in Part 1 of this paper series.

It was initially assumed that the critical chloride threshold data were normally distributed. This assumption was then checked using quantile plots for both the actual chloride threshold data and its natural logarithmic data. The Shapiro-Wilkinson test for normality with the actual chloride threshold data resulted in p-values of 0.7161, 0.0704, and 0.7899 for microcomposite, SS304, and SS316LN steel samples, respectively. The Shapiro-Wilkinson test for normality with the natural logarithms of actual chloride threshold data resulted in *p*-values of 0.7811, 0.0425, and 0.7115 for microcomposite, SS304, and SS316LN steel samples, respectively. All the data, except the natural logarithms of chloride threshold data of SS304 samples, met the requirements that the Shapiro-Wilkinson p-values be greater than α (0.05) and as such the assumption of normal distribution is valid for the actual chloride threshold data. Using this, a statistical analysis of all the critical chloride threshold data was completed.

The results indicate that the ACT canisters containing microcomposite reinforcement exhibits a mean critical chloride threshold level of 4.6 kg/m³ (7.7 lb/yd³) based on the mortar unit weight. Using a 95% confidence level for this steel reinforcement, the critical chloride threshold value for the microcomposite reinforcement tested in this program would range from 3.8 to 5.3 kg/m³ (6.5 to 9.0 lb/yd³). The SS304 stainless steel reinforcement evaluated in this research exhibited a mean critical chloride threshold level of 5.0 kg/m³ (8.5 lb/yd^3) , with a 95% confidence range from 4.1 to 6.0 kg/m³ (6.9 to 10.1 lb/yd³). The SS316LN stainless steel reinforcement evaluated in the test program exhibited a mean critical chloride threshold of 10.8 kg/m³ (18.1 lb/yd³) and a 95% confidence range from 9.5 to 12.0 kg/m³ (16 to 20.2 lb/yd³). The 95% confidence range indicates a 95% probability that the critical chloride threshold for the reinforcing steels and conditions used will be in this range. Figure 6 shows a box plot of the critical chloride threshold values obtained from ACT testing for the different steel reinforcing bars.

The mean time (and standard deviation) of the time of the applied potential gradient required to activate the ACT samples was 138 (23), 181 (35.7), and 322 (23.5) h for the microcomposite, SS304, and SS316LN steel reinforcing bars, respectively. For test conditions where 60 h of potential gradient is applied during the first week and 12 h of potential gradient is applied every week thereafter, the critical chloride threshold values for the microcomposite and SS304 reinforcement can be determined in approximately 10 and 14 weeks, respectively. For the SS316LN reinforcement where the potential gradient is applied for 10 days at 12 h per day, the critical chloride threshold value can be determined in approximately 20 weeks. These test times are substantially shorter than conventional testing that includes cyclic ponding and drying of RC samples with chloride solutions.

The results of the ACT testing indicate that the SS316LN has a higher critical chloride threshold than the SS304 and microcomposite reinforcement and the SS304 and microcomposite reinforcement have higher critical chloride threshold values than conventional plain carbon steel reinforcement. The critical chloride threshold values (as a percent of cement mass) obtained from the ACT canisters with ASTM A 615 steel reinforcement agrees with lower values obtained by Hope and Ip (1987). Critical [Cl⁻]/[OH⁻] obtained for the ACT canisters containing SS316LN steel reinforcement agrees with values published by Hurley and Scully (2002). Critical chloride threshold results for ASTM A 706, SS304, and microcomposite reinforcement were not found in the literature and no comparisons can be made at this time with other findings. Results indicate that the ACT test procedure may be a viable test procedure for determining critical chloride threshold values for steel reinforcement embedded in cementitious materials. Further testing including more ACT testing and long-term testing is currently under way to further justify these findings.

CONCLUSIONS

Results from the ACT testing indicate that the critical chloride threshold values for corrosion-resistant steel reinforcement can be determined in substantially shorter times than conventional test methods. Using the proposed ACT test method with microcomposite, SS304, and SS316LN reinforcing bars embedded in a 1:2:4.5 water:cement:sand mortar mixture resulted in mean critical chloride threshold values of 4.6 kg/m³ (7.7 lb/yd³), 5.0 kg/m³ (8.5 lb/yd³), and 10.8 kg/m³ (18.1 lb/yd³),



Fig. 6—Critical chloride threshold values for reinforcing steels evaluated with ACT test method.

respectively. These values are significantly higher than the critical chloride threshold values of $0.5 \text{ kg/m}^3 (0.9 \text{ lb/yd}^3)$ for the ASTM A 615 steel reinforcement and $0.2 \text{ kg/m}^3 (0.3 \text{ lb/yd}^3)$ for ASTM A 706 steel reinforcement determined in Part 1 of this paper. Although further testing is required, the ACT test method seems to be a viable accelerated test for quantitatively and reliably determining the critical chloride threshold value of steel reinforcement in cementitious materials.

ACKNOWLEDGMENTS

The authors wish to thank MMFX Technologies Corp. and the Department of Civil Engineering at Texas A&M University for supporting the research. The assistance of D. Cline from the Statistics Department at Texas A&M University and C. Halmen, A. Bell, and V. Salgado of the Department of Civil Engineering at Texas A&M University is also appreciated.

REFERENCES

Al-Amoudi, O. S. B.; Rasheeduzzafar; Malsehuddin, M.; and Al-Mana, A. I., 1993, "Prediction of Long-Term Corrosion Resistance of Plain and Blended Cement Concretes," *ACI Materials Journal*, V. 90, No. 6, Nov.-Dec., pp. 564-570.

Ammar, A. B.; Loche, J. M.; and Dumargue, P., 1998, "Accelerated Testing of Chloride Diffusivity in Cement Based Materials: New Test Operating Mode," *Advances in Concrete Technology*, Proceedings of the Fourth CANMET/ACI International Symposium, SP-179, V. M. Malhotra, ed., American Concrete Institute, Farmington Hills, Mich., pp. 587-603.

Andióna, L. G.; Garcés, P.; Cases, F.; Andreua, C. G.; and Vazquez, J. L., 2001, "Metallic Corrosion of Steels Embedded in Calcium Aluminate Cement Mortars," *Cement and Concrete Research*, V. 31, No. 9, Sept., pp. 1263-1269.

Balabanic, G.; Bicanic, N.; and Durekovic, A., 1996, "The Influence of w/c Ratio, Concrete Cover Thickness and Degree of Water Saturation on the Corrosion Rate of Reinforcing Steel in Concrete," *Cement and Concrete Research*, V. 26, No. 5, May, pp. 761-769.

Baweja, D.; Roper, H.; and Sirivivatnanon, V., 1998, "Chloride-Induced Steel Corrosion in Concrete: Part I—Corrosion Rates, Corrosion Activity, and Attack Areas," *ACI Materials Journal*, V. 95, No. 3, May-June, pp. 207-217.

Berke, N. S., and Hicks, M. C., 1993, "Predicting Chloride Profiles in Concrete," Corrosion 93, *Paper* 341, National Association of Corrosion Engineers, pp. 1-15.

Bertolini, L.; Bolzoni, F.; Pastore, T.; and Pedeferri, P., 1996, "Stainless Steel Behaviour in Simulated Concrete Pore Solution," *British Corrosion Journal*, V. 31, No. 3, Sept., pp. 218-222.

Bleszynski, R.; Hooton, R. D.; Thomas, M. D. A.; and Rogers, C. A., 2002, "Durability of Ternary Blend Concrete with Silica Fume and Blast-Furnace Slag: Laboratory and Outdoor Exposure Site Studies," *ACI Materials Journal*, V. 99, No. 5, Sept.-Oct., pp. 499-508.

Castellote, M.; Andrade, C.; and Alonso, C., 2000, "Electrochemical Removal of Chlorides: Modeling of the Extraction, Resulting Profiles and Determination of the Efficient Time of Treatment," *Cement and Concrete Research*, V. 30, No. 4, Apr., pp. 615-621.

Chamberlin, W. P., and Weyers, R. E., 1991, "Protection and Rehabilitation Treatments for Concrete Bridge Components: Status and Service Life Opinions of Highway Agencies," *Transportation Research Record* TRR 1304, pp. 114-121. Cui, F.; Sagüés, A. A.; and Powers, R. G., 2001, "Corrosion Behavior of Stainless Steel Clad Rebar," *Corrosion/2001*, National Association of Corrosion Engineers, Paper No. 0224, 24 pp.

Darwin, D.; Locke, C. E., Jr.; Senecal, M. R.; Smith, J. L.; and Schwensen, S. M., "Corrosion-Resistant Steel Reinforcing Bars," *IDEA Program Contract* No. NCHRP-93-ID009, Transportation Research Board.

Gu, P.; Elliott, S.; Beaudoin, J. J.; and Arsenault, B., 1996, "Corrosion Resistance of Stainless Steel in Chloride Contaminated Concrete," *Cement* and Concrete Research, V. 26, No. 8, Aug., pp. 1151-1156.

Hope, B. B., and Ip, A. C., 1987, "Corrosion of Steel in Concrete Made With Slag Cement," ACI Materials Journal, V. 84, No. 6, Nov.-Dec., pp. 525-531.

Hurley, M. F., and Scully, J. R., 2002, "Chloride Threshold Levels in Clad 316L and Solid 316LN Stainless Steel Rebar," *Corrosion/2002*, National Association of Corrosion Engineers, Paper No. 02224, 24 pp.

Hussain, S. E.; Al-Gahtani, A. S.; and Rasheeduzzafar, 1996, "Chloride Threshold for Corrosion of Reinforcement in Concrete," *ACI Materials Journal*, V. 93, No. 6, Nov.-Dec., pp. 534-538.

Leng, F.; Feng, N.; and Lu, X., 2000, "An Experimental Study on the Properties of Resistance to Diffusion of Chloride Ions of Fly Ash and Blast Furnace Slag Concrete," *Cement and Concrete Research*, V. 30, No. 6, June, pp. 989-992.

Li, Z.; Peng, J.; and Ma, B., 1999, "Investigation of Chloride Diffusion for High-Performance Concrete Containing Fly Ash, Microsilica, and Chemical Admixtures," *ACI Materials Journal*, V. 96, No. 3, May-June, pp. 391-396.

Lorentz, T., and French, C., 1995, "Corrosion of Reinforcing Steel in Concrete: Effects of Materials, Mix Composition, and Cracking," *ACI Materials Journal*, V. 92, No. 2, Mar.-Apr., pp. 181-190.

Luping, T., and Nilsson, L.-O., 1993, "Rapid Determination of the Chloride Diffusivity in Concrete by Applying an Electric Field," *ACI Materials Journal*, V. 89, No. 1, Jan.-Feb., pp. 49-53.

MacDonald, K. A., and Northwood, D. O., 2000, "Rapid Estimation of Water-Cementitious Ratio and Chloride Ion Diffusivity in Hardened and Plastic Concrete by Resistivity Measurement," *Water-Cement Ratio and Other Durability Parameters—Techniques for Determination*, SP-191, M. S. Khan, ed., American Concrete Institute, Farmington Hills, Mich., pp. 57-67.

Malhotra, V. M.; Zhang, M. H.; and Leaman, G. H., 2000, "Long-Term Performance of Steel Reinforcing Bars in Portland Cement Concrete Incorporating Moderate and High Volumes of ASTM Class F Fly Ash," *ACI Materials Journal*, V. 97, No. 4, July-Aug., pp. 409-417.

Pal, S. C.; Mukherjee, A.; and Pathak, S. R., 2002, "Corrosion Behavior of Reinforcement in Slag Concrete," *ACI Materials Journal*, V. 99, No. 6, Nov.-Dec., pp. 521-527.

Pech-Canul, M. A., and Castro, P., 2002, "Corrosion Measurements of Steel Reinforcement in Concrete Exposed to a Tropical Marine Atmosphere," *Cement and Concrete Research*, V. 32, No. 3, Mar., pp. 491-498.

Rasheeduzzafar; Dakhil, F. H.; Al-Gahtani, A. S.; Al-Saadoun, S. S.; and Bader, M. A., 1990, "Influence of Cement Composition on the Corrosion of Reinforcement and Sulfate Resistance of Concrete," *ACI Materials Journal*, V. 87, No. 2, Mar.-Apr., pp. 114-122.

Rasheeduzzafar; Dakhil, F. H.; Bader, M. A.; and Khan, M. M., 1992, "Performance of Corrosion-Resisting Steels in Chloride-Bearing Concrete," *ACI Materials Journal*, V. 89, No. 5, Sept.-Oct., pp. 439-448.

Sa'id-Shawqi, Q.; Arya, C.; and Vassie, P. R., 1998, "Numerical Modeling of Electrochemical Chloride Removal from Concrete," *Cement and Concrete Research*, V. 28, No. 3, Mar., pp. 391-400.

Sivasundaram, V.; Carette, G. G.; and Malhotra, V. M., 1991, "Mechanical Properties, Creep, and Resistance to Diffusion of Chloride Ions of Concretes Incorporating High Volumes of ASTM Class F Fly Ashes from Seven Different Sources," *ACI Materials Journal*, V. 88, No. 4, July-Aug., pp. 407-416.

Trejo, D.; Monteiro, P.; Thomas, G.; and Wang, X., 1994, "Mechanical Properties and Corrosion Susceptibility of Dual-Phase Steel in Concrete," *Cement and Concrete Research*, V. 24, No. 7, Oct., pp. 1245-1250.

Trejo, D.; Monteiro, P. J. M.; Gerwick, B. C., Jr.; and Thomas, G., 2000, "Microstructural Design of Concrete Reinforcing Bars for Improved Corrosion Performance," *ACI Materials Journal*, V. 97, No. 1, Jan.-Feb., pp. 78-83.

Trejo, D., and Pillai, R., 2003, "Accelerated Chloride Threshold Testing: Part I—ASTM A 615 and A 706 Reinforcement," *ACI Materials Journal*, V. 100, No. 6, Nov.-Dec., pp. 519-527.

Yoon, S.; Wang, K.; Weiss, W. J.; and Shah, S. P., 2000, "Interaction Between Loading, Corrosion, and Serviceability of Reinforced Concrete," *ACI Materials Journal*, V. 97, No. 6, Nov.-Dec., pp. 637-644.

Zemajtis, J., and Weyers, R. E., 1995, "Service Life Evaluation of Concrete Surface Coatings," *Transportation Research Record* TRR 1490, pp. 67-74.

View publication stats