

Factors Affecting Cathodic Disbondment of Epoxy Coatings for Steel Reinforcing Bars

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Abstract

Fusion-bonded-epoxy coatings have been used successfully for more than 20 years to prevent corrosion of concrete reinforcing steel. attention in the epoxy-coated rebar industry has focused on service in hot/wet environments. New specifications mandate quality control programs that include cathodic-disbondment testing. A number of systems are available to meet the new industry requirements; including chromate and non-chromate surface treatments, coatings for prefabricated rebar, and developmental coatings that combine bendability and good adhesion retention. Beyond the choice of a coating system, various other factors play a role in the cathodicdisbondment resistance of ECR. The application temperature and thickness of the coating, and steel surface preparation and contamination are significant contributors. If a surface treatment is used, the weight of treatment applied is important. Finally, the test conditions of time, temperature, and pH have a dramatic impact on the cathodicdisbondment test results for a given system, and even on the relative test performance of different systems. Copyright © 1996 Elsevier Science Ltd.

INTRODUCTION

Coatings for rebar corrosion mitigation are in the midst of a paradigm shift. The original expectation (and hope) was that the use of epoxy-coated rebar (ECR) would extend the life of the structure. When ECR was first introduced, concrete bridge decks with uncoated rebar often required repair in as little as seven to ten years.¹ Since then, over 100,000 structures have utilized ECR, and only a handful of problems have been documented. A recent survey of the first known bridge deck utilizing ECR protection in each of thirteen states — some constructed over twenty years ago — showed that each boasted an 'outstanding maintenance history' and no maintenance was required due to rebar corrosion.²³

Discovery of premature corrosion on Florida Keys bridges resulted in two important changes. First, it raised a concern about all structures utilizing ECR and caused many surveys of existing structures. The results of those surveys have been very reassuring — epoxy-coated rebar is performing to reduce the corrosion that damstructures. **Evaluations** concrete ages hundreds of structures in many different environments point to that same conclusion. Second, it resulted in many research studies designed to understand the few problems that were unearthed during these surveys. That understanding has caused significant changes in industry standards, procedures, and expectations.⁴ One of the key expectations is the requirement for retention of coating adhesion after service exposure.

ADHESION TESTING — HISTORICAL BACKGROUND

The Ministry of Transportation of Ontario (MTO) has taken a leading role in promoting quality control (QC) adhesion tests for epoxycoated rebar in North America. Early in 1993, the MTO mandated a QC test program including a hot water immersion test (HWT), cathodic disbondment test (CDT), and salt spray test

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(SST). These tests were adapted from the European ECR industry, where chromate surface treatments are used to promote coating adhesion retention. To satisfy the new MTO requirements, Canadian coating applicators also used surface treatments.

During the test development phase, the 2 day, 75°C hot water test was chosen as the primary QC procedure because of its short duration and ease of testing. However, the subjectivity of the rating scale led to problems with meeting the requirement of 95% ratings of '1' on a 5 point scale. A '1' rating indicates no loss of adhesion. Round robin test programs were used to refine the method and rating scale, but the large test variance remained.

Subsequently, the focus shifted to the two remaining tests — the 7 day, room temperature cathodic-disbondment test and the 800 h salt spray test. The current CDT (LS 420, 94-07-01)⁵ requires a maximum disbondment radius of 2·0 mm on greater than 95% of the samples tested for a three month rolling average. The disbondment radius is the radius of the disbonded area minus the radius of the intentional holiday. Excluding prefabricated rebar, only chromate-containing surface treatments have consistently satisfied this requirement.

The salt spray test has been less controversial as the acceptance criteria can often be met without the use of a chromate pretreatment. The current test method (LS-421, 94-07-01)⁶ requires a disbondment of less than 3·0 mm on greater than 95% of the samples tested for a three month rolling average.

In the United States, adoption of new adhesion test requirements has been slower to develop. These requirements will be dictated by American Society for Testing and Materials (ASTM) specifications which are currently under revision.⁷ These specifications will likely include a test regime similar to that of the MTO. However, the exact test conditions and failure criteria may differ.

Another approach to the adhesion issue is the use of epoxy coatings on already fabricated rebar. This, so called 'coat after fab', 'prefab', or 'prebent' coating, was pioneered in California for rebar used in coastal environments. Because the coated bars are not bent, highly-filled epoxy formulations with low water permeability can be used. These tend to show better adhesion retention under hot and wet conditions than traditional rebar coatings.⁸ This type of epoxy-

coated rebar is covered by a separate ASTM specification.⁹

HOT WATER IMMERSION TEST

All organic coatings allow permeation of moisture. Water permeability is determined by such factors as the coating's free volume, molecular mobility, and hydrophilicity. Water that reaches the coating/steel interface can lead to a loss of adhesion. Presumably, the water breaks hydrogen bonds between the coating and the steel.

Hot water immersion testing requires minimal equipment and is quick and easy to run in a plant environment. On the other hand, sample evaluation requires considerable skill on the part of the quality control (QC) technician, the rating scale is subjective, and tap water composition may vary; all leading to inconsistent results. The experience of the MTO indicates that this test should not be used as the key quality control tool for epoxy-coated rebar.

Hot water immersion testing has, however, been used successfully for many years in the pipe-coating industry. As a QC test, its primary utility has been to evaluate the cleanliness and lack of contamination of the steel substrate. Because of the subjectivity of the test, the acceptance criteria have been more relaxed than the MTO requirement of 95% ratings of '1' discussed above. For example, in the Canadian Standards Association specification for fusion-bonded-epoxy pipe coating, CAN/CSA-Z245·20-M92, the requirement is a rating of '3' or better on a 5 point scale. These criteria have resulted in significant quality improvement over the years.

CATHODIC DISBONDMENT TEST

When a coated steel surface is subjected to a cathodic potential, disbondment will occur around holidays in the coating. This disbondment is thought to occur primarily because of hydroxyl ion formation at the coating/steel interface.^{13,14} However, depending on the test conditions, water permeation and hydrogen gas generation may also play important roles.

Cathodic-disbondment testing (CDT), like hot water immersion, has a long, useful history in pipe-coatings. In that industry, there is a recognition that there are two significant components to long term adhesion retention:

- (1) The coating material.
- (2) The application process.

Long-term, more severe testing protocols are used to differentiate among coating materials. Quality control test procedures are designed to give rapid feedback on the application process. Therefore, short-term test procedures were designed to identify specific problems such as contamination of the substrate. For example, National Association of Corrosion Engineers (NACE) Standard RP0394-94¹⁵ calls for a 24 h test at 66°C (150°F) with an acceptance criterion of 12 mm radius disbondment and a suggested criterion of 8 mm. This test has been very effective in discerning cleaning or contamination problems in the application process.

Clearly, when cathodic protection is used in conjunction with an epoxy coating, as for piperesistance cathodic applications, to disbondment is an important coating property. However, even in cases where cathodic protection is not normally used, the CDT may still be of value. Other industries, like the American Petroleum Institute, use cathodic disbondment testing as a QC tool for coatings which will not be under cathodic protection. 16 Also, if cathodic disbondment is a key adhesion loss mechanism for ECR in concrete, as proposed,17 then the CDT may have significant predictive value as well.

COATING ALTERNATIVES FOR ADHESION IMPROVEMENT

There are several viable technologies available to improve resistance to coating adhesion loss for epoxy-coated rebar:

- (1) Surface treatments:
 - (a) Chromate.
 - (b) Non-chromate ('water-based').
- (2) Developmental coatings.
- (3) Coatings for prefab.

Surface treatments

Surface treatment materials have been used for over sixty years. 18 Chromate treatments to improve the performance of pipeline coatings have been in use for nearly twenty years. Their primary purpose is to improve cathodic-dis-

bondment resistance and adhesion retention under hot wet conditions. A major concern is that they contain chromium and, thus, are potentially dangerous to people and the environment.

Figure 1 shows the effect of a chromate surface treatment on the cathodic-disbondment test performance of several classes of coating materials at various temperatures. Improved CDT results were obtained for all three coatings by using the chromate surface treatment.

Another recently introduced system is the proprietary 'water-based' surface treatment. This system is also designed to improve cathodic-disbondment resistance and adhesion retention of fusion-bonded-epoxy (FBE) coatings. The materials are chromium free and, therefore, eliminate that concern about their use and handling. They form a surface that is readily wettable and has a microporous structure to provide mechanical keying of the epoxy top coat.

Both treatment systems have the advantage of working with currently available FBE coating materials to improve adhesion performance without reformulation. The equipment for the surface-treatment process readily fits into the plant layout for currently operating in-line plants. While the liquid should be dry, or nearly so, at the time it reaches the heating unit, there is no need for a lengthy curing or 'set' time that requires reengineering of existing coating plants.

Developmental coatings

The most desirable coating is one for which a surface treatment is unnecessary. Here an adhesion promoter is compounded into the coating formulation to give the desired adhesion retention. Unfortunately, this is often accompanied by a loss of other desirable properties, such as flexibility and application speed. Balancing such properties has proven to be difficult, and so far, these coatings are still in the developmental stages.

Coatings for prefab

Some structures, such as those with concrete exposed to the splash-zone area in warm, saltwater environments, are particularly susceptible to corrosion. Although there were several other factors involved, the experience with some Flor-

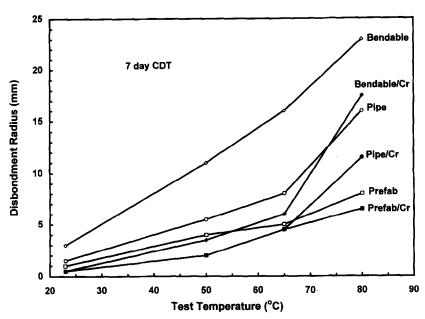


Fig. 1. Effect of a chromate surface treatment (Cr) on cathodic-disbondment for several coatings at various temperatures. Test conditions: 7 days, 1.5 V, 3% NaCl, 3 mm holiday.

ida Keys bridges demonstrates that even epoxy-coated rebar is not a panacea. Under these circumstances, a different application technique for epoxy coating of the rebar may be warranted. One such procedure is to fabricate the bar prior to the cleaning and application process.

Coating of prefabricated bar gives improved protection from severe corrosion environments. It eliminates the exposed metal caused by shearing in the fabrication process. It also eliminates the reduction of adhesion and potential cracking associated with the bending process.

Application of the coating to prefabricated reinforcing steel provides, possibly, an even greater advantage in that less flexible formulations can be used that are designed for improved adhesion retention. The first material used to take advantage of this process was a pipe coating. It had a twenty-year history of protecting pipe in the soil and in the sea and was based on a technology in use for thirty-five years.

While pipe coatings have to be flexible — pipelines are bent to fit the contours of the earth — such as when crossing mountain ranges, they do not require the extreme flexibility needed for rebar fabrication. Because less flexibility is required, the pipe-coating material can be more easily tailored to provide improved adhesion retention under conditions such as warm salt water.

The newest materials introduced to the prefabrication market have leapfrogged the traditional pipecoating technology with even better adhesion retention and have been redesigned for the application characteristics required by the prefabricated coating process.

MATERIALS AND METHODS

For this study, commercially available materials were used to evaluate the factors affecting cathodic disbondment test performance. 3M Scotchkote Brand Fusion Bonded Epoxy Coatings were applied by dipping heated bars in a fluidized bed of powder. These included a standard rebar coating, a prefabricated rebar coating, and a standard external pipeline coating; labeled 'bendable', 'prefab', and 'pipe', respectively. Where indicated, Iridite PC (10% solution) chromate surface treatment from J R Hancock Associates, labeled 'Cr', or a non-chromate 3M Scotchkote Surface Treatment, labeled 'WB' for water-based, was applied with a foam brush and dried with forced air.

Two systems that show exceptionally good cathodic-disbondment test performance are;

- (1) Standard bendable rebar coatings used over chromate surface treatments, and
- (2) Coatings designed for prefabricated rebar.

Therefore, examples of these two systems were chosen for much of the work in this study. In this work, the prefab system usually performed better than the chromate system. However, since no attempt was made to fully optimize either system, this should not be drawn as a general conclusion. Significant variation in CDT performance may occur depending on the weight of chromate treatment applied.¹⁹

Unless otherwise indicated, testing was performed on rectangular $1 \times 2.5 \times 18$ mm hot rolled mild steel strips. The strips were gritblasted to near white metal with an average anchor depth of 40 mm (1.6 mils), heated in an induction coil to approximately 238°C (460°F), and coated with 250 μ m (10 mils) of fusion-bonded epoxy.

Before beginning the study, CDT results for these rectangular samples were compared to those for 20 mm rebars. The results for the two substrate geometries were essentially identical.

Cathodic-disbondment tests were performed according to MTO Test Method LS-420⁵. Test conditions were varied as indicated. At room temperature, 23°C, the cathodic disbondment radius is often on the same order as the test variance. Therefore, an elevated temperature of 50°C was used for most of the testing in this study. This also resulted in greater differentiation between sample groups. A minimum of three replicates were run for each data point. Separate 'control' samples were tested for each part of the study.

RESULTS AND DISCUSSION

Time/temperature behavior

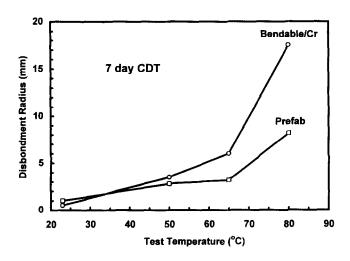
For low temperature cathodic-disbondment tests, the chromate system with a standard bendable rebar coating performed exceptionally well, yielding a disbondment radius below 1.0 mm for the room temperature test. For high temperature testing, however, the prefab system was considerably better than the chromate system. At a low temperature, one would expect the water permeation rate to be quite small and nearly all of the disbondment could be attributed to hydroxyl ion formation. At high temperature, disbondment due to water permeation is likely to be significant. The elevated temperature speeds the water permeation process that naturally occurs in a wet environment.

The sharp increase in disbondment for the chromate system at 80°C may be due to plasticization. The glass transition temperature $(T_{\rm g})$ of the standard rebar coating is only about 90°C. The prefab coating has a higher $T_{\rm g}$, about 110°C, and a lower water permeability than the bendable coating.

This temperature phenomenon translates into a similar behavior with respect to test duration. Fig. 2 shows CDT results comparing the variation of test temperature for a constant duration of 7 days to the variation of test duration for a constant temperature of 50°C. For all but short test times, the prefab system performed better than the chromate system.

Test pH

Cathodic-disbondment tests were performed at three pH values in the following solutions;



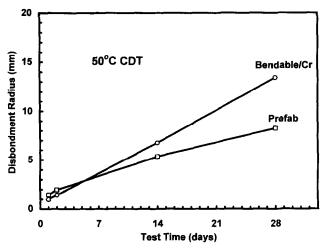
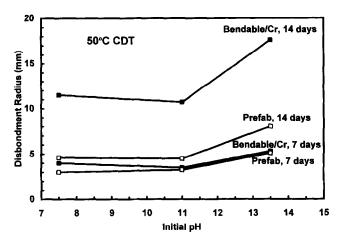


Fig. 2. Temperature and time dependence of cathodic-disbondment for two coating systems. Test conditions: 1.5 V, 3% NaCl, 3 mm holiday.

pH 7 —3% NaCl
pH 11 —3% NaCl, 9×10^{-4} N KOH, 1.5×10^{-4} N NaOH
pH 13.5 —3% NaCl, 0.3N KOH, 0.05N NaOH

As seen by McDonald et al.⁷ considerably more disbondment was observed at the highest pH level. The chromate system was affected more dramatically than the prefab coating. The low concentrations of KOH and NaOH in the pH 11 solution had little effect on either coating system. The test cells were capped, but apparently, the effect of these species was overwhelmed by carbonation and electrochemical reaction products within the test cells. As shown in Fig. 3, the solutions with initial pH values near 7 and 11 converged over the first two days of the experiment to a common equilibrium pH of about 9. Therefore, disbondment values were nearly identical for the two solutions.



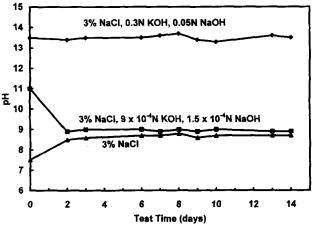


Fig. 3. Effect of pH on 7 day and 14 day cathodic-disbondment tests for two coating systems, and the variation of pH with test duration. Test conditions: 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

Powder application parameters

Beyond the choice of a coating material, several application parameters can be significant with regard to cathodic-disbondment test results. Previous work in this and other laboratories has shown that coating thickness is important. This is especially true in the high temperature or long duration test regimes where water permeation is important. A thicker coating provides a better water barrier and thus improved CDT performance compared to a thin coating. Further work on this topic was not pursued here.

Figure 4 shows the effect of powder application temperature and cure time for the prefab coating. Significantly better CDT results were obtained at 238°C (460°F) than at 191°C (375°F), presumably due to superior wetting of the substrate at the higher application temperature. Cure time beyond 15 s was not significant nor was the presence or absence of a water quench. It should be noted that this coating material is relatively quick gelling, and a major portion of the cure takes place in the first 15 s. One might expect a more grossly undercured powder coating to show some deterioration in CDT performance. These results indicate that cathodic-disbondment testing should not be used as a measure of cure.

Steel surface preparation and contamination

Not surprisingly, a key step toward achieving maximal coating disbondment resistance is the steel surface preparation.²¹ As expected, the best adhesion retention is obtained when the steel is contaminant free and given a high surface-area anchor pattern.

Figure 5 shows the effect of three different blast media on CDT results. Shot blasting gave a shallow anchor depth (25 μ m) and low peak count (44/cm) compared to sand (30 μ m, 100/cm) or grit (40 μ m, 66/cm) resulting in far inferior disbondment resistance. Since a handheld blast unit was used, these anchor depths are all somewhat shallow compared to plant profiles. However, the above conclusions have been confirmed under plant conditions, as well.

Surface contamination can also have a large impact on coating disbondment resistance. In Fig. 6, several contaminant solutions were prepared using sodium chloride, pump oil, or blaster dust residue and applied to the blasted

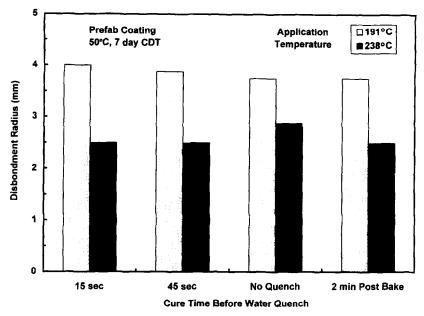


Fig. 4. Effect of powder application temperature and cure on cathodic-disbondment for a prefab rebar coating. Test conditions: 7 days, 50°C, 1·5 V, 3% NaCl, 3 mm holiday.

steel surface prior to coating application. All of the contaminants were detrimental to CDT performance, especially the salt solution.

Under normal application conditions, the clean, preheated steel surface appears golden just prior to powder coating. Periodically, a blue surface is seen. In this study, blue iron oxide was purposefully formed by either preheating the blasted test bars to 260°C (500°F) in an oven for 4 h then air cooling to room tempera-

ture or induction heating them to 360°C (680°F) and immediately air cooling to room temperature. The blued bars were then preheated and coated in the normal way. As shown in Fig. 7, the blued samples were consistently better than the controls for both coating systems. The method of blueing was not a factor. Plant experience in the pipe-coating industry confirms this result. Nevertheless, blue steel is generally avoided because it is usually an indication of

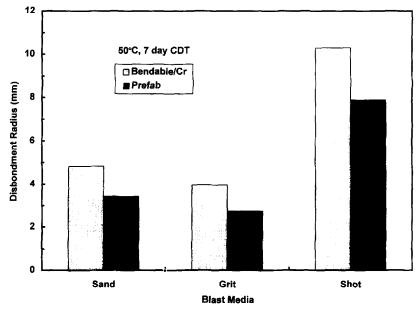


Fig. 5. Effect of steel surface preparation using different blast media on cathodic-disbondment for two coating systems. Test conditions: 7 days, 50° C, 1.5 V, 3% NaCl, 3 mm holiday.

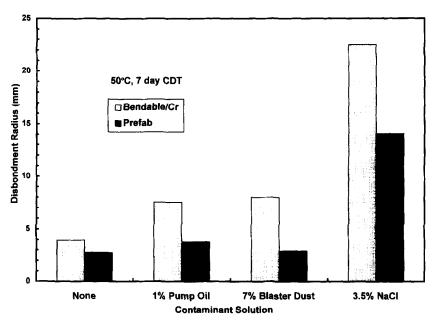


Fig. 6. Effect of intentional steel surface contamination with oil, dust, or salt (NaCl) on cathodic-disbondment for two coating systems. Test conditions: 7 days, 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

some other problem, such as excessive heating or contamination.

Surface treatment application

Several factors related to surface-treatment application were investigated for the chromate treatment and the non-chromate, or 'water-based', treatment. The weight of treatment applied to the steel was the most significant

factor. In this study, this weight was varied by diluting or concentrating the treatment solution or by applying the treatment twice with a drying step between applications. As shown in Fig. 8, increased treatment weight led to improved CDT performance for both surface treatments. A concentrated solution of the water-based treatment could not be made without gelation of the mixture. These results indicate that the treatment weight used for all other portions of

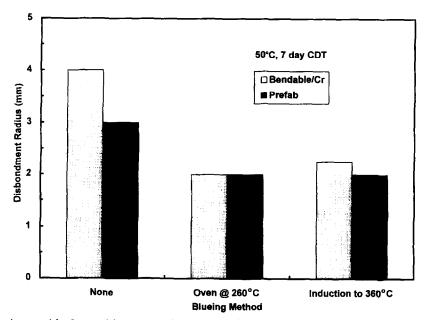


Fig. 7. Effect of blue iron oxide formed by two preheat methods on cathodic-disbondment for two coating systems. Test conditions: 7 days, 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

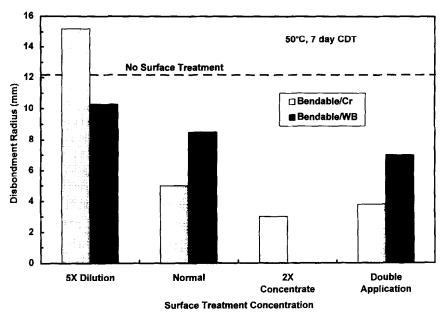


Fig. 8. Effect of surface treatment weight on cathodic-disbondment for a chromate treatment (Cr) and a non-chromate 'water-based' treatment (WB). Test conditions: 7 days, 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

this study, labeled 'normal', is not the optimum. For the dilute chromate treatment, CDT performance was even worse than with no treatment at all. This same phenomenon was reported by Higgins and Cable¹⁹ and suggests that surface treatment weight must be well controlled for optimum performance.

Application temperature and drying method for the surface treatments were also studied. These had little effect on cathodic-disbondment test performance as shown in Figs 9 and 10.

CONCLUSION

Fusion-bonded-epoxy coatings have been used successfully for more than 20 years to prevent corrosion of concrete reinforcing steel. Despite this success, concern has been raised about long-term adhesion retention in severe service environments. New industry standards are aimed at addressing this issue.

As new specifications and test programs are instituted for epoxy-coated rebar, it is necessary to understand the factors that affect test per-

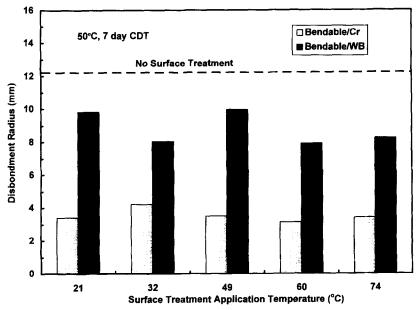


Fig. 9. Effect of surface treatment application temperature on cathodic-disbondment for a chromate treatment (Cr) and a non-chromate 'water-based' treatment (WB). Test conditions: 7 days, 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

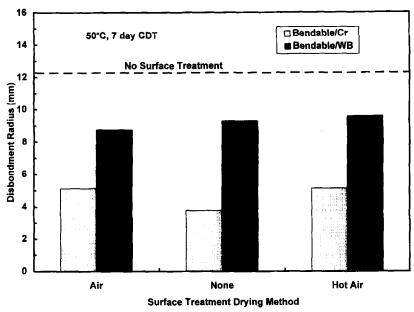


Fig. 10. Effect of surface treatment drying method on cathodic-disbondment for a chromate treatment (Cr) and a non-chromate 'water-based' treatment (WB). Test conditions: 7 days, 50°C, 1.5 V, 3% NaCl, 3 mm holiday.

formance. A number of systems are available to meet the new industry requirements, including chromate and non-chromate surface treatments. coatings for prefabricated rebar, and developmental coatings that combine bendability and good adhesion retention. However, choosing a high quality coating system may not insure maximum performance. Various other factors affect the cathodic-disbondment resistance of ECR. The application temperature and thickof the coating, and steel surface preparation and contamination play significant roles. If a surface treatment is used, the amount of treatment applied is important. Finally, the test conditions of time, temperature, and pH have a dramatic impact on the cathodic-disbondment test results for a given system.

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