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Marcus Yudt
mny7@uakron.edu

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Electrochemical Chloride Removal (ECR) of Reinforced Concrete

Report Submitted by:

Marcus Yudt

Date: 4/15/2021

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Abstract

Electrochemical chloride removal (ECR) was tested to see its effectiveness in preventing corrosion on rebar samples within 3.5 wt% sodium chloride concrete. In order to do this, three scenarios were evaluated using electrochemical techniques such as cyclic potentiodynamic polarization and electrochemical impedance spectroscopy as well as visual inspections. The three scenarios that were evaluated were one with no chlorides present within the concrete, one that was a worst-case scenario where extreme pitting and corrosion were initiated, and one that had chlorides present and received the ECR treatment. Cyclic polarization curves showed that the ECR and reference without chlorides had similar corrosion current densities, within one order of magnitude, while the ECR corrosion potential was much more negative than that of the reference cell. Both the reference and the ECR samples outperformed the worst-case scenario sample in the electrochemical testing and the visual inspection. CPP was used to confirm that even in the presence of chlorides, when ECR is applied, the rebar sample within the contaminated concrete receives additional protection and inhibition to the corrosion that would often be occurring within. The corrosion rates for the three samples were as follows- Reference- 2.45 mpy, Worst-Case- 525.42, and ECR-10.47. The calculated corrosion rate for the ECR sample was approximately 50 times smaller than that of the worst-case scenario situation. These calculated corrosion rates were also used to confirm the belief and understanding that as the ECR is applied, chlorides are expelled from the concrete and thus the sample becomes more protected and thus has better corrosion resistance and the lifespan of the sample increases.

Executive Summary

Problem Statement

The current reinforced concrete structures within the United States were initially thought to last hundreds of years, but they are not. These structures are failing at a much quicker rate than anyone anticipated and that can partially be linked to the fact that corrosion within the concrete is difficult to see early on. Corrosion of the rebar structure within the concrete itself due to chloride contaminations causes the concrete surrounding to fail. This project seeks to test the effectiveness of a process known as electrochemical chloride removal (ECR). This process works by expelling the chlorides within the concrete and thus protecting the carbon steel from its corrosive attack. The electrochemical tests that were conducted were testing for the open circuit potential (OCP), electrochemical impedance spectroscopy (EIS), and cyclic potentiodynamic polarization (CPP). These tests were used to compare the control specimen to the ECR specimen. The environment used for this testing was a reinforced concrete specimen with an 8-inch piece of AISI 1018 carbon steel rebar submerged within 6 inches of a concrete solution that was made up of 3.5 wt% NaCl.

Results

Three samples were studied using various electrochemical methods. The CPP graphs which contain the Tafel fit data are shown within **Figures 4-7**. These graphs show how rebar reacts both in a non-chloride environment, an aggressive corrosion chloride environment, and a protected chloride contaminated environment. The non-chloride environment performed the best in terms of corrosion resistance with the ECR sample following closely behind. The aggressive environment, which performed the worst, showed signs of extreme pitting and corrosion which lead to an eventual concrete failure as evidenced in **Figures 13-15**.

Conclusions

It was concluded that ECR is a viable and effective way of remediating and removing chloride ions from attacking the carbon steel within reinforced concrete. This method provided for effective removal of chloride ions which protected the carbon steel and returned it to a state of protection by a passive film formation. Although this testing scale was that of a small-scale in lab environment, it was concluded that this set-up would continue to perform well on larger-scale lab set-ups and real-world applications. Electrochemical chloride removal was found to be effective in this environment.

Implications

The work presented within this report pose various significant positive impacts. The first of which involves an increased awareness and understanding of an electrochemical process that can be used to reduce the risk of major chloride induced corrosion failures. Although this process was already known, proving and studying the effectiveness of this process were important to carrying on and furthering the knowledge of this research topic. The second was a better understanding of the research applications and instrumentation that can be found within common corrosion labs. Along with better understanding of lab applications, being within the lab and gathering the necessary materials to complete this report helped to gain real-world laboratory experience.

Recommendations

Further research should be done in this topic to study and prove a variety of applications and situations can be met. One such application that should be studied further is that of varying initial chloride concentrations for the starting reinforced concrete specimen as this study only considered that of 3.5 wt% NaCl. In addition to this, as field situations would not be of corrosion free

specimens, samples with precorroded carbon steel rebar should be studied to see the change in effectiveness and the level of protection that ECR may still be able to provide. These recommendations would help to show additional applications for ECR.

In addition to the suggestion for additional environments to be tested, testing the concrete in various places post-ECR treatment for a chemical analysis would better help to understand the efficiency of removing the chloride ions and also to give a better understanding of the speed at which this could be expected to be completed on a large-scale project.

Introduction

Rebar is used within concrete to offer a reinforced, more versatile large-scale building material. Although this reinforcement offers additional strength and is very commonly used, carbon steel corrosion within the concrete can be increasingly difficult to detect and mitigate. Corrosion within reinforced concrete highway bridges is estimated to directly cost the United States roughly \$8.3 billion [1]. Even with this mitigation, failures are becoming increasingly more common. One such failure occurred on June 24, 2021 at the Surfside condominium in Surfside, Florida, resulting in 98 deaths [2,3]. Prior to this condominium collapse, the estimate of the cost to repair the building's corrosion problems had approached nearly \$12 million [4].

Concrete and rebar systems have been used in order to provide additional reinforcements since the mid-19th century [5]. Although many of the initial reinforced concrete structures have since been demolished and replaced, there is still a large amount in service well past its realistic lifespan. It is estimated that reinforced concrete has a lifespan of 50-100 years with deterioration beginning in as little as 10 years [6]. Even with lifespan estimates of 50-100 years, environmental factors can play a large role in how long the reinforced concrete will last- as evidenced by the fact that the Surfside condominium, as previously mentioned, failed almost 40 years after it was constructed. This premature failure may have been in part to the heavy seawater environment that it was exposed to continuously being just blocks from the ocean. Seawater contains chlorides that promote and accelerate the corrosion of the carbon steels used to reinforce the concrete structures, thus resulting in weaknesses and premature failures.

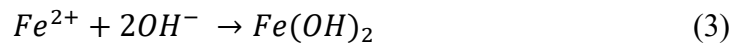
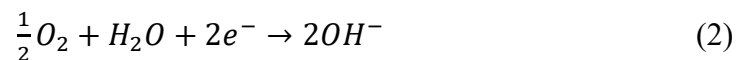
In addition to the cost to replace these reinforced concrete structures and the lives that it prematurely takes or alters, concrete production is a very large producer of CO₂ emissions. Cement produces approximately 8% of the world's carbon dioxide emissions [7]. For this reason, extending the lifespan of large buildings and bridges that would otherwise need to be replaced by another

massive concrete structure upon failure could prove very beneficial in the fight against climate change.

Although corrosion of reinforced concrete is difficult to identify, affordable and effective mitigation methods need to be implemented to ensure that these failures do not become even more common. One such method to be tested within this study is electrochemical chloride removal. This process works to expel chlorides from the concrete and thus away from the carbon steel by an electrochemical method which would result in a potentially increased lifespan of the system. This study used this method and applied approximately 30 volts to the rebar in a concrete encasement in order to actively expel chlorides that were in the system. Once this was completed, the samples were studied using the OCP, EIS, and CPP results.

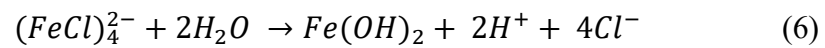
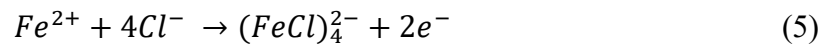
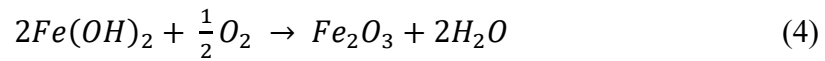
Background

Corrosion of reinforced carbon steel occurs by the anodic dissolution of iron and the reduction of oxygen. This is shown by equations (1) and (2). Once the reactions of equations (1) and (2) occur, their products can react with each other to form $Fe(OH)_2$ as shown in equation (3).



In the presence of chlorides, such as in a seawater environment, extreme corrosion can occur on carbon steel. The chlorides penetrate through the porous concrete systems and accelerate the corrosion of the carbon steel inside. In some environments, a protective film can form on the

carbon steel as shown in equation (4). In this equation, the product from equation (3), $Fe(OH)_2$ reacts with oxygen to form Fe_2O_3 , the protective film. Once this protective film forms in environments where chlorides are present, the chlorides will react with iron ions to form equation (4) resulting in the formation of $(FeCl_4)^{2-}$ as shown in equation (5). Once the formation of $(FeCl_4)^{2-}$ has begun, it will react with moisture to form three products- one of which being chloride ions. This reaction is shown in equation (6).



As shown in equation 6, this process will result in chloride ions acting as a catalyst to further degradation. As this process continues to occur, the process becomes autocatalytic and results in the continuous corrosion and degradation of the carbon steel within the concrete system. Once these products are formed, the corrosion by-product can lead to concrete failures such as cracks, delamination, and spalling due to the large size of the products being formed.

Removal of the chloride contaminants to protect the carbon steel reinforcements is of utmost importance to ensuring the continued safe operation and strength of these structures in chloride rich environments. The process by which this study seeks to remove these chloride contaminants is through electrochemical chloride removal (ECR). ECR involves charging the rebar, the carbon steel within the concrete, negatively by using a direct current (DC) power supply while simultaneously charging the anode on the outside of the concrete positively. When the rebar becomes negatively charged, the chlorides become repelled from the rebar, as they are also negatively charged, and attracted to the anode on the outside. Once all of the chlorides are expelled

from the system and attached to the anode, the anode can then be removed and allow for the ECR procedure to be repeated as necessary. Once this process is completed, the concrete system becomes a higher alkaline environment and the carbon steel structure reforms its passive film and is again protected.

While this testing can be accomplished easily in the lab, as the concrete samples can be made in a manner that allows for a DC connection to be made without additional work needing to be done, in the field this process would involve drilling holes into the concrete so that a direct connection can be made to the reinforcing rebar in the system.

This process was completed on the Rainbow Bridge in Idaho in 2006. The electrochemical chloride removal system was installed on the bridge on July 20, 2006, and treatment was completed on September 14, 2006 [8]. This bridge was approximately 8000 ft², or 745m², of concrete surface. Once this treatment process was completed, the installed ECR system was able to be removed and the bridge returned to the way it had looked prior but was now protected from the chloride attack it was previously under. This protection method cost Idaho \$10 million dollars, but replacement cost would have been significantly higher.

Experimental Methods

Testing Preparation

Two different types of concrete were made for this study- with and without chlorides. The concrete that was mixed with chlorides was made using the concrete mix and adding sodium chloride such that there was 3.5wt% added into the mixture. The concrete and mixture that was made was 14.9 wt% cement, 29.7 wt% sand, 44.5 wt% gravel, 7.4 wt% water, and 3.5 wt% sodium chloride. The

concrete that was made without the chlorides was made using one part cement, two parts sand, three parts gravel, and half part water. Once these mixtures were made up an AISI 316LN stainless steel rebar sample of known length and area was submerged into the concrete such that the area submerged was known and repeated for all samples. The concrete without chlorides was marked so that it would be easily known which specimen did, or did not, have chlorides in it. These concrete specimens were then allowed to dry over four days to ensure they were properly set and adequately dried.

Laboratory Preparation-

Once the samples were given adequate time to dry, electrochemical testing began. The solution that this testing occurred in was a 2-gram calcium hydroxide per liter solution. This mixture was made using a magnetic stirrer to ensure adequate and thorough mixing. The solution was poured into this container up to 8.9 cm up the concrete sample. The first test to be run was the OCP, EIS, and CPP testing for the control sample (without chlorides). After this testing was completed, baseline testing was done for a sample with chlorides that had not received the ECR treatment. The testing set-up for this can be seen in **Figure 1**. In this test set-up, a steel mesh, wrapped uniformly around the concrete sample, was used for the counter electrode, a saturated calomel electrode was used for the reference electrode, and the concrete and rebar was the working electrode. These electrodes were then hooked-up to the Gamry potentiostat accordingly.



Figure 1: Photograph of the reference cell set-up that was used to gather OCP, EIS, and CPP data.

As the control samples were being tested, ECR treatment began on one of the concrete samples with chlorides in it. This treatment was done in a 2-gram calcium hydroxide per liter solution. Enough solution was made so that the concrete sample was submerged 11.4 cm. This was done to ensure that treatment was applied above that of what would later be tested electrochemically. Once

the solution was poured into the container such that the concrete sample was submerged appropriately, the wrapped steel mesh was placed around the concrete sample. Ensure that this mesh is not in contact with the concrete sample. After the set-up was verified, the ECR treatment set-up was applied. This was applied using a DC power supply with the negative output being connected to the rebar coming out of the concrete and the positive output being connected to the steel mesh. This testing was done at 15 volts for 5 hours. A multimeter can be used to check that the DC power supply is applying the correct voltage to the sample. This set-up can be seen in **Figure 2.**



Figure 2: Testing set-up for the electrochemical chloride removal (ECR) set-up shown above. In this image, the positive, red wire, was connected to the steel mesh, and the negative, black wire, was connected to the rebar with the voltage being set at 15 volts.

After the ECR treatment was applied to the rebar, the same electrochemical testing that was done to the reference samples was repeated. The set-up for this testing was the same as that of **Figure 1** except the concrete sample had been treated with ECR.

Once all electrochemical testing was completed, the concrete encasement for each rebar sample was broken open with a hammer so that the rebar samples themselves could be photographed and studied for any signs of corrosion.

Laboratory Safety

While in the lab, proper personal protective equipment (PPE) must be worn and used. This PPE is, but is not limited to, gloves, lab coats, face masks, safety goggles, long pants or skirt, close toed shoes. Fire extinguishers and chemical showers were located within the lab in case of emergency.

In addition to the PPE being worn while in the lab, this testing method required that high voltages (30 volts) be applied to the sample which required extreme caution when setting up and monitoring the sample. This testing set-up was disconnected and turned off as soon as possible when testing was complete to reduce the risk of accidental shock.

With the COVID 19 pandemic still in effect, additional safety protocols were necessary and followed. These additional safety measures included, but were not limited to, remaining socially distant (6 feet of separation when possible), properly always wearing a mask, additional hand washing, and reporting any positive, or close contact, cases.

While working with the DC power supply, extreme caution was taken as a serious shock hazard was present.

Regulations/ Standards

This ECR testing method followed the guidelines within NACE Standard SP0107-2021, Electrochemical Realkalization and Chloride Extraction for Reinforced Concrete. This standard provided guidance for the laboratory set-up and the voltage (30 volts) that would be required for this test to be carried out. The reference electrode, counter electrode, and working electrode were each connected to their respective outlets on the DC power supply using alligator clips. The testing set-up was monitored to ensure that proper connections were maintained throughout the testing period.

Testing Budget

The testing that was completed for this study was conducted within the University of Akron's Engineering Research Center (ERC) where students can conduct research under the guidance of professors. Because of this labor and the use of computers for this study within the ERC were not calculated into the budget. The cost of this testing is shown in **Table 1**. The total cost for this test is **\$16,075**. The majority of this budget comes from the potentiostat that is used for the electrochemical testing.

Table 1: Estimated budget for this study

Material	Cost
Gamry 600 Potentiostat	\$10,000
0-30 V DC Power Supply	\$2,500
Fisher Science Edu Scale	\$2,000
Gamry Vista Shield (Faraday Cage)	\$1,000
Steel Mesh	\$100
Saturated Calomel Electrode	\$100
AISI 316LN SS Rebars	\$100
Personal Protective Equipment	\$75
Glassware	\$75
Copper Wires	\$50
Hammer	\$25
Sodium Chloride	\$25
Calcium Hydroxide	\$25
Total	\$16,075

Data and Results

Cyclic Potentiodynamic Polarization-

Throughout this study, there was three separate CPP graphs generated. The first CPP scan was done for the controlled, no chloride environment. The second scan was done for the worst-case scenario with extreme pitting and corrosion. The final scan was done for the sample that had received the ECR treatment. From these graphs, i_{corr} and E_{corr} values were gathered using a Tafel fit. In addition to gathering the corrosion current density and corrosion potential, the anodic and cathodic beta values were able to be gathered in order to better look at the corrosion rates. An example of this Tafel fit is shown in **Figure 3**[9].

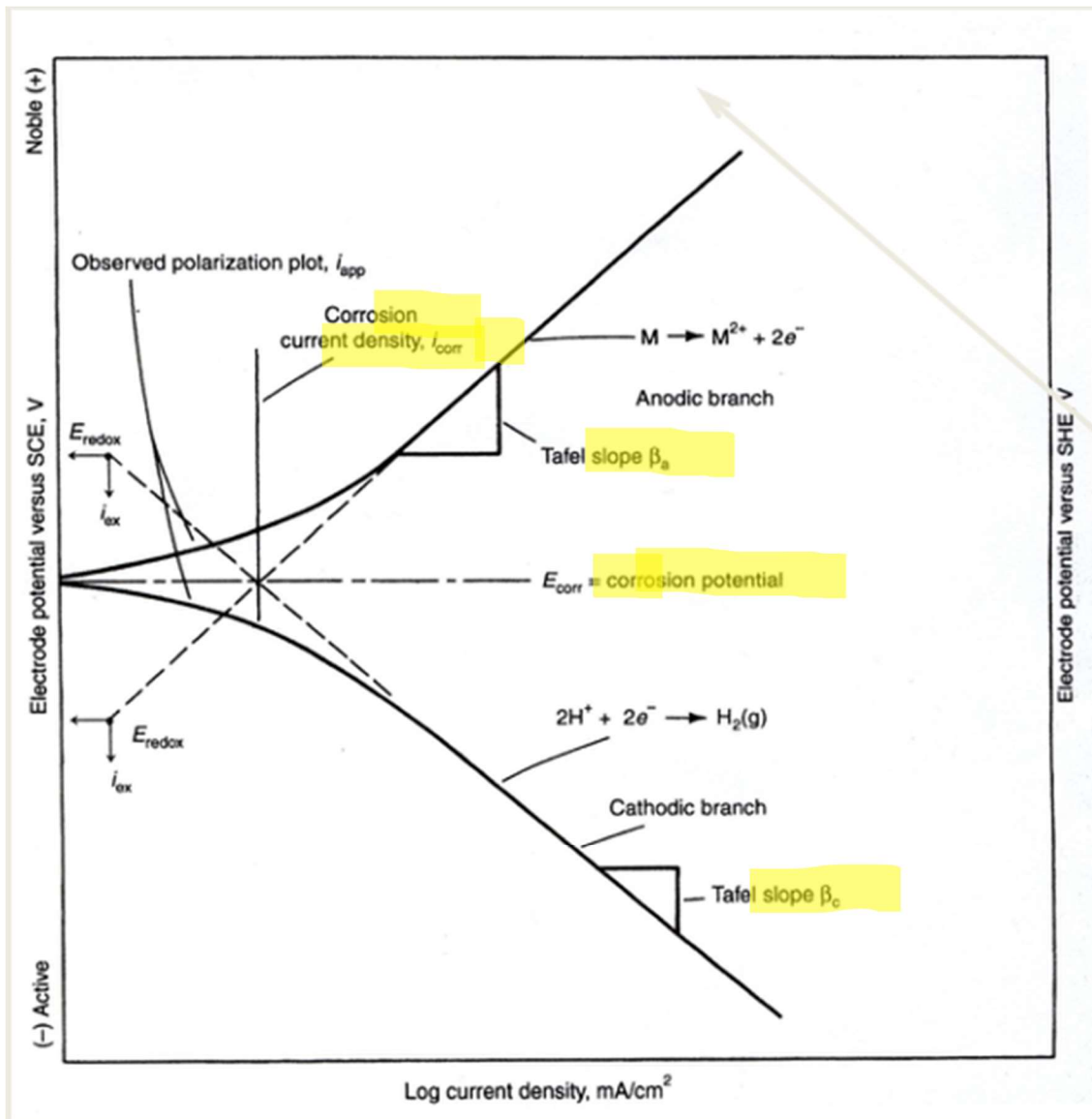


Figure 3: Tafel fit showing where E_{corr} , i_{corr} , β_a and β_c come from.

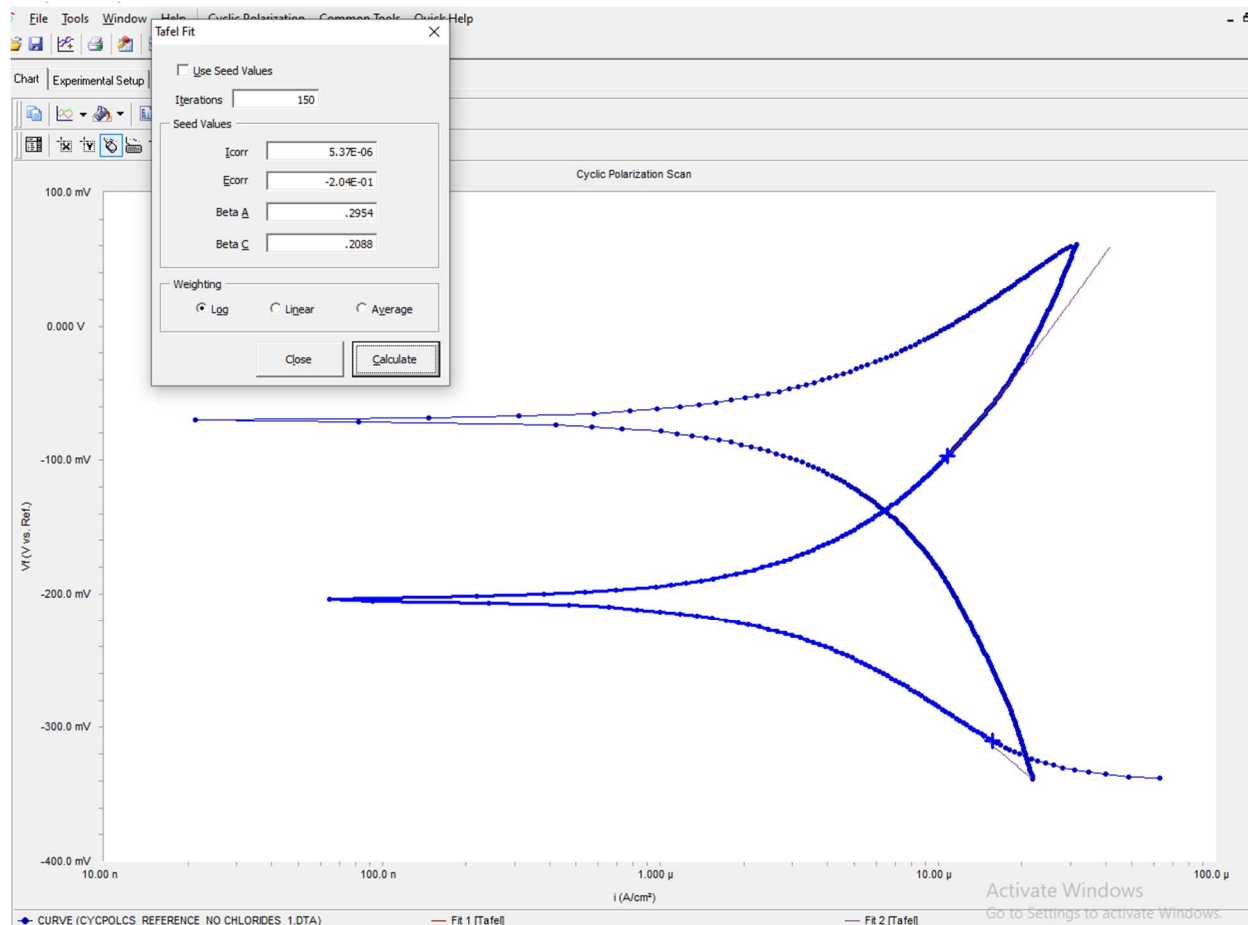


Figure 4: CPP scan with the Tafel fit values for the controlled testing environment. This environment had no chlorides present and yielded an i_{corr} value of 5.37×10^{-6} and an E_{corr} value of -204 mV vs. SCE.

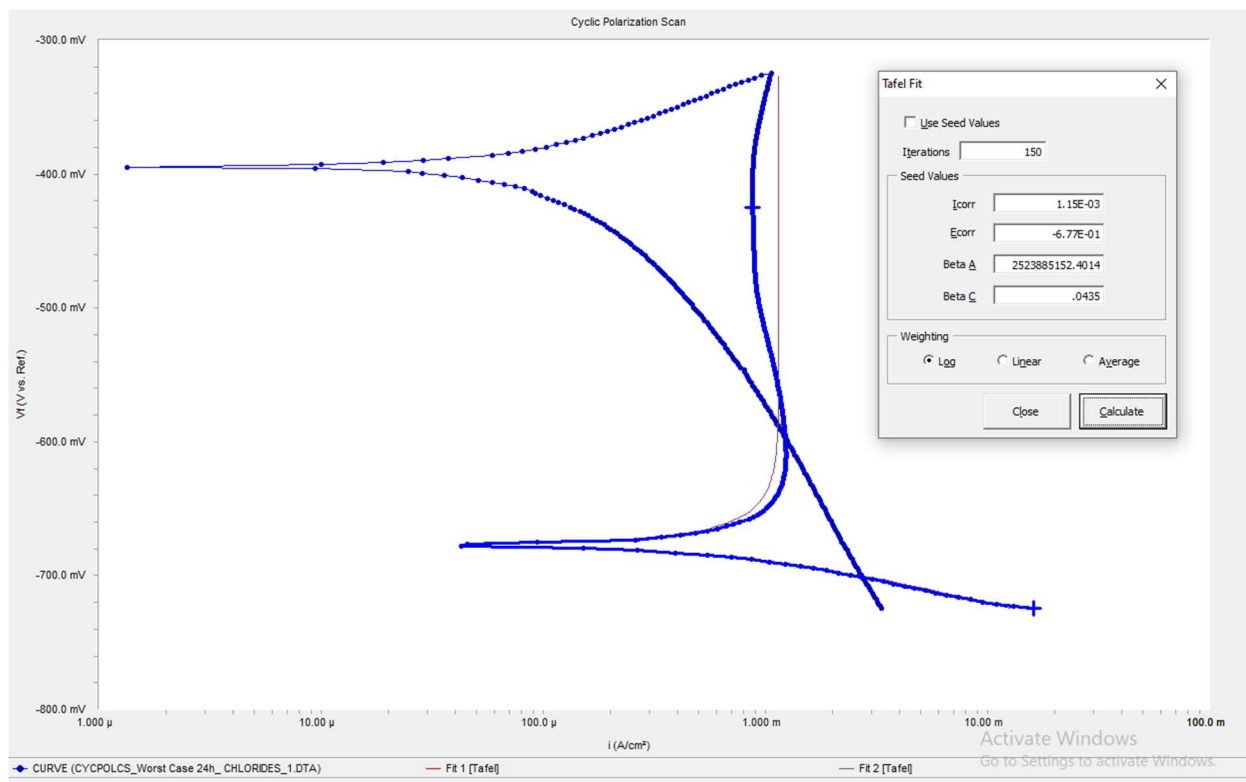


Figure 5: CPP scan and values for the worst-case scenario environment. This test yielded an i_{corr} value of $1.15 \cdot 10^{-3}$ and an E_{corr} value -677 mV vs. SCE. This scan had a corrosion current density nearly 3 orders of magnitude greater than that of the reference test.

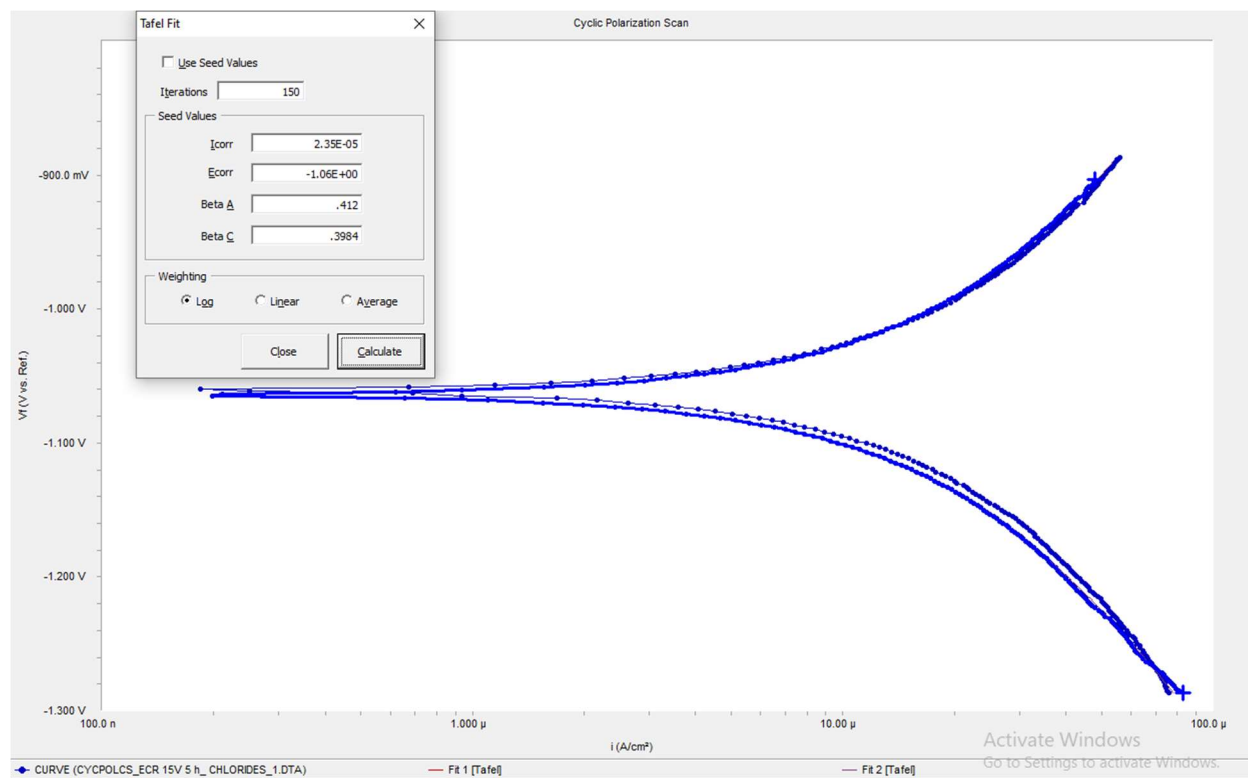


Figure 6: CPP graph for the ECR sample. E_{corr} here was found to be -1.06V vs. SCE and i_{corr} was found to be 2.35*10⁻⁵ A/cm². For this test, the E_{corr} value was found to be more negative than the control and the worst-case scenario situation potentially because of the previously applied negative charging of the rebar sample to repel the chloride ions from attacking. The corrosion current density within this test was approximately one order of magnitude greater than that of the reference test, but two orders of magnitude less than that of the worst-case scenario test.

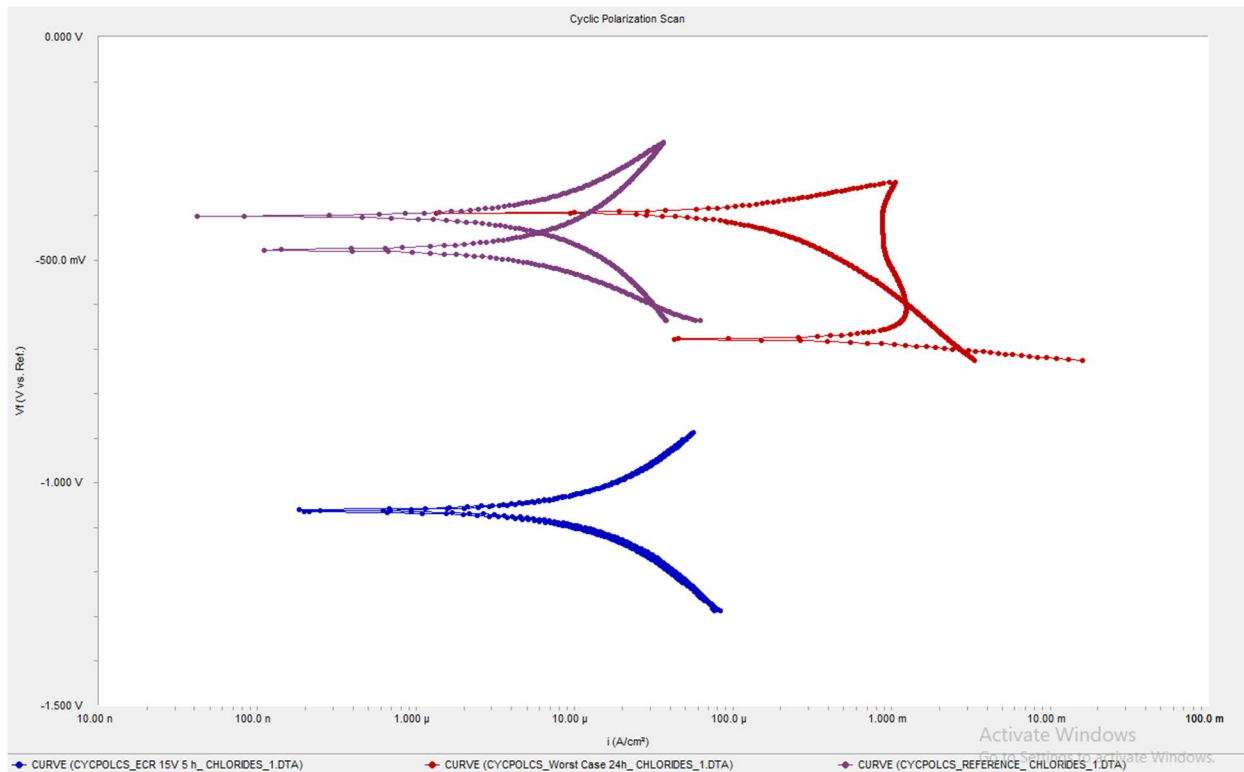


Figure 7: This figure shows the three tests conducted and the CPP scans that were gathered from them. The scan in the top left of the graph, in purple, is that of the reference cell with no chlorides, the scan on the top right of the graph, in red, is that of the worst-case scenario situation, and the scan on the bottom, in blue, is that of the ECR protected sample. As shown here, the icorr values for both the reference and the ECR are like each other, whereas the worst-case scenario has an icorr much greater than the other two.

Open Circuit Potential Measurements-

Open circuit potential (OCP) values were recorded and graphed to check that the system had reached a stable state. This test was completed prior to both the CPP and EIS testing. Ensuring that there was a stable OCP measurement prior to completing the other tests was crucial to making sure that their values were accurate. An example of what this OCP graph looks like is shown below for the reference, no-chloride environment in **Figure 8**.

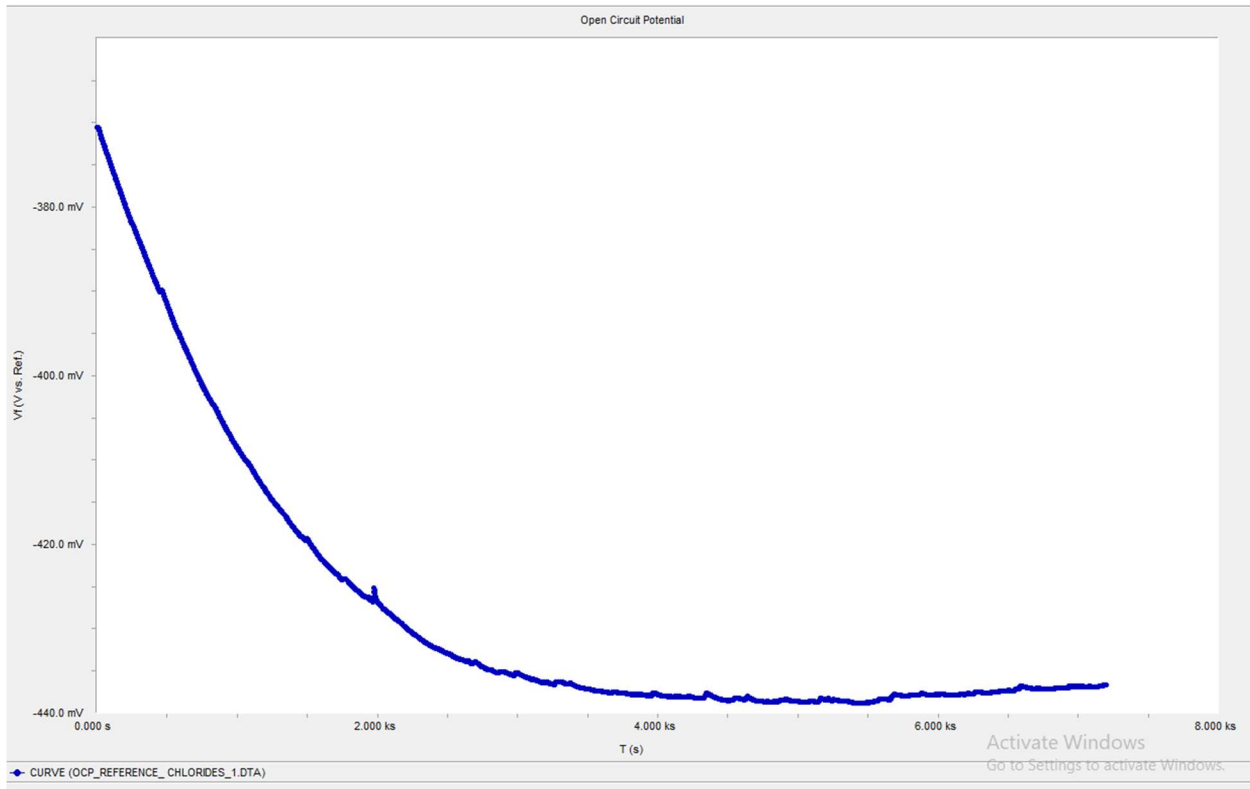


Figure 8: Prior to taking any other measurements or completing any additional tests, OCP testing was conducted on the reference no-chlorides sample. As shown at the end of this curve a semi-steady state was achieved.

Visual Inspections

Visual inspections were done on each sample post-electrochemical testing. These inspections were done by breaking the concrete and releasing the rebar from the middle. Once the rebar was freed, they were cleaned and then inspected to determine if there was any sign of corrosion or pitting. In these examinations, no pitting or extreme corrosion was found on either the reference no-chloride sample, **Figure 9**, or the ECR treated sample, **Figure 13**. Extreme pitting and extreme weight loss was shown in the worst-case scenario sample as shown in **Figures 10-12**.

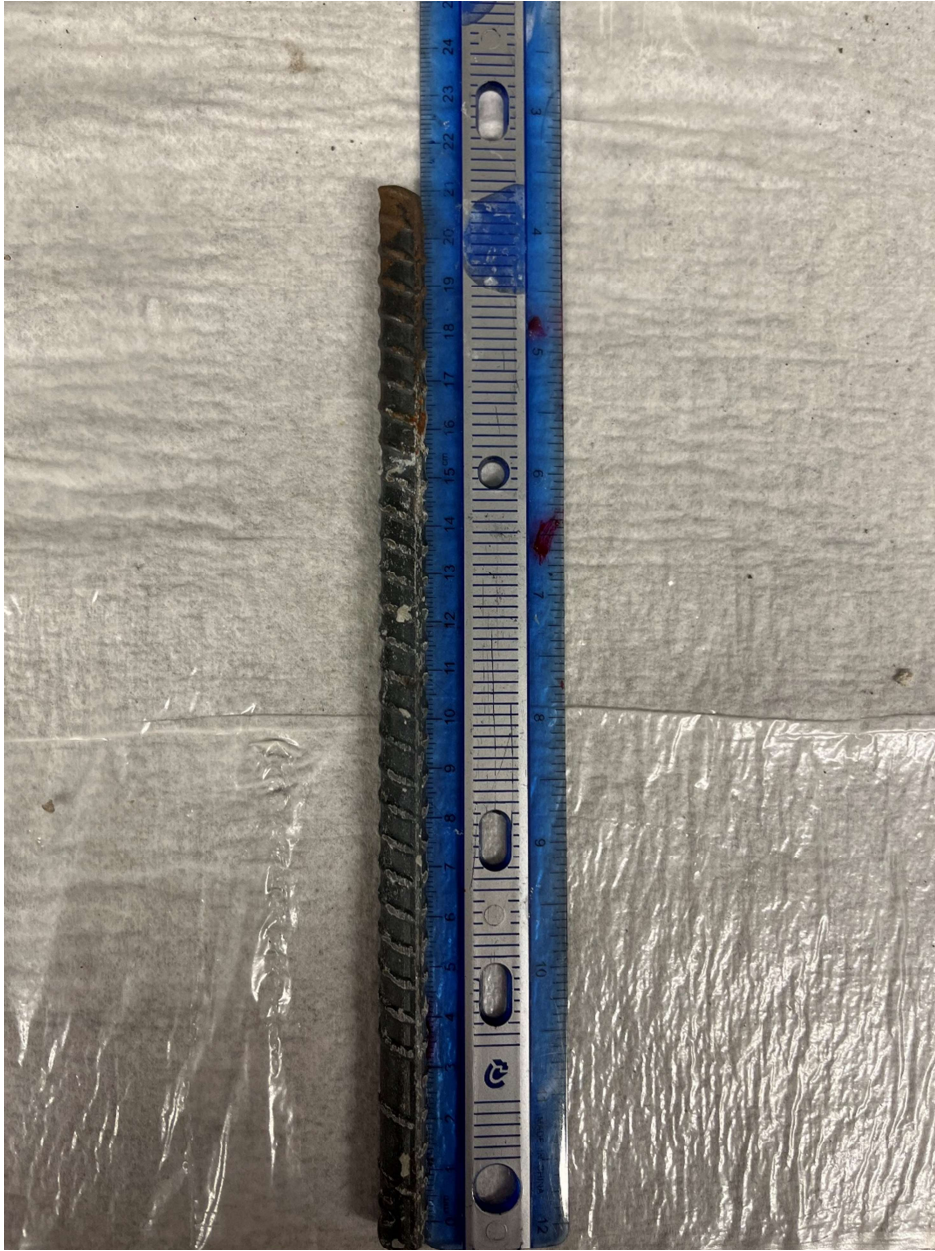


Figure 9: This image shows the rebar sample that was in the concrete mix without any chlorides. This rebar showed no signs of obvious corrosion, both on the bar or on the outside of the concrete.

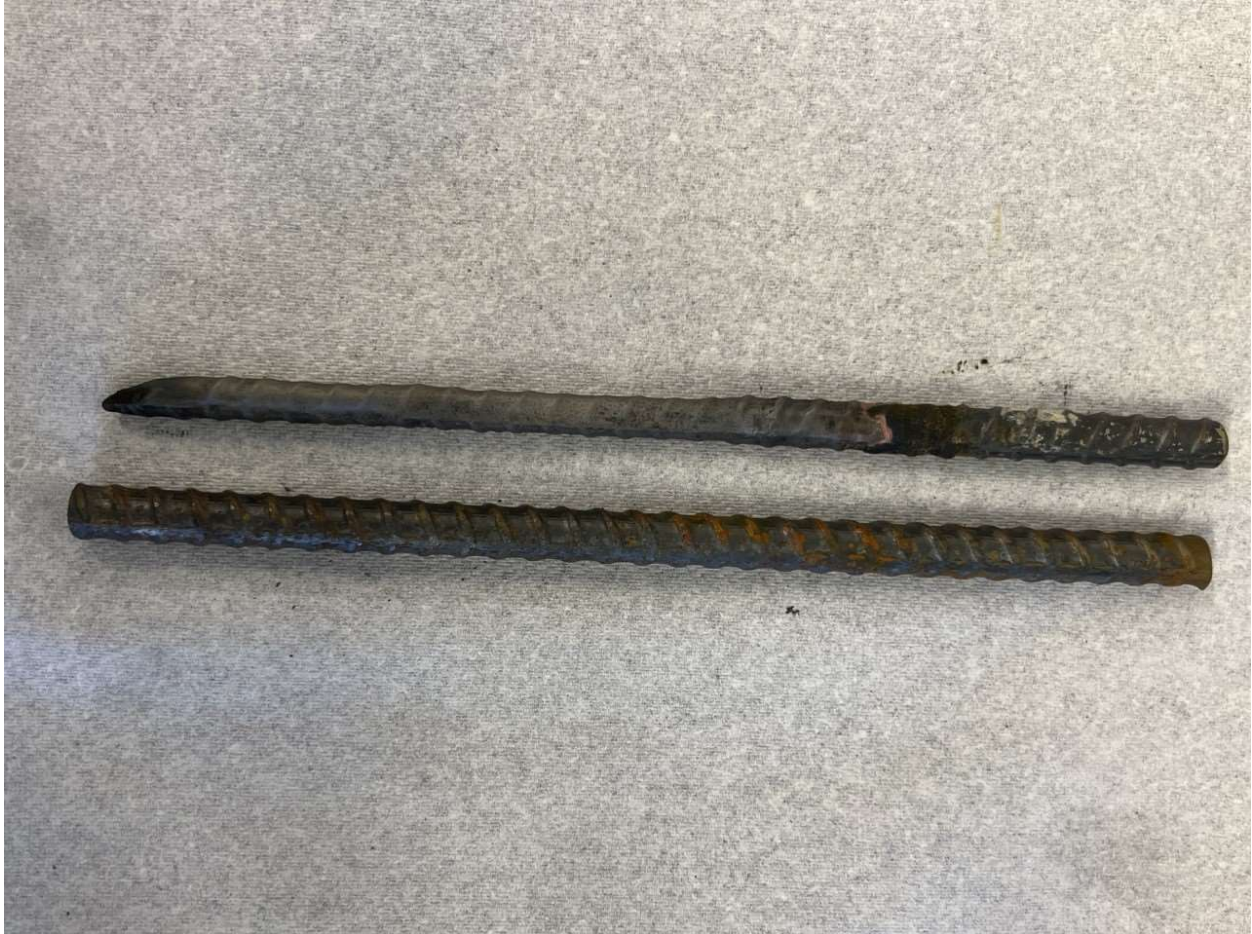


Figure 10: This image shows the extreme corrosion that took place in the worst-case scenario sample (top sample). The rebar on the bottom is the same size as what was initially tested in the worst-case scenario environment. This sample showed extreme mass loss and severe pitting over a 24-hour period.

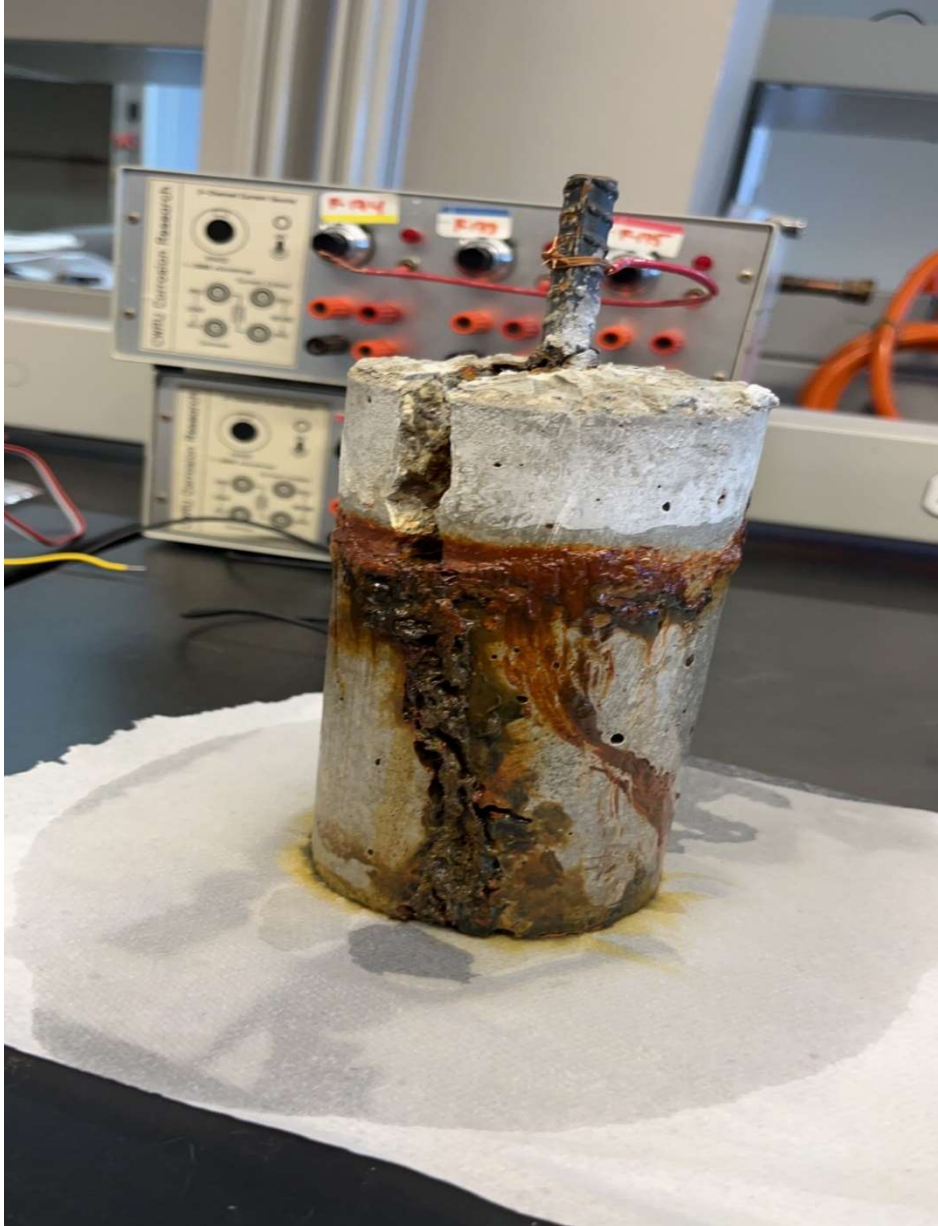


Figure 11: This image shows what the worst-case scenario looked like as soon as it was taken out of the testing environment. A severe crack can be seen in the concrete with corrosion product filling that crack. This observation supports the suggestion that corrosion on rebar within concrete will lead to concrete failure.



Figure 12: This image was taken shortly after the image from **Figure 12**. When this sample was picked up to be cleaned, a crack that was initially not visible on the backside of this specimen cracked all the way through. This led to the concrete looking like the picture above with two halves of the initial concrete cylinder, both with corrosion product staining inside. This failure supports the idea that extreme corrosion within reinforced concrete will not only lead to failures of the reinforcements, but also total concrete failures leading the structure to collapse under any additional stresses.

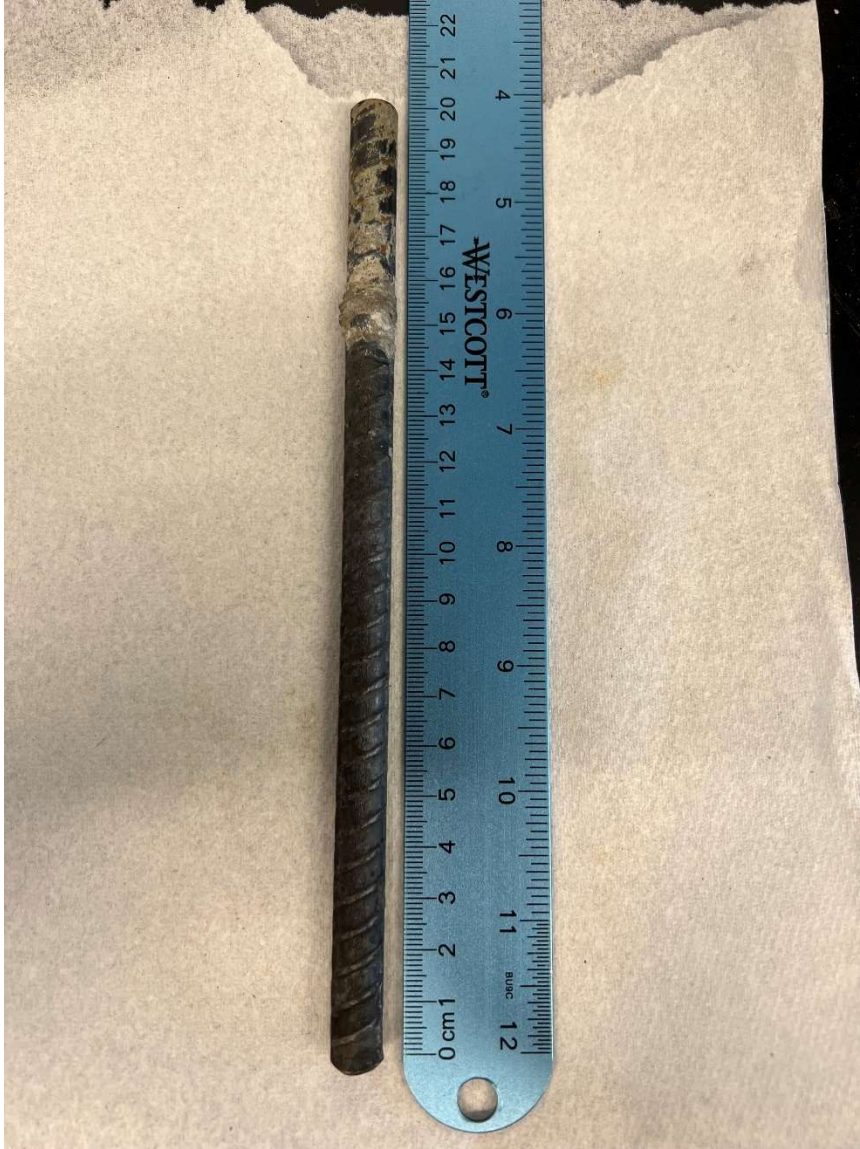


Figure 13: This image shows the rebar that received the ECR treatment. This sample showed no signs of corrosion, both on the concrete and on the rebar itself. This specimen also had chlorides within the concrete which could have caused corrosion, but the ECR treatment prevented this from happening. The concrete encasement of this sample was found to still be very strong with no obvious signs of imminent failure.

Discussion/ Analysis

The ECR that was applied to the rebar sample improved its electrochemical performance by assisting in the removal of chlorides. This treatment was able to repel chlorides from the concrete by negatively charging the rebar sample which in turn repulsed the negatively charged chloride ions. As these chloride ions were being repulsed from the rebar due to them both having a negative charge, the steel mesh, that was placed such that it surrounded the concrete sample, was being positively charged. This repulsion from the rebar and attraction to the steel mesh helped to prevent the chlorides from actively attacking, pitting, and corroding the rebar sample within the concrete. This treatment provided substantial benefits to protecting the rebar in very short amounts of time. When a worst-case scenario situation was studied in which the rebar was positively charged and the steel mesh was negatively charged, extreme pitting was observed and excessive corrosion products caused the concrete sample to split into two pieces. Although this was an accelerated situation, this failure was what has been witnessed in bridges and buildings across America. ECR should be explored whenever chloride attacks may be suspected within a reinforced concrete specimen.

Using the corrosion current densities that were found during the electrochemical tests, a corrosion rate for each sample was able to be calculated. The equation to convert from the corrosion current density to a corrosion rate is as shown in **Equation 7**. Using **Equation 7**, the corrosion rates were able to be calculated for each scenario and are presented in **Table 2**. As shown within the table, the scenario with the lowest corrosion rate was the reference with no chlorides, followed by the sample that had received ECR treatment, and finally the worst-case scenario. The ECR treated sample had a corrosion rate approximately 50 times smaller than that of the worst-case situation.

$$CR (mpy) = 0.129 * icorr \left(\frac{\mu A}{cm^2} \right) * \left(\frac{EW}{\rho} \right) \quad (7)$$

Table 2: Corrosion Rates for Each Scenario

	icorr ($\mu A/cm^2$)	CR (mpy)
Reference- No Chlorides	5.37E+00	2.453472
Worst Case	1.15E+03	525.4178
ECR	2.35E+01	10.7368

While a worst-case scenario may not be what is seen in the field all the time, ECR can be expected to reduce the chloride content of the cement by nearly 40-45% which would reduce even an average corrosion rate substantially [11]. ECR considers a constantly applied potential to the rebar specimen but applying this potential intermittently over time has been found to potentially decrease the chloride concentration within the concrete by up to 70% while also proving beneficial in reducing potential side effects [12].

Marketing

Proving that the ECR process is effective and efficient within this study results in extreme marketability of this process to companies and government agencies which are looking for corrosion treatment methods in these environments. ECR treatments do not require equipment to remain on any structure once completed. With no equipment needing to remain on the structure, there is no maintenance that needs to be completed on the ECR system itself, unlike something such as a cathodic protection system, and the original look of the structure can be preserved. Many groups want to maintain the look of old bridges and buildings that were constructed using the reinforced concrete method and ECR treatments to these structures can ensure that they remain as

they were prior to its application [10]. The findings within this study need to be confirmed and published by AMPP (Association for Materials Protection and Performance) prior to its marketing. Continued research should be done in this subject to improve upon the effectiveness research as well as testing in different and more aggressive environments. This study should be used informally as a means of treatment suggestion to companies that may be able to provide the ECR to a given environment.

Scale-Up/ Economics

Electrochemical chloride removal poses increasing applications to the industrial and commercial world as our buildings and structures become dated and are approaching the end of their lifespan. ECR can be applied to a reinforced concrete structure and address the cause of corrosion in the system. Because of the way this process removes the chlorides in the system and helps to create an environment that will allow for passivation of the carbon steel structure, it can be applied and then have the entire system removed from the structure. This results in the cost of this process drastically decreasing as it does not require routine maintenance and monitoring as some other processes do. In addition to the fact that this system can be removed upon completion of treatment, this treatment can result in a 50-to-100-year lifespan extension prior to re-receiving the ECR treatment. This mitigation treatment method saves millions of dollars in rebuilds and reduces the risk for loss of life in a failing building or bridge. Preventing reinforced concrete failures with this treatment method could save the United States millions, if not billions, of dollars every year.

Acknowledgements

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