

**2019-DC/SPG (IN-HOUSE) CORROSION TESTING
nCT; Penetration with Iodide (ASTM C1152 Modified),
Half Cell/Migration (ASTM C876)
& Cracked-Beam Ponding (G109 Modified)
- Testing & Results
(DRAFT)**

October 15, 2019

HYPOTHESIS – In an effort to illustrate the substantial benefits of a complex nano-silica admixture (Vapor Lock 40/40) in addressing both the Initiation and Propagation phases of (primarily chloride) corrosion in structural/reinforced concrete, we have modified several ASTM and AASHTO protocols as follows. Along with the results, we aim to ultimately illustrate the benefits relative to other commercially available concrete admixtures and pozzolanic choices; making this a performance and value proposition-based decision.

BACKGROUND - We begin with PCA's premise of "Low Permeability" concrete and proper concrete cover being the best prescription to combat steel corrosion in concrete. From almost the beginning of kiln-fired (Portland) cement-based concrete, there has been an effort to achieve surface densification by adding various types of silicates. By applying silicates to the surface of concrete within the first 48 hours of concrete placement, we've attempted to lower shrinkage, water/damp proof, impart enhanced abrasion and densification characteristics, and improve final surface tolerances, all to varying degrees of success. The majority of these silicates are the product of silicon dioxide manipulation, with the premise being a pozzolanic reaction with calcium hydroxide and water, creates relatively large amounts of additional calcium silicate hydrate (c-s-h) gel. The greatest advancement to this technology has been the careful manipulation of silicon dioxide (temperature and pH level) to create very small (Colloidal) silicate formation. This additional c-s-h is the main vehicle of enhanced concrete characteristics and the impetus to move beyond just surface modification and towards an Integral approach with relevant technology throughout the entire concrete matrix. This is the basis for the development of Vapor Lock 40/40 – *a complex Nano-Silica Integral concrete admixture*.

For over 11 years, I have been personally involved with the commercial sales and service of a similar formula (Vapor Lock 20/20, the basis of the 40/40 admixture) in the Moisture/Vapor Reduction Admixture (MVRA) marketplace; or Integral Silicate Admixtures. The standard for the industry is ASTM D584 – Coefficient of Permeability. With the Vapor Lock 20/20 admixture dosed adequately, I have never seen a result above 0.174 US Perms (and usually in the 0.02 US Perm range). I am able to consistently document the Ultra Low Permeability of Vapor Lock enhanced concrete (available under separate copy). I have been part of almost 300 mix designs that have combatted moisture/vapor issues, indoor air quality, shrinkage reduction, general water proofing and corrosion inhibition. I have applications in flooring, architectural coatings, roofing, water proofing membrane replacement/reduction, and shrinkage reducing admixture projects by the dozens; and totally in the hundreds. Over time, we have proven Vapor Lock to be an effective admixture in **guaranteeing construction critical paths** and **eliminating risk** involved with the installation of all the previous applications. As this technology has proven successful in thousands of applications and millions of square feet of flooring and roofing, throughout North America, in perfect service to date, the Integral

Silicate Admixture technology has been expanded to a number of other applications all with increasing scope and performance. It is to the satisfaction of ASTM C1582 - Standard Specification for Admixtures to Inhibit Chloride-Induced Corrosion of Reinforcing Steel in Concrete, that we address Vapor Lock 40/40 in inhibiting corrosion two ways:

- I. The *Initiation phase* of chloride corrosion - by illustrating a concrete structure with a **permanently, severely disrupted capillary system**. Through this mechanism, we will illustrate the reduction in the migration of deleterious material (transport mechanism) through the concrete matrix, making the steel reinforcement (rebar) less vulnerable.
- II. The *Propagation phase* of chloride corrosion - by illustrating a second mechanism (introduced in conjunction with the Integral Silicate Admixture) of a migrating, robust film coating that is attracted to all ferrous material in the plastic stage of concrete – a physical layer of steel protection in concrete (focusing on the area around a crack, where steel is most vulnerable).

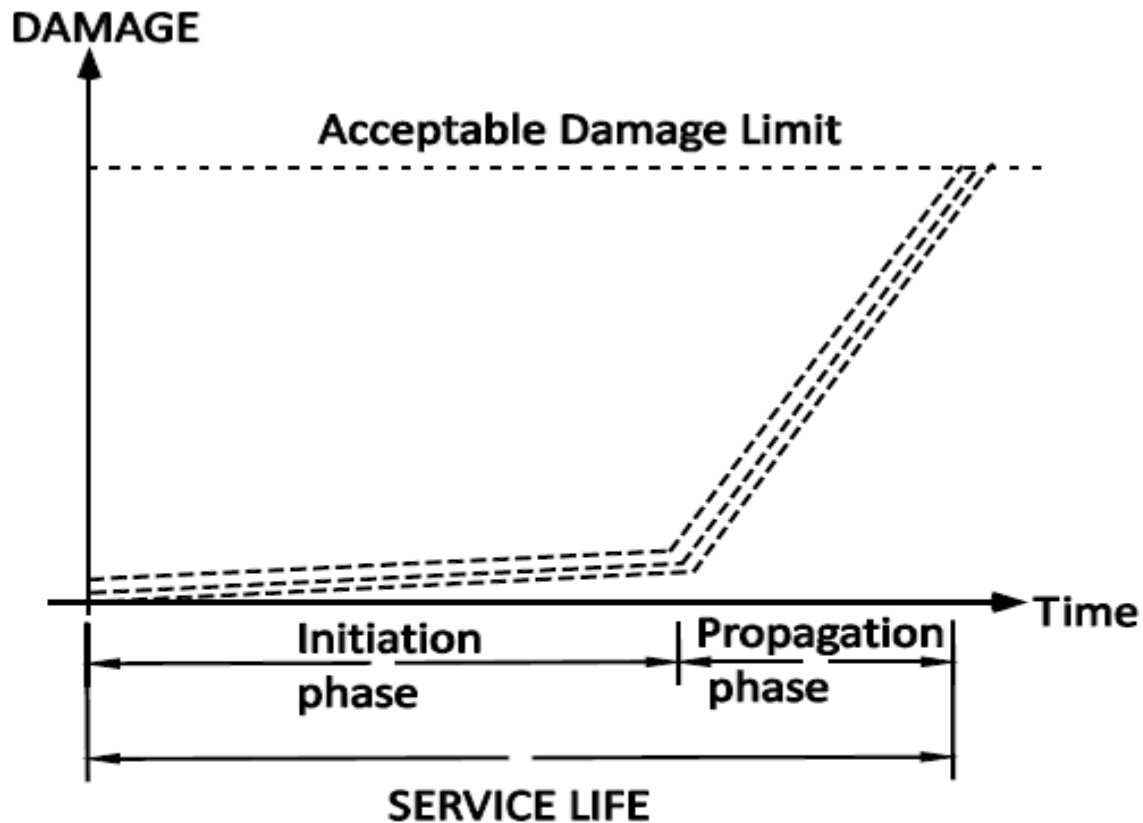


Figure II-5 Conventional Schematic of the Service Life of a Reinforced Concrete Structure.

RESULTS –

We have reached out beyond the Coefficient of Permeability results (numbering well over a thousand individual, documented tests), with ASTM C1152 & ASTM C1218 – Chloride (Acid & Water Soluble)

Testing for Concrete & Mortars - Modified. These original standard test methods of taking pulverized sections of concrete (moving your way down a ponding or injected specimen), adding nitric acid or water (respectively), boiling that mixture and measuring the chloride content produce large swings in results, with chlorides pulverized from aggregate being included; which we know have little intent or purpose on the corrosion of steel. Plus, some chlorides are bound by cement hydration product and aren't a factor in steel corrosion either. For these reasons, we have adopted Dr. Tyler Ley's **Nano Computed Tomography (nCT); Penetration with Iodide** - Standardized test method using Iodide ion solution (similar characteristics to chloride ions) to penetrate concrete and at different time intervals take an X-Ray of the Iodide. Fast, easy way to illustrate and quantify liquid penetration into 1" diameter, by 1" deep concrete specimens. We feel this protocol is a logical and practical *modification* from the standard ASTMs C1152 and C1218 – Chloride (acid & water soluble) Testing for Concrete & Mortars and shows greater agreement and precision consistently. Although not currently readily available from most commercial labs, it can be performed relatively quickly and economically at the University engineering program level. Protocol is under separate copy.

Mix Designs

4500 psi, Pump Mix

1" top size agg., w/c = 0.45

*Sand is a blend of local and Orca (Vancouver/Polaris Materials) sand.

**This was pre-project testing for Kaneohe-Kailua Wastewater Conveyance and Treatment Facilities Project; Oahu, Hawaii.

Materials	Weights
1" Aggregate	1,053 lbs.
3/8" Aggregate	708 lbs.
Sand	1,393 lbs.
Cement	630 lbs.
Water	283 lbs./34.0 gallons
MasterPozz 322	25.2 ounces
- 4 ounces per hundred weight	
Control C Mix - amine salts/carboxylic acids admixture	24 ounces
Control V Mix - Vapor Lock 40/40 admixture	63 ounces

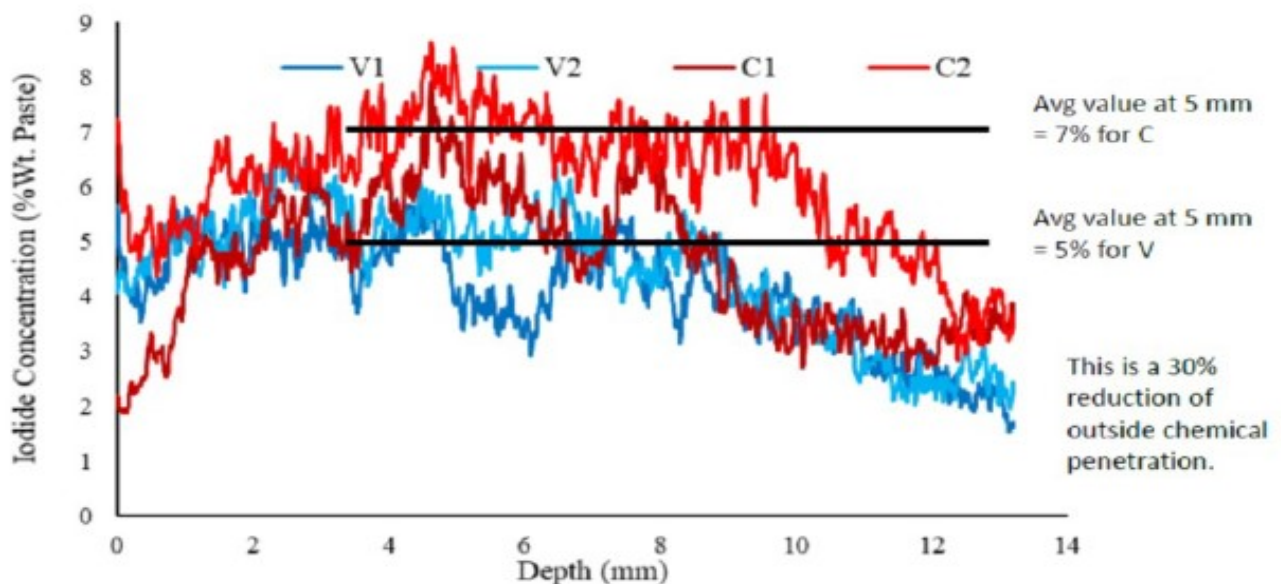
* It's important to note this mix utilized imported Polaris/US Concrete's Orca sand blended with local sand. This is the same sand imported to Southern California to meet current MOE specifications in high-rise construction.

** Relatively High-Performance Mixes at around 60 days showed a 30% reduction in surface/shallow penetration.

***Complete tests at https://docs.wixstatic.com/ugd/925158_71c25f21c24d4aa3aa0be9b6b1772d66.pdf

Overall Comparison

Concentration profiles of all the samples after 10 days of ponding.



Summary

- Two cores were tested from each cylinder.
- Both cores showed good agreement with each other for sample C and V.
- There is a 30% reduction at 10 days of exposure for sample V when compared to C. Similar results were found at 5 days of exposure.

RESULTS, Continued - The severely disrupted capillary system of Vapor Lock enhanced concrete can be inferred by first, the hundreds of Coefficient of Permeability results in the 0.02 US Perm range (1,000s on a National level). Secondly, by the superior reduction in chemical surface penetration of potassium iodide relative to the controls (above).

The third illustration of the disrupted capillary system should be measuring/judging the “transport mechanism” in a concrete or mortar; how much deleterious material can move through the paste to negative, harmful affect. This has been established previously by physical ponding protocols; numerous variations of G109 protocols, including the Southern Exposure test, Cracked Beam tests and variations thereof. And in conjunction with, or in lieu of, “electrical resistivity” of a concrete or mortar; usually involving an electrical current. Examples include ASTM C1202 – Rapid Chloride Permeability, ASTM C1556 - Bulk Diffusion, Accelerated Chloride Threshold (ACT) tests and **ASTM C876 - Standard Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete.** This last protocol measures resistance between a rebar connection and the surface of the concrete (with a constant, measuring copper sulfate probe) and is considered one of the most effective and true gauges of “transportability” (deleterious migration) in concrete. This is also referenced as Half-Cell testing, and our protocols and results are summarized below.

*This was “In House” testing performed by consultants and local ready-mix providers in the Southern California area; with modified protocols and procedures suggested by WJE and other professionals. Testing & Results stand on their own, and will likely be reproduced by a commercial lab and/or engineering department upon further review for good agreement.

**The ASTM C876 protocol was based off of congruently run Cracked-Beam Ponding specimens for corrosion inhibiting testing; specimens were constructed around that protocol/testing procedure and measured during the corrosion process with the Half-Cell instrument.

The Mix design tested; and six industry accepted variations are below –

Compressive Strength: 4,500 psi at 28 days, w/cm Ratio: 0.45, Slump: 4.00 +/- 1 inch

Cement Type II/V – 658 lbs.

Water – 300.4 lbs. (36 gallons)

1” x #4 – 1,392 lbs.

3/8” x #8 – 310 lbs.

Washed Concrete Sand – 1,393 lbs.

WR-91 Type A – 26.32 oz/yd³

Air – 0.27 (1%), 149.8 lbs./ft³ Plastic Unit Weight, 4,045 lbs., 27.0 ft³

(Aggregate Gradations available upon request)

Specimen X - Crystalline Growth mix; (new) concentrated dry powder added at 1.25% of cement weight (this product replaces the previous 2.5% by weight commercial product).

Specimen D - Calcium nitrite mix; popular admixture with 30% calcium nitrites at 5.5 gallons per yard.

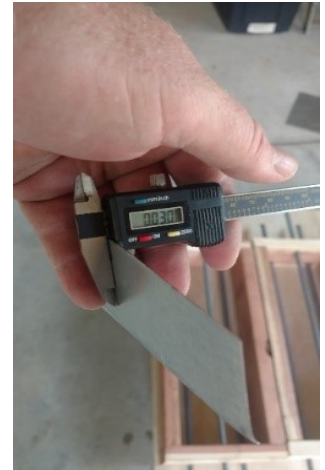
Specimen F - 20% fly ash mix; with a direct 20% replacement of cement.

Specimen V - Vapor Lock 40/40 mix; 10 ounces per hundred weight of cement.

Specimen VL - Vapor Lock 40/40 with lightweight sand; 10 ounces per 100 weight of cement plus 22% replacement of sand with lightweight sand (provided by Arcosa Lightweight; SoCal source).

Specimen C - Plain Control – straight mix, above.

Specimens were made in the shape to simulate common floor/roof deck assemblies; 24" long, by 8" wide (3 to 1 to help promote mid-point cracking) off of 2x6s (5.5" nominal) thickness. *The beams were made with a vibrating stinger, wood strike/float, mag troweled, and steel rounded edges around the entire specimen. There was ***no ability to stand on the specimens with a power-trowel***, which would be assumed to be common finishing practice; and should be taken into account. Three number 4 bars (1/2" diameter) placed in a standing triangle pattern, with the top bar being 1" from the surface. Steel was purchased from a standard building material center and not cleaned or treated in any way. A 0.03-inch (1/32") steel shim was placed mid-point (12") across the specimen, laying on the top #4 bar for four hours, and then removed. This is to provide a "weakened" plane to mimic a small, acceptable crack and expedite corrosion locally.



After the beams were cast, 3-mil black poly was laid over the specimens and weighted down with wet sand for 7 days to provide a "wet cure". After seven days of common curing (wet cure), the specimens were stripped of forms and air dried for 24 hours. At Day 9, four-inch-high plexi-glass reservoirs were adhered to the surfaces with silicon; 6" wide and 16" long. This was to produce ponding at roughly half the surface area of the specimens.



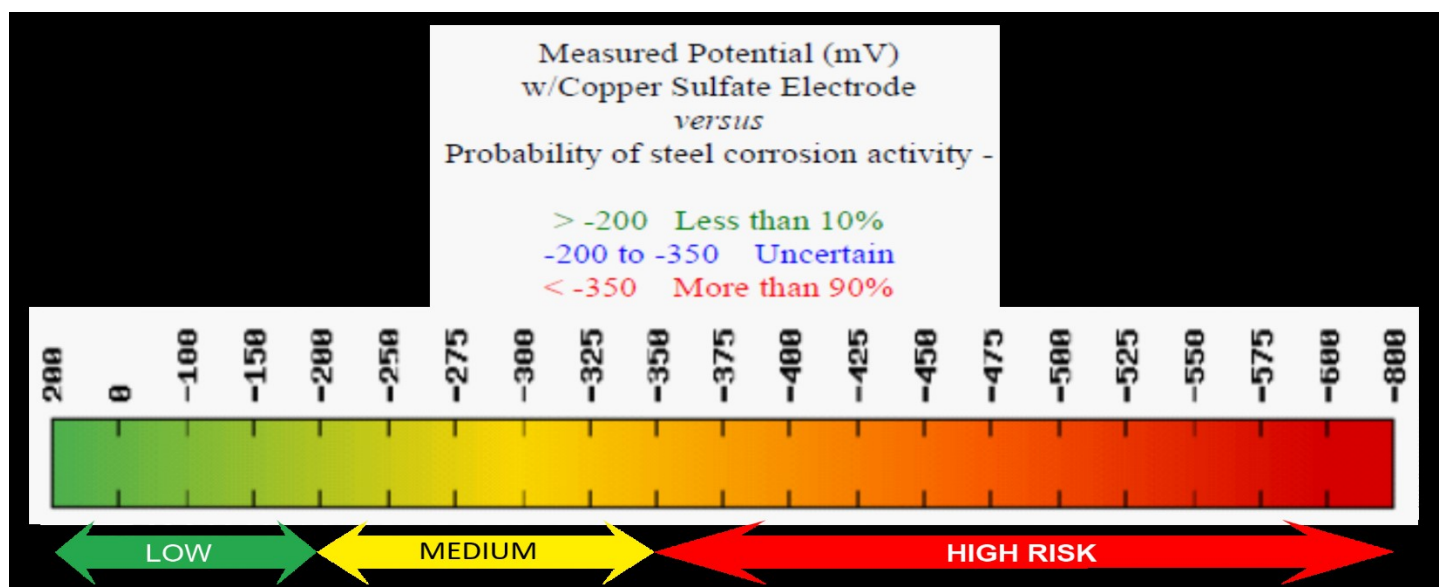
At Day 9, a 20% salt water bath was added to each specimen at approximately 1" deep. Ponding Protocol – Specimens are out in the open and have semi to direct sun on them, with full thermal night and day cycles. With April weather in Southern California, they are getting about a 30 to 35 degree

swing in temperature; should hold constant throughout the year. Wind and rain are sporadic. Monday mornings, a new 20% salt solution is added to a depth of 1" for each specimen. It might take all week to evaporate (April, May) or it might take a day and half to evaporate (July, August). Once a month, the inside of the reservoirs are rinsed out with water to remove the salt build-up. *The tap water is heated to approximately 115 degrees Fahrenheit. 20% (by weight) fine salt crystals are added and agitated/stirred for approximately 5-7 minutes. It is thought that the increase in water temperature should allow for the highest saturation levels.

At the 150-day mark, specimens will be deconstructed in such a way as to expose for inspection as much of the top piece of rebar as possible. Physical inspection should be enough to properly discern actual damage caused by salt water ponding with most of the damage assumed to be nearest the induced crack (mid-point of the top bar) and exposed ends where rebar sustained rust production. *We expect all specimens to sustain varying degrees of rebar damage from the quick and severe chloride exposure. Our inspection will lend some subjectivity to rate the damage sustained by each specimen.

ASTM C876 - Standard Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete. In essence, a basic Half Cell instrument (Miller LC-4.5 voltmeter & wide head copper sulfate probe able to measure 360 degrees) is used to ascertain the "resistivity" (ability of deleterious material to migrate) throughout the concrete between the steel and the surface; a scientific determination of corrosion activity. Every 7 days starting at Day 1 (Day 9 actual) there are three readings taken inside each full reservoir. Because of the relatively small service area being mapped and 2.5 inch, radial probe head, numbers are usually constant or less than 0.2% change (i.e. -250 mV vs. -252mV max.) in value at different areas. Per this protocol, concrete specimens can be taken at any time during the life cycle of a structure and there is no finite time period or number of readings needed to be taken; only the ability to have a complete electrical circuit (i.e., access to the rebar).

The Scope and Significance of Use are summarized with current results.



ASTM C876 - Standard Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete

(Summarized)

1. Scope

1.1 This test method covers the estimation of the electrical corrosion potential of uncoated reinforcing steel in field and laboratory concrete, for the purpose of determining the corrosion activity of the reinforcing steel.

1.2 This test method is limited by electrical circuitry. Concrete surface in building interiors and desert environments lose sufficient moisture so that the concrete resistivity becomes so high that special testing techniques not covered in this test method may be required (see 5.1.4.1). Concrete surfaces that are coated or treated with sealers may not provide an acceptable electrical circuit. The basic configuration of the electrical circuit is shown in Fig. 1.

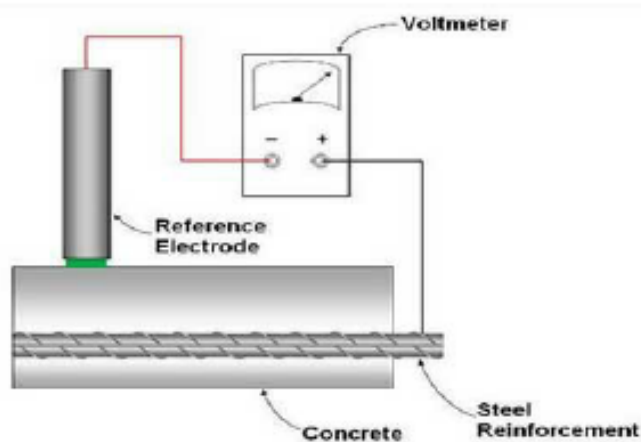


Figure 1

4. Significance and Use

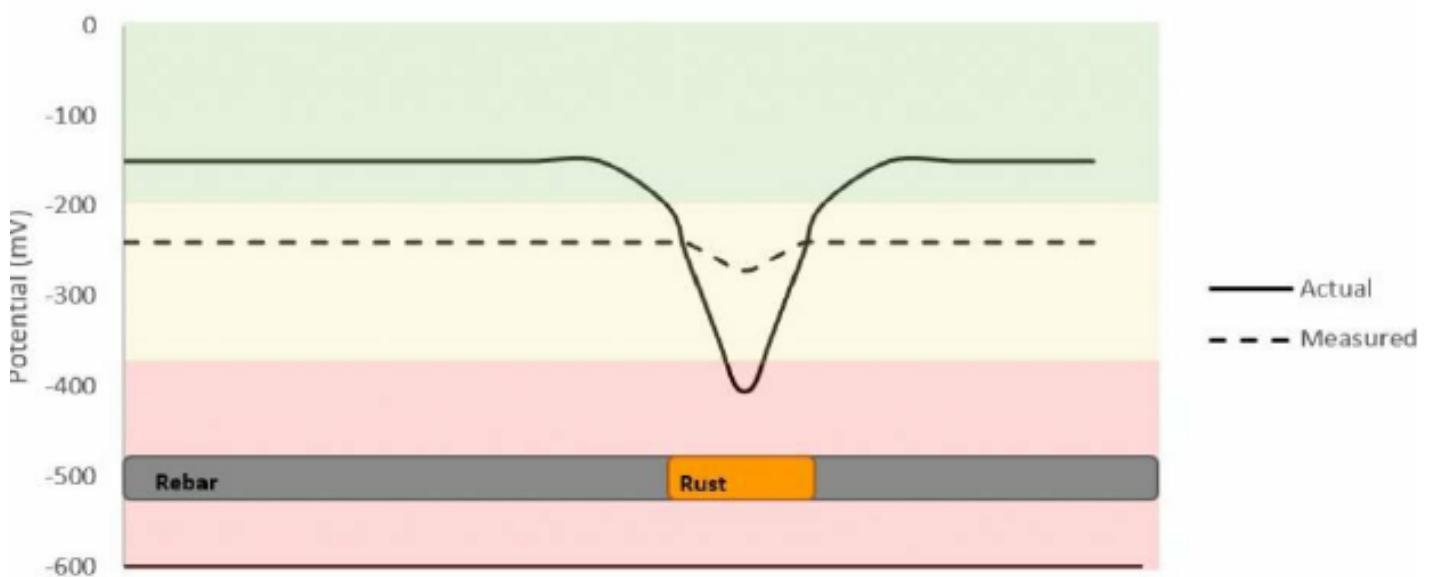
4.1 This test method is suitable for in-service evaluation and for use in research and development work.

4.2 This test method is applicable to members regardless of their size or the depth of concrete cover over the reinforcing steel. Concrete cover in excess of 3 in. (75 mm) can result in an averaging of adjacent reinforcement corrosion potentials that can result in a loss of the ability to discriminate variation in relative corrosion activity.

4.3 This test method may be used at any time during the life of a concrete member.

4.4 The results obtained by the use of this test method shall not be considered as a means for estimating the structural properties of the steel or of the reinforced concrete member.

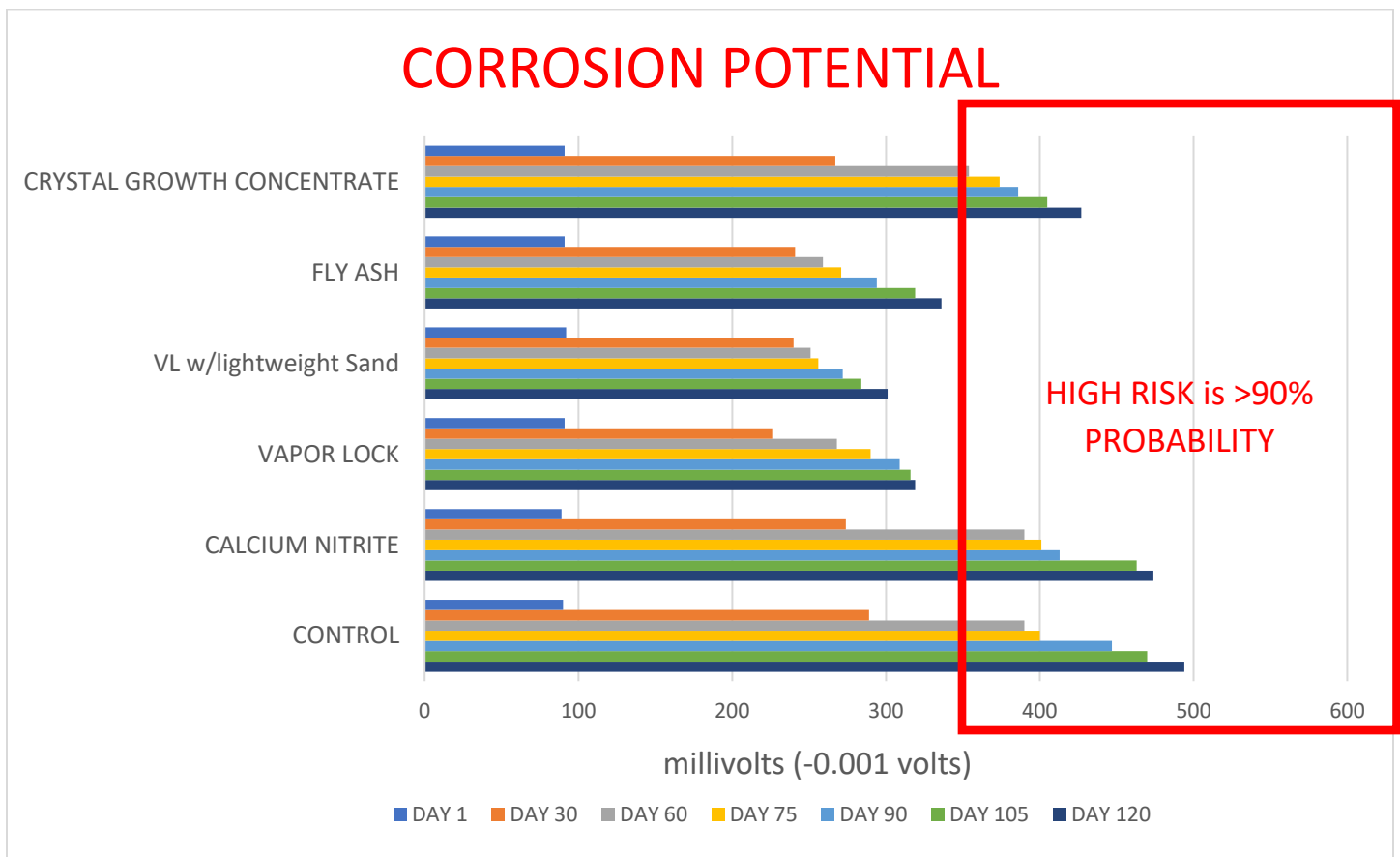
4.5 The potential measurements should be interpreted by engineers or technical specialists experienced in the fields of concrete materials and corrosion testing. It is often necessary to use other data such as chloride contents, depth of carbonation, delamination survey findings, rate of corrosion results, and environmental exposure conditions, in addition to corrosion potential measurements, to formulate conclusions concerning corrosion activity of embedded steel and its probable effect on the service life of a structure.



Actual corrosion rate is usually greater than half-cell readings.

Half Cell Readings

<u>(average of 3 readings, - 0.001 volts)</u>	Day 1	Day 30	Day 60	Day 75	Day 90	Day 105	Day 120
X (crystal growth, concentrate)	91	267	354	374	386	405	427
D (calcium nitrite)	89	274	390	401	413	463	474
VL (Vapor Lock & lightweight sand)	92	240	251	256	272	284	301
F (Fly Ash)	91	241	259	294	294	319	336
V (Vapor Lock)	91	226	268	290	309	316	319
C (Control, Plain mix)	90	289	390	400	447	470	494



***Per ASTM C876, readings below -350 millivolts move into the “High Risk” area with over 90% Probability of Steel Corrosion occurring.** The graph leaves out the negative, falling millivolts. Numbers/readings are actually getting smaller (drops in millivolts).

****Figure 2 (below) shows how the surface may show readings in the -200 to -300 millivolt range, and the affected spot of the rebar is closer to -700 millivolt; a factor of 3 to 4 times. Also, the small area being measured within the ponding reservoirs facilitates only three, similar readings (within 0.2% delta) because of the wide head and the 360-degree sensitivity on the copper sulfate measuring probe and relatively small area being measured. These probes are designed for in-service, large parking structures and similar structural decks.**

*****Further examination shows substantial deviations in saturation levels of the specimens when the readings are actually taken. We’ve found this to be true; about -100 drop in millivolts when long-term super-saturated sample readings. To stabilize this effect, all period readings are taken approximately 30-45 minutes after weekly salt bath is added. This would be considered “saturated” readings; but not “super-saturated” readings.**

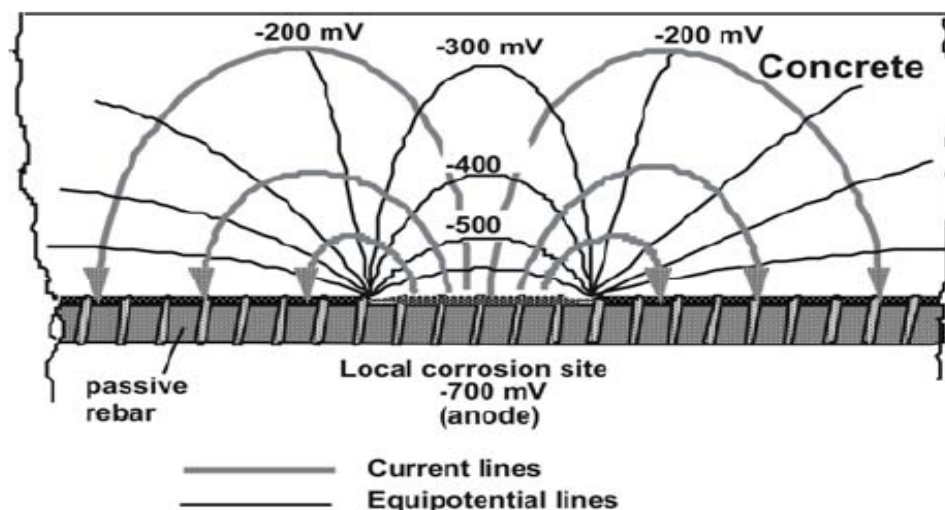


Fig. 2 - Schematic view of the electric field and current flow in an active / passive macrocell on steel in concrete.

RECOMMENDATIONS & CONCLUSIONS

With respect to PCA’s prescription of “...a low permeability concrete...” to combat steel corrosion in concrete, the Chemical Penetration protocol put forth by Dr. Ley (modified ASTM C1152) works intuitively towards accuracy, speed and the ability to compare specimens with admixtures. In conjunction with 1,000s of Coefficient of Permeability test results (ASTM D5084) in and around the 0.02 US Perm area, the ability to provide a relatively quick and permanent capillary break (low permeability/permeance) seems apparent and can be inferred to drastically slow the *Initiation* period of corrosion.

In regards to the second *Propagation* period of corrosion, we can infer certain characteristics from the *Cracked Beam specimens* (prior to autopsy) with the help of a Half-Cell/Voltmeter. The “off-the-shelf”,

non-enhanced rebar protruding from both ends of the specimens and the relatively harsh/aggressive ponding (20% salt solution) quickly moved all specimens into the 'Medium Risk' (yellowish zone) by Day 30. From there, the specimens move apart in regards to the Probability of Steel Corrosion Activity; with the Vapor Lock specimens and Fly Ash specimen showing superior readings; below -350mV at Day 120.

Regarding both the “conventionalism” of the test specimens and Half-Cell protocols, we can only look at the latest recommendations from ASTM G01 – Corrosion of Metals, sub-committee 14.02 – in Concrete & Mortar and their latest prescribed changes to G109, including:

- *leaving scale on rebar to promote greater cathode activity,*
- *using 10 ohms resistance for improved voltmeter sensitivity and more acute low-end resistance.*

Both were incorporated in the above protocols; steel was purchased from a local building supply company and not cleaned in any way. Also, the millivolt (one thousand of a volt) measurements above, were all from the 10 ohms resistance setting prescribed in the newest changes of the sub-committee.

The crystal growth and calcium nitrite specimens performed similarly to the Control (plain) specimen; into the “High Risk” area by the end of the second month. The Vapor Lock mixes performed similarly (if not slightly better) than the fly ash specimen; all three specimens proving to be excellent in “resisting” corrosion potential as exhibited by the Half Cell results. Their “similar mechanics” of creating additional, enhanced c-s-h, superior cement product, and a significant “Disruption” of the capillary system are all highly attractive for both the *Initiation* and *Propagation* phases of corrosion. Our ultimate goal, would be the greatest increase in *Time (and thus Service Life)*, relative to the Initiation phase and Propagation phases; which would be further quantified if the testing period was extended beyond that prescribed by ASTM C876.

*It would be prudent in extending this protocol, to modify and protect the “protruding” rebar from exposure to the salt/chloride environment. This would focus on the concrete specimens themselves and the protection gained from each. The rust product on the protruding rebar ends greatly affects the Half-Cell readings and would skew any extended results/readings taken after 120 Days.

*****With the second, Migrating Film Coating component being the key “pro-active” feature only supplied by the Vapor Lock admixture (in both specimens). Only the final, destructive examination of the Cracked-Beam Ponding tests (forthcoming) should illustrate any realized benefits in mitigating damage to the rebar; the last (and most difficult) component to significantly extending the Propagation phase of corrosion.***