



I. Executive Summary

Since 2009, EonCoat has quietly made a difference in the campaign against corrosion. Led by the Founder and CEO, Tony Collins, the agile Leadership Team at EonCoat can pivot from domestic to global, interact with customers from a small start-up to large corporations, and from a trial test in an emerging industry to a tried-and-true product with repeat customers.

Headquartered in Fuquay-Varina, North Carolina, USA, EonCoat manufactures its product at its secure facility. Inhouse ownership of manufacturing allows for the highest level of quality control while ensuring close management oversight. The team-wide focus on quality control starts with the daily morning team meetings. The day begins with a focus on customers, orders, and process improvements – and while the day may progress - that focus stays the same.

II. Financial Analysis

The current estimated cost of corrosion is [\\$2.5 trillion USD](#), the equivalent of 3.4% of the GDP. NACE estimates that recognized savings of 15% and 35% would be realized if companies used the currently available corrosion control practices.

With EonCoat's single application, 30-year warranty, and focus on environmental and applicator safety, we have data that shows savings with EonCoat would be at the highest level referenced above – if not even higher.

III. Product Definitions

EonCoat Corrosion Protection

As the company's flagship product, EonCoat Corrosion is a perennial favorite of repeat customers whose application does not require an excessive temperature rating. The temperature rating for this product is 110°C (230°F). This coating has been used in various industries, including pharmaceuticals, food manufacturing, and pipelines, to name a few.



EonCoat Corrosion Under Insulation Coating (CUI)

The Research and Development Team at EonCoat created the EonCoat CUI Coating after relentless demand for a coating that can tolerate significant temperature variances. With a temperature rating of 450°C (842°F), you can easily see why this coating is a preferred protective coating for any carbon steel asset that you must insulate. In addition, the 30-year warranty means you don't have to remove costly insulation as frequently as necessitated by lesser coatings.

EonCoat Weldable Coating

What sets EonCoat Weldable Coating apart from traditional coatings is its ability to protect carbon steel from corrosion before, during, and after welding. It enables welding after coating on the backside of coated steel without damaging the EonCoat. This one-of-a-kind product is rated at 600°C (1112°F). No coating in the world has ever held the heat produced by welds until EonCoat Weldable Coating. EonCoat Weldable Coating can be applied at the fabrication shop, and you can then transport the asset to the tank site. Additionally, because EonCoat has a wide range of weather application standards, you can apply the EonCoat Weldable Coating onsite as the tank is field erected.



IV. Third-Party Testing

EonCoat has always had a commitment to valid and reliable third-party testing, and that remains a steadfast practice. We will outline some of the more pertinent tests for you.

Client or Third-Party Testing Lab	Type of Test	Additional Details	Applicable Results
NASA +	Beachside 18 months test	Comparison by NASA	First 10 ever awarded by NASA
Assured Testing	ASTM B117	1500 hours	NO CORROSION
EDISON WELDING INSTITUTE +	ASTM B117	5000 hours	NO CORROSION
FIU	Houston Pipe Test	No corrosion	PASSED
Intertek	ASTM E84 UL 723	EonCoat on styrofoam	A1 fire rating
NSF International	ANSI 61-2012 / NSF 61		Certified for drinking water
	ASTM D4060 - 07	Taber Abrasion	3000 wear cycles per mil CS-17 wheel
Assured Testing	ASTM 4541		PASSED
Columbia Analytical Services +	VOC, HAP, Toxins	tested for all known	ZERO VOC's, HAP's and Toxins
ETT Environmental	Daphnia Magna 48-Hour Test		
ETT Environmental	Pimephales promelas 96-Hour Test		
Intertek	ASTM D3179 / Gravelometer		9A to 10
	ISO 20340:2009	35 cycles	PASSED
Assured Testing	Cyclic Salt Fog/UV Exposure ASTM D5894		PASSED
EWI Edison Welding Institute	FRA Rail Base Corrosion and Cracking Prevention		3.6 MILLION CYCLES W/O FAILURE
Chevron	REPORT ON CUI TESTING -	LAB AND REAL WORLD	NO CORROSION
FIU	Cyclic Polarization Curve		COMPLETE REPASSIVATION

+ indicates the report is hyperlinked, not an appendix at the end of this report.



V. Application Options

We engineered EonCoat as a plural component system applied at a 1:1 ratio. As a result, part A (acid) and Part B (base) mix during the application process as the EonCoat is applied to the carbon steel substrate.

We recommend a high-pressure plural pump with stainless steel lowers for large jobs. If an applicator has applied any other plural components, they will find this application straightforward. There are no heated lines or hoppers.

For small jobs, the dual component cartridge spray gun holds 600 mL cartridges pre-filled with EonCoat. This method is excellent for small jobs and touch-ups. Many of our customers start with this spray gun and a set of cartridges for demonstrations and small trial jobs.

For EonCoat's 30-year warranty to attach to any application, the job must be completed by a Certified EonCoat Applicator. We offer in-person and virtual training based on customer preferences and needs. Eoncoat maintains the global list of certified applicators.

VI. Technical Support

Whether the application job is domestic or global, EonCoat's team of corrosion experts is always available to support you through the application process. We can even advise you about topcoats if one is needed.

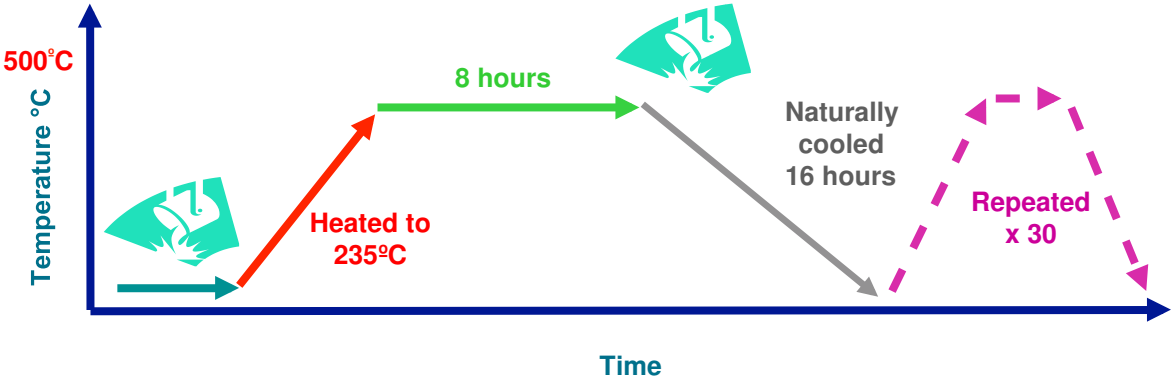
EonCoat solves Corrosion under insulation

- Inorganic
- Non-Toxic
- No VOCs (Volatile Organic Content)
- No HAPs (Hazardous Air Pollutants)
- Non-Flammable
- Odorless
- Dry fall – less worry about contamination of nearby surfaces

Houston Pipe Test

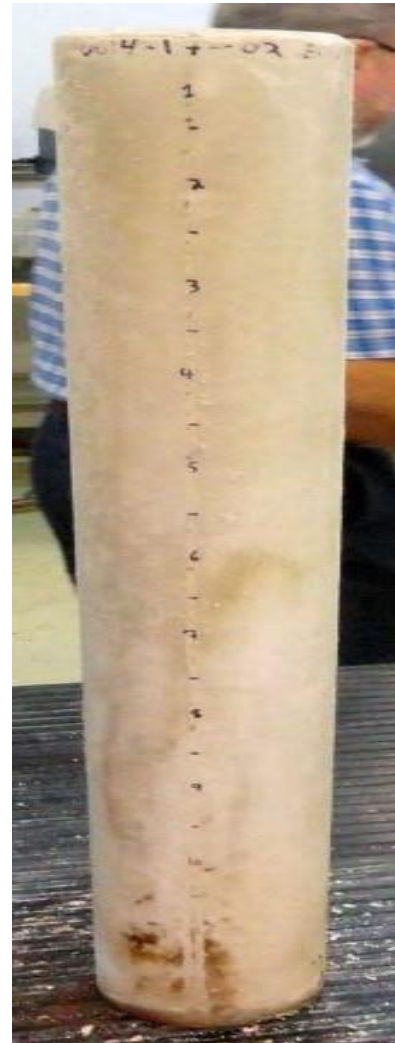


Cyclic Testing: Solution of 1% NaCl in water.

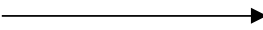


EonCoat after Houston Pipe Test

Close to ambient



250°C (~482°F)



Sandy Collins

From: Dave Purkiss <watermarketing@nsf.org>
Sent: Monday, September 24, 2012 11:45 AM
To: Sandy Collins
Subject: Implementation Plans for NSF/ANSI Standard 61-2012



NSF International

To: NSF 61, NSF 14, NSF 24 Listed and Applied Companies
From: Dave Purkiss, General Manager, Water Treatment and Distribution Products
Nasrin Kashefi, General Manager, Plumbing Products
Date: September 7, 2012
Re: Implementation Plans for NSF/ANSI Standard 61-2012

NSF/ANSI Standard 61 – 2012 has been published. PDF copies are available via your NSF Online account (<http://clients.nsf.org>) or by contacting your Certification Project Manager. Please contact Dave Purkiss at 734-827-6855 or email: Purkiss@nsf.org if you have any questions about these changes to NSF 61.

All changes are effective immediately unless otherwise indicated.

This version of NSF/ANSI Standard 61 has the following changes:

Issue 94: Additional materials have been added to Table 3.1 – Material-specific analyses, and the material type header of “Elastomer materials ” was changed to “Joining and sealing materials”.

Issue 97: This revision reduces the formulation information required for components of mechanical devices and mechanical plumbing devices that are composed from materials that are less than or equal to 2.0 sq inches per liter and listed in Table 3.1 Material-specific analyses.

Issue 99: Evaluation criterion has been specified for fire sprinklers and associated fittings that are used in piping systems intended to serve both drinking water and fire protection needs under section 4.

Issue 100: Language has been added under section 5 to clarify the coating manufacturer’s instructions for lab testing, and the relationship to manufacturer’s published use instructions for field and factory use. A tolerance of +/- 4°C for cure temperature has been added. A requirement for airless plural component systems to be operated at the midpoint of the coating manufacturer’s recommended pressure and temperature range has been established.

Issue 102: Table 3.1 has been updated to require nitrosamine analysis. The effective implementation date for this requirement is January 1, 2015.



09/20/2011

Mr. Tom Wall
EonCoat LLC
4000 Airport Drive Northwest
Wilson, NC 27896

Subject: Initial Authorized Registered Formulation for Standard 61

Enclosed is a copy of your initial Authorized Registered Formulation. This complete formulation (original copy with blue watermark) must be retained and on file at the identified plant location for review by an NSF Field Representative, conducting the annual/follow-up audits. Please forward the Authorized Registered Formulation to the appropriate plant. Each product is identified by Document Control Code (DCC number) located in the upper left hand corner of each page.

The NSF audit of your plant, including materials/process verification and product sampling, will be guided by this formulation. Failure to maintain this information at the plant may require special follow-up audits or result in removal of products from Listing.

Only those specific material/ingredients and use levels indicated in the Authorized Registered Formulation are authorized for use in the Certified Product. To obtain authorization for an alternate supplier (or other modification) please contact your Certification Project Manager at 1-800-NSF-MARK to request the appropriate forms. For customers outside the USA, please use 1-734-769-8010 and ask for your Certification Project Manager. As a reminder, you are not permitted to make any formulation changes to NSF Certified products without prior written approval from NSF.

If you have any questions about the Authorized Registered Formulation, please contact your Certification Project Manager indicated below.

Enclosure: Authorized Registered Formulation

Certification Project Manager: Julie Chappen, 734-769-5135, chappen@nsf.org

Plant: C0078024
DCC: PM12354

NSF/ANSI Standard 61 - Drinking Water System Components - Health Effects
Authorized Registered Formulation

This product may require additional evaluation or testing prior to authorization for Listing. Only products included in NSF's Official Listing are NSF Certified and authorized to bear an NSF Certification Mark.

Customer Name: EonCoat LLC

Facility Location: Wilson, NC

Customer Number: C0078023

Facility At: Wilson, NC

Facility Number: C0078024

Trade Name(s)	Function(s)	Size
EonCoat	Coatings - Fittings	>= 100 in
	Coatings - Tank	>= 1,000,000 gal
	Coatings - Valve	>= 60 in
	Coating-Pipe-Immediate Return to Service	>= 100 in
	Coatings - Pipe	>= 100 in

Listing Notes

Evaluated for immediate return to service.

Coating Notes

Colors: White

Number of coats: 1

Maximum field use dry film thickness (in mils): 36 (maximum 6 mils per pass)

Recoat cure time and temperature: Allow 5 second pause between each pass

Final cure time and temperature: 24 hours at 35° - 110°F

Special comments: The mix ratio of Part A:Part B is 1:1 by volume.

Category: PMTL, SECTION 5 - BARRIER MATERIALS**Material Type:** Ceramic (CERAM)**Temperature:** Cold (73 F/23 C)**Auditor Notes**

Please complete a sample disclosure form and send to NSF International's confidential and secure fax at 734-827-7728. Include a photocopy of the Authorized Registered Formulation designating the components used to make the sample submitted for testing (circle or underline the alternate used).

Sample Notes

Please collect two of the smallest kits available of EonCoat, totaling one gallon or greater. Kits should include both Part A and Part B. A representative from EonCoat LLC will perform the coating application at NSF.

DCC: PM12354

Date: 09/20/2011

Formulation

Customer Name: EonCoat LLC
Customer Number: C0078023

Facility Location: Wilson, NC
Facility At: Wilson, NC
Facility Number: C0078024

Formulation Description: FORMULATION

Chemical Description	Trade Name	Supplier	% or PPW	DCC	Acceptance Date
COATING	EONCOAT	EONCOAT LLC	100	1A26539	09/20/2011

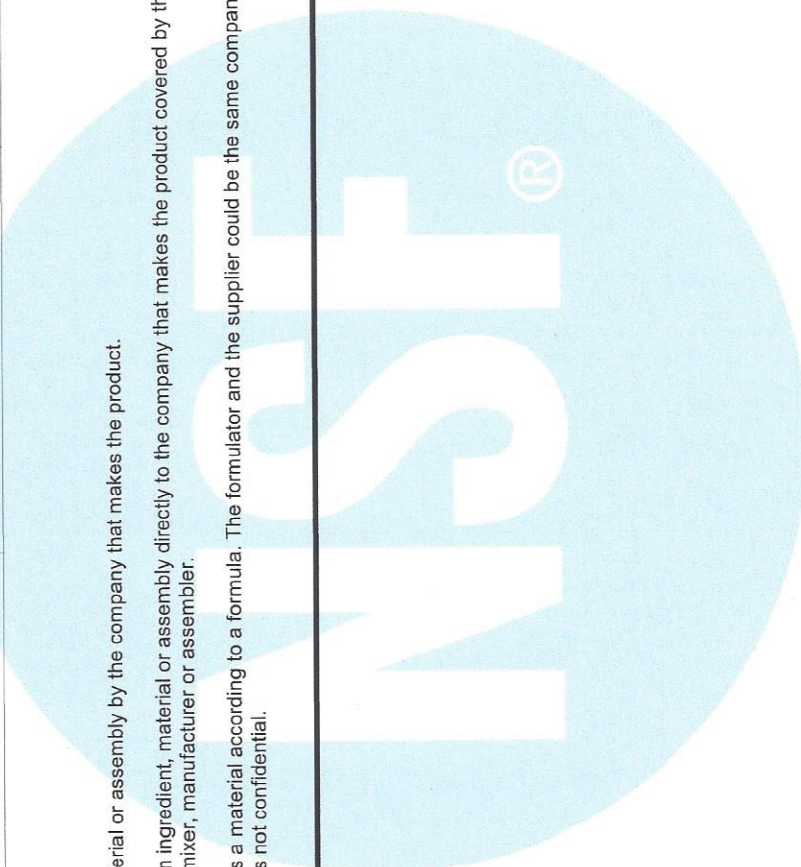
Notes

Definitions of Terminology used in this Document:

Trade name: The name given to the ingredient, material or assembly by the company that makes the product.

Supplier: The name of the company that provides an ingredient, material or assembly directly to the company that makes the product covered by this registration. The supplier could be a formulator, distributor, fabricator, molder, extruder, mixer, manufacturer or assembler.

Formulator: The name of the company that prepares a material according to a formula. The formulator and the supplier could be the same company. This field may be blank as this information is only reported when the information is not confidential.





Contract for Certification by NSF International

A Contract made and entered into this 28th day of March, 2011 between NSF International, a corporation organized and existing under the laws of the State of Michigan, with its principal office in Ann Arbor, Michigan USA (hereinafter called "NSF") and EonCoat LLC, 4000 Airport Drive Northwest, Wilson, NC 27896 (hereinafter called "COMPANY").

1. NSF is an independent, third party certifying organization. It does not guarantee or warranty any product or service, and does not approve or verify any product or service. NSF also does not review or evaluate every product or service, but instead follows the protocols set forth in the materials described in paragraph 2 of this Contract. Certified product(s) are any goods, equipment, component(s), system(s), service(s), material(s), compound(s), or ingredient(s) that have been specifically authorized by NSF for Certification and use of an NSF Certification Mark (the Mark). The terms Certification and Certified are synonymous with the terms Listing or Listed.
2. COMPANY hereby certifies and represents that it has received and read the Certification standards, protocols, or criteria (hereinafter called "the Standard(s)") and Certification policies and guidelines (hereinafter called "the Policies"). The Standard(s) and Policies shall be that which NSF may list in any written notice to COMPANY, which list may be amended from time to time. COMPANY represents and warrants that, to the best of its information and knowledge, the information given to NSF for purposes of Certification is true and accurate. COMPANY assumes sole responsibility for the truth and accuracy of such information, including, but not limited to, information about the products or product names for which COMPANY requests Certification. COMPANY expressly acknowledges and agrees that NSF may utilize subcontract laboratory services when NSF deems suitable.
3. Upon determination by NSF that the evaluated products comply with the applicable requirements of the materials referenced in paragraph 2 of this Contract, and upon execution of this Contract and payment of the annual Certification fee and other outstanding fees, NSF agrees to authorize the COMPANY for Certification and use of the Mark on COMPANY's Certified products.
4. COMPANY expressly acknowledges and agrees that execution of this Contract, of and by itself, is not authorization to use the Mark. In accordance with the materials referenced in paragraph 2 of this Contract, NSF will notify COMPANY in writing of Certification and authorization to use the Mark.
5. COMPANY hereby certifies and represents that if authorized to use the Mark, the Mark will be placed only on products fully complying with all NSF requirements. COMPANY further certifies and represents that it will abide by all NSF requirements, as specified in the materials referenced in paragraph 2 of this Contract.
6. It is understood and agreed that the materials referenced in paragraph 2 of this Contract shall be periodically revised in accordance with procedures that expressly provide for representation and comment by all parties of interest. Any revision shall be announced by NSF by written notice to COMPANY. Upon receipt of notice of any applicable revision, COMPANY agrees that it will abide by the announced revision; or, at its option, COMPANY may terminate this Contract in accordance with the provisions of this Contract.
7. COMPANY agrees that its use of the Mark is its representation that its products are Certified by NSF and comply with all NSF requirements. COMPANY assumes full and complete responsibility for its use of the Mark or other representation that its products are Certified. COMPANY agrees that it will make claims regarding Certification only in respect to the scope for which Certification has been granted. COMPANY shall not: (i) use its Certification in any manner that brings NSF into disrepute; (ii) make any statement concerning its Certification that is misleading or unauthorized by NSF; or (iii) use in any misleading manner any Certification certificate or report related to Certification. NSF assumes no liability, and COMPANY shall be solely responsible, for any claims arising from the COMPANY's misuse of the Mark or misrepresentation of the Certification status of its products, or failure at all times to comply with the materials referenced in paragraph 2 of this Contract.
8. It is understood and agreed that the Mark on a product and its Certification are invalid if, as determined by NSF, the product has been altered or has been represented as being Certified for any purpose or end use other than that Certified by NSF.
9. COMPANY shall be solely responsible for, and assumes all risk of property damage, personal injury (including death), or other damages of any kind arising out of or relating to: (i) the use, misuse, sale and resale of any of its products or related goods, whether or not such products or goods are Certified; (ii) the failure to comply with all applicable laws, rules, codes, regulations and industry practices relating to COMPANY's products; (iii) any of COMPANY's negligent acts or omissions, or its willful misconduct; and (iv) any matters covered by paragraph 10 of this Contract.
10. COMPANY shall indemnify, defend and hold harmless NSF, its affiliates, successors and assigns and its and their respective directors, officers, employees, representatives and agents (hereinafter referred to collectively as the "NSF Indemnified Persons") from, against and with respect to any and all demands, claims, complaints, actions or causes of action, suits, proceedings, investigations, arbitrations, assessments, losses, damages, liabilities, costs and expenses (including, but not limited to, interest, penalties and reasonable attorneys' fees and other costs) asserted or alleged against, imposed upon or incurred by such NSF Indemnified Persons, directly or indirectly, by reason of or resulting from or in connection with any use or misuse of COMPANY's products, any negligent acts or omissions of COMPANY (or allegations thereof), or COMPANY's willful misconduct (or allegations thereof), and any material breach by COMPANY of this Contract. This indemnity provision shall survive all terminations of this Contract.



11. NSF makes no other representations or warranties, whether express or implied, with regard to its obligations hereunder, or the merchantability or fitness of any goods or products for a particular purpose.
12. COMPANY may terminate this Contract at any time upon thirty (30) days written notice to NSF, but shall be liable for costs for services provided by NSF through the date of receipt of notice, and for any additional costs necessary to terminate services. Services provided by NSF may include, but are not limited to audit, laboratory testing, toxicological review, and initial Listing fees. All quotes for services are estimates and do not include the cost of retesting in the event of a product failure during the initial Certification. Upon acceptance of COMPANY's application fee or project deposit by NSF, the application fee or deposit is non-refundable. NSF shall repay only pre-paid fees for services that were not provided. NSF may terminate this Contract at any time upon thirty (30) days written notice to the COMPANY.
13. Unless terminated by either party, this Contract shall continue in effect from year to year. COMPANY shall notify NSF by December 30 if it wishes to cancel the Contract for the next year. If NSF has not received such notice in writing by December 30, COMPANY shall make payment of the required annual fees by January 31. COMPANY agrees that all payments are due 30 days net. A finance charge shall be imposed on all invoices which are over 30 days past due. The finance charge is computed by application of the periodic rate of 1% per month (12% per annum) to the previous month's balance after deduction of payments made since the previous statement date. COMPANY is responsible to pay, without any corresponding withholding from NSF, any and all taxes and fees that may be imposed by any and all governmental agencies outside of the United States having jurisdiction over COMPANY'S business transactions with NSF. If any annual fee or any other fee is not paid when due, and such failure shall continue for a period of 60 days after written notice of discontinued Listing services, then this Contract shall be deemed irrevocably terminated without further notice.
14. After termination of this Contract for any reason, COMPANY agrees to immediately discontinue all use of the Mark and shall promptly confirm, in writing, to NSF that COMPANY has discontinued use of the Mark on its products and/or in its product literature and advertising. COMPANY further agrees that, upon termination of the Contract for any reason, it shall surrender, efface, or otherwise dispose of, in a manner acceptable to NSF, any unused Marks and data labels, dies, molds, stencils, marking devices, literature, advertisement, or other information bearing the Mark or referencing NSF Certification. If NSF has reason to question conformance by COMPANY with this provision of the Contract, COMPANY agrees to allow NSF reasonable access to COMPANY's facilities to conduct inspections to verify conformance.

COMPANY agrees that NSF's remedies at law to enforce the provisions of this paragraph 14 are inadequate and that accordingly NSF shall be entitled to and COMPANY agrees to the entry of an order in any court of competent jurisdiction specifically enforcing the provisions of this paragraph 14. In the event COMPANY shall become a debtor in any insolvency or bankruptcy proceeding (whether under the laws of the United States of America or the laws of any other country, territory or jurisdiction) after termination of this Contract, the COMPANY agrees and stipulates that NSF shall be entitled to relief from any applicable stay, order or injunction to immediately pursue enforcement of this paragraph 14 under applicable law.

15. NSF shall have no liability to COMPANY or to any third party with respect to NSF's obligations under this Contract or otherwise for consequential, exemplary, special, incidental, or punitive damages even if NSF has been advised of the possibility of such damages. In any event, NSF's liability shall be limited to twenty percent (20%) of the amount actually paid to NSF during the current year of this Contract. This limitation applies to all claims in the aggregate, including, without limitation, claims based on breach of Contract, breach of warranty, professional negligence, strict liability, misrepresentations, and other torts or claims.
16. In connection with determining whether initial Certification is warranted and/or as to any re-Certification, NSF agrees to provide COMPANY written notice of nonconformance with any NSF requirement. NSF reserves the right to withdraw authorization for Certification and use of the Mark for any product, at any time, for COMPANY's failure to correct the nonconformance within a reasonable time.
17. NSF offers to COMPANIES that are in good standing access to "NSF On-Line", the web address of which is <http://clients.nsf.org>.

At COMPANY's request, COMPANY's access to NSF On-Line will include access to COMPANY's product formulations. Does COMPANY request access to COMPANY's product formulations when using NSF On-Line? Please initial one:

NO _____
Initials

YES _____
Initials

COMPANY's access to and use of NSF On-Line is subject to the NSF On-Line User Access License ("Access License") which is published on NSF On-Line. Use of the NSF On-Line website by any person accessing NSF On-Line with a user name and password of the COMPANY constitutes agreement to the Access License by COMPANY, its employees and anyone else accessing NSF On-Line with a user name and password of COMPANY. NSF may change any of the terms or conditions of the Access License at any time and from time to time by publishing the amended Access License on the NSF On-Line website. No other notice of changes to the Access License will be given by NSF. After publication of changes to the Access License on NSF On-Line, use of the NSF On-Line website by any person accessing NSF On-Line with a user name and password of the COMPANY constitutes agreement to the changed Access License by COMPANY, its employees and anyone else accessing NSF On-Line with a user name and password of COMPANY.

COMPANY understands that NSF On-Line may contain COMPANY's confidential, proprietary or sensitive information. If COMPANY uses NSF On-Line, COMPANY is solely responsible for maintaining the confidentiality of COMPANY's user name(s) and password(s) and for restricting access to COMPANY's computers. COMPANY accepts sole responsibility for all activities that occur under COMPANY's name(s)



and password(s). NSF will not be liable for any loss that COMPANY may incur as a result of someone else using or having access to COMPANY's name(s) and password(s), either with or without COMPANY's knowledge. In addition, COMPANY is solely responsible for maintaining the secrecy of COMPANY's name(s) and password(s) and for monitoring the appropriate use of user names and passwords by COMPANY's employees and anyone else that has access to COMPANY's computer(s) at any time. COMPANY may request a change in a user name and password at any time. NSF may from time to time notify COMPANY that it is appropriate to change COMPANY's name(s) or password(s), but NSF disclaims any responsibility for giving any such notice.

- 18. Any legal action by either party or any person or party claiming any right or seeking to avoid any obligation under the Contract, or otherwise relating in any way to this Contract, shall be brought and maintained exclusively in the United States District Court for the Eastern District of Michigan if it has subject matter jurisdiction, and otherwise in the appropriate District Court or Circuit Court in the State of Michigan as provided in the venue statutes of the State of Michigan. The parties consent to personal jurisdiction and venue in such courts, and further agree not to seek to invoke the jurisdiction of any other court. This Contract shall be governed, interpreted and construed in accordance with Michigan law, without regard to its conflicts of law provisions.
- 19. The invalidity or unenforceability of any particular provision(s) of this Contract and/or the materials referenced in paragraph 2 of this Contract shall not affect the other provisions.
- 20. This Contract, any Addenda, the materials set forth in paragraph 2, and the Access License for NSF Online users, constitute the entire agreement between the Parties with respect to the subject matter hereof and supersede all previous communications, representations or agreements, whether oral or written, between the Parties with respect to said subject matter. No modification will be binding upon either Party unless it is made in writing and is signed by duly authorized representatives of both Parties. Any written modifications on this document are not acceptable and invalidate this Contract.

I am authorized to sign this contract on behalf of the above referenced COMPANY.

For
NSF International

For
EonCoat LLC

Michael P. Walsh

Tom Wall

Signature

Signature

Michael P. Walsh, Chief Financial Officer

Tom Wall, VP Operations

Typed Name and Title

Typed Name and Title

March 28, 2011

May 12, 2011

Date

Date

www.assuredtestingservices.com
 198 River Road
 Ridgway, PA 15853 USA
 P: +1 814-773-3224
 F: +1 814-773-3225

TEST REPORT -19531 A

CLIENT BILL TO

CLIENT SENT TO

BILL TO: NCRALEIGH--2300	OurSupplier ID
Eoncoat LLC	
551 Pylon Drive	
Unit D	
Raleigh NC 27606	
USA	Supplier Code
Ph: 919-244-3790	

SENT TO: NCRALEIGH--2300	OurSupplier ID
Eoncoat LLC	
551 Pylon Drive	
Unit D	
Raleigh NC 27606	
USA	Supplier Code
Ph: 919-244-3790	

Test Report#:19531 **Entry Date:** Monday, April 10, 2017

Contact: Sameer Patel

PO#: 2359

*Pull-Off Adhesion Testing IAW ASTM D4541 for One (1) Painted Panel.
 See attached photo.*

REVISED TEST REPORT TR19531 A: Revised to update results.

Part Test Information										
PART NUMBER	LOT NUMBER	S#	SAMPLE DESCRIPTION							
TEST SPECIFICATION NAME	TEST PROCEDURE NAME		HOURS	CYCLES						
TEST RESULTS	INDATE	OUTDATE	TESTCNT	RETCNT	REQ	COMP	REQ	HOURS	COMP	
	032717-3		4" x 4" Panel							
Unspecified			Adhesion ASTM D4541-09e1 Method E Pull-off Adhesion Bond Strength							
	11-Apr-17	12-Apr-17	1	1						
<i>Results: 1253 psi maximum pull-off bond strength. ~ 40% intercoat adhesion failure. The rest failure to substrate or glue failure.</i>										
<i>Requirements: Report maximum pull-off adhesion strength.</i>										

Michele Singer

Revision# 1.11 Date 11/18/16

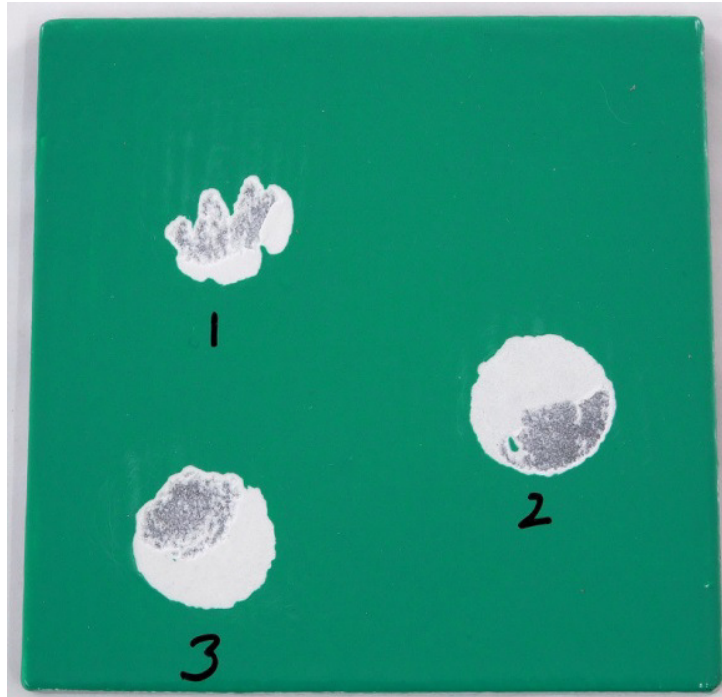
End Of Report



Assistant Laboratory Manager

The results stated above relate only to the specific items tested. Information and statements in this report are derived from material, information, and/or specifications furnished by the client and exclude any expressed or implied warranties as to the fitness of material tested or analyzed for any particular purpose or use. This report is confidential property of our client and may not be used for advertising purposes. This report shall not be reproduced except in full, without written approval of this laboratory. This recording of false, fictitious or fraudulent statements or entries on this document may be punished as a felony. Sample remnants are retained for a minimum of 6 months following issuance of test results, at which point they will be discarded unless notified in writing by the client. Any client claims or damages arising from the use of information in this report are strictly limited to the cost of the services provided by Assured Testing Services.

**Photo: Testing IAW ASTM D4541 Pull-Off Adhesion Testing for
One (1) Painted Panel**



4" x 4" Panel Sample after Pull-off Adhesion Bond Strength Testing (see above)

End of photo page

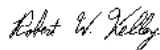
Client: EonCoat, LLC
Sample ID: Process Water
NPDES #: 77484

Daphnia magna 48 Hour Acute Definitive Test
EPA-821-R-02-012 Method 2002

Pimephales promelas 96 Hour Acute Definitive Test
EPA-821-R-02-012 Method 2000

Test Date: 23-Feb-21

Test Reviewed and Approved By:



Robert W. Kelley, Ph.D.
QA/QC Officer



Certification #E87819

Test results presented in this report conform to all requirements of
NELAC, conducted under NELAC Certification Number E87819
Florida Dept. of Health. Included results pertain only to provided samples.

SCDHEC Certification #23104

NCDENR Certification # 022

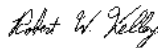
BBBBB人수 // 、 수B 수0A、 수BLLH7B공。 》 / B수20 》 《0A B수0A0A、
△ πL 수H7E 수· | 수7E 수7E 수B수 《。 0B 수77E

Client: EONCOAT
수 // 수0A B수H7Bπ· | 수△ 수B수L 수△· |
NPDES #: NC

Test Date: 09-Feb-21

Laboratory ID #: 수LH7E 수B수B수 수0 수

Test Reviewed and Approved By:



Robert W. Kelley, Ph.D.

QA/QC Officer



Certification #E87819

SCDHEC Certification #23104

Test results presented in this report conform to all requirements of

NELAC, conducted under NELAC Certification Number E87819
Florida Dept. of Health. Included results pertain only to provided samples.

NCDENR Certification # 022

Facility: EONCOAT PROCESS H2O	NPDES# NC	Pipe # N/A	County: Wake
Laboratory Performing Test: ETT Environmental, Inc.		Comments	
X			
Signature of Operator in Responsible Charge <i>[Signature]</i>			
X			
Signature of Laboratory Supervisor			

MAIL ORIGINAL TO

Environmental Sciences Branch
Div. of Water Quality
N.C. DENR
1621 Mail Service Center
Raleigh, North Carolina 27699-1621

North Carolina Ceriodaphnia Chronic Pass/Fail Reproduction Toxicity Test

CONTROL ORGANISMS	1	2	3	4	5	6	7	8	9	10	11	12
# Young Produced												
Adult (L)ive (D)ead												
Effluent %	[]											
TREATMENT 2 ORGANISMS	1	2	3	4	5	6	7	8	9	10	11	12
# Young Produced												
Adult (L)ive (D)ead												

Chronic Test Results	
Calculated t=	
Critical Value=	
% Reduction=	
% Mortality	Avg. Reprod.
Control	Control
Treatment 2	Treatment 2
Control CV	
% 3rd Brood	PASS FAIL

pH	1st sample	1st sample	2nd sample
Control	[] []	[] []	[] []
Treatment 2	[] []	[] []	[] []
D.O.	start end 1st sample	start end 1st sample	start end 2nd sample
Control	[] []	[] []	[] []
Treatment 2	[] []	[] []	[] []

Complete This for Either Test		Test Start Date	
Collection (Start) Date		23-Feb-21	
Sample 1	22-Feb-21	Sample 2	NA
Sample Type (Duration)		1st	2nd
	Grab Comp Duration	Tox	Tox
Sample 1	X [] []		
Sample 2	[] [] []	Dilution	Sample Sample
	Hardness (mg/L)	83.3	
	Spec. Cond. (µmhos)	315	N/A
	Chlorine (mg/L)		N/A
	Sample Temp. at receipt (°C)		ambient

LC50/Acute Toxicity Test

(Mortality expressed as %, combining replicates)

0	6.25	12.5	25	50	100					Concentration
0	5	5	50	50	100					Mortality

LC50 = 34.1 [] %	Method of Determination	
95% Confidence Limits	Moving Average	Probit []
27.0 % 43.2 %	Spearman Karber	X [] Other []

Organism Tested	<i>Daphnia magna</i>
------------------------	----------------------

start/end	Control	start/end
7.8 7.8		7.8 7.1
10.2 8.8	High Conc.	8.1 6.8
pH		D.O.

48 Hour Acute Toxicity Test

Test Method: EPA 821 R-02-012 ; Method 2002 Daphnia magna

Client: EONCOAT	Sample ID: PROCESS H2O	Lab ID#: T58592 D MA
Start Date: 02/23/21	Time: 03:45 PM	Set By: AM
End Date: 02/25/21	Time: 03:29 PM	Ended By: AM

Daphnids	Test Vessel	Test Solution Volume	Incubator	Transfer Volume	Dilution Water
	20 dram glass vial	20 mL	#1	0.05 mL	MHSF
Randomization pattern per SOP		Neonates from common holding vessel		Light: 50 -100 ft-c. 16 hr light / 8 hr dark	

TEST ORGANISMS

for <i>Daphnia magna</i>			
Date Removed: 2/23/21	Between:	and:	
	Source: ABS	Days old: <24 hr	
<i>Mysidopsis bahia</i>			
	Source:	Days old:	
Test Organisms Pre Fed by: AM Time: 1300			

Comments:
Young collected from adults that were originally received as neonates on 2/9/21

MORTALITY DATA

Conc.	Rep.	Initial # organisms	Cumulative Mortality				Final Mortality
			24 hr	48 hr	72 hr	96 hr	
Control	A	5	0	0			0%
	B	5	0	0			
	C	5	0	0			
	D	5	0	0			
6.3%	A	5	0	0			5%
	B	5	0	1			
	C	5	0	0			
	D	5	0	0			
12.5%	A	5	0	0			5%
	B	5	0	0			
	C	5	0	1			
	D	5	0	0			
25.0%	A	5	0	2			50%
	B	5	0	3			
	C	5	0	1			
	D	5	0	4			
50.0%	A	5	1	3			50%
	B	5	0	1			
	C	5	0	5			
	D	5	0	1			
100.0%	A	5	5	5			100%
	B	5	5	5			
	C	5	5	5			
	D	5	5	5			
	A						
	B						
	C						
	D						

Initial					Final				
Temp	D.O.	pH	Cond/ Salini	By:	Temp	D.O.	pH	Cond/ Salini	By:
25.2	7.8	7.8		AM	25.7	7.1	7.8		AM
25.2	7.9	8.6		AM	25.7	7.1	7.8		AM
25.2	8.0	8.9		AM	25.7	6.8	7.8		AM
25.2	8.0	9.4		AM	25.7	7.1	8.0		AM
25.2	8.0	9.8		AM	25.7	7.0	8.2		AM
25.2	8.1	10.2		AM	25.7	6.8	8.8		AM
°C	mg/L		PPT		°C	mg/L		PPT	

48 Hour LC50 = 34.1% □

Pimephales promelas 96 Hour Acute Definitive Test

EPA-821-R-02-012 Method 2000

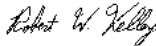
Client: EONCOAT

7A 22A // 777 MA 8888 9999 0000 | 1111 2222 3333 4444 |
NPDES #: NC

Test Date: 11/08/2018

Laboratory ID #: 1111 2222 3333 4444 | 5555 6666 7777 8888

Test Reviewed and Approved By:



Robert W. Kelley, Ph.D.

QA/QC Officer



Certification #E87819

SCDHEC Certification #23104

Test results presented in this report conform to all requirements of

NELAC, conducted under NELAC Certification Number E87819

Florida Dept. of Health. Included results pertain only to provided samples.

NCDENR Certification # 022

Facility: EONCOAT PROCESS H2O		NPDES# NC	Pipe # N/A	County: Wake
Laboratory Performing Test: ETT Environmental, Inc.		Comments		
X Signature of Operator in Responsible Charge				
X <i>[Signature]</i>				
Signature of Laboratory Supervisor				

MAIL ORIGINAL TO

Environmental Sciences Branch
 Div. of Water Quality
 N.C. DENR
 1621 Mail Service Center
 Raleigh, North Carolina 27699-1621

North Carolina Ceriodaphnia Chronic Pass/Fail Reproduction Toxicity Test

CONTROL ORGANISMS	1	2	3	4	5	6	7	8	9	10	11	12
# Young Produced												
Adult (L)ive (D)ead												
Effluent %	[]											
TREATMENT 2 ORGANISMS	1	2	3	4	5	6	7	8	9	10	11	12
# Young Produced												
Adult (L)ive (D)ead												

Chronic Test Results	
Calculated t=	
Critical Value=	
% Reduction=	
% Mortality	Avg. Reprod.
Control	Control
Treatment 2	Treatment 2
Control CV	
% 3rd Brood	PASS FAIL

pH	1st sample	1st sample	2nd sample
Control	[] []	[] []	[] []
Treatment 2	[] []	[] []	[] []
D.O.	start end 1st sample	start end 1st sample	start end 2nd sample
Control	[] []	[] []	[] []
Treatment 2	[] []	[] []	[] []

Complete This for Either Test		Test Start Date	
Collection (Start) Date		23-Feb-21	
Sample 1	22-Feb-21	Sample 2	NA
Sample Type (Duration)		1st	2nd
	Grab Comp Duration	Tox	Tox
Sample 1	X [] []		
Sample 2	[] [] []	Dilution	Sample Sample
	Hardness (mg/L)	83.3	
	Spec. Cond. (µmhos)	315	N/A
	Chlorine (mg/L)		N/A
	Sample Temp. at receipt (°C)		ambient

LC50/Acute Toxicity Test

(Mortality expressed as %, combining replicates)

0	6.25	12.5	25	50	100					Concentration
0	40	100	100	100	100					Mortality
LC50 = 6.34 %		Method of Determination								
95% Confidence Limits		Moving Average		Probit		[]				
4.6 % 8.7 %		Spearman Karber		X		Other				

start/end	Control	start/end
7.8 7.7		7.8 8.0
10.2 8.6	High Conc.	8.1 7.8
pH		D.O.

Organism Tested *Pimephales promelas*

48 Hour Acute Toxicity Test

Test Method: EPA 821 R-02-012 ; Method 2000 Pimephales promelas

Client: EONCOAT	Sample ID: PROCESS H2O	Lab ID#: T58592 P PRO
Start Date: 02/23/21	Time: 03:45 PM	Set By: AM
End Date: 02/27/21	Time: 03:34 PM	Ended By: JC

Test Vessel	Test Solution Volume	Incubator	Transfer Volume	Dilution Water	
Fathead Minnows	500 mL plastic cup	200 mL	#1	0.5 mL	MHSF
Randomization pattern per SOP		Neonates from common holding vessel		Light: 50 -100 ft-c. 16 hr light / 8 hr dark	

TEST ORGANISMS

for <i>Ceriodaphnia dubia</i>			
Date Removed:	Between:	and:	
<i>Pimephales promelas</i>	Source: ABS	ays old:	7
<i>Mysidopsis bahia</i>	Source:	Days old:	
Test Organisms Pre Fed by: AM Time: 1300			

Comments: FISH HATCHED ON 2/16/21 BTW 1130-1300 ET

MORTALITY DATA

Conc.	Rep.	Initial # organisms	Cumulative Mortality				Final Mortality
			24 hr	48 hr	72 hr	96 hr	
Control	A	10	0	0	0	0	0%
	B	10	0	0	0	0	
	C						
	D						
6.25%	A	10	1	4	4	4	45%
	B	10	1	4	5	5	
	C						
	D						
12.5%	A	10	1	10	10	10	100%
	B	10	4	10	10	10	
	C						
	D						
25.0%	A	10	10	10	10	10	100%
	B	10	10	10	10	10	
	C						
	D						
50.0%	A	10	10	10	10	10	100%
	B	10	10	10	10	10	
	C						
	D						
100.0%	A	10	10	10	10	10	100%
	B	10	10	10	10	10	
	C						
	D						
	A						
	B						
	C						
	D						

Initial					Final				
Temp	D.O.	pH	Cond/ Salini	By:	Temp	D.O.	pH	Cond/ Salini	By:
25.2	7.8	7.8		AM	25.3	8.0	7.7		JC
25.2	7.9	8.6		AM	25.3	7.9	7.8		JC
25.2	8.0	8.9		AM	25.3	7.8	7.9		JC
25.2	8.0	9.4		AM	25.3	7.9	8.0		JC
25.2	8.0	9.8		AM	25.3	7.6	8.2		JC
25.2	8.1	10.2		AM	25.3	7.8	8.6		JC
°C	mg/L		PPT		°C	mg/L		PPT	

96 Hour LC50 = 6.34%

July 21, 2021

M/M Sameer Patel
EonCoat LLC
4000 Airport Dr. NW
Wilson, NC 27896
USA

Intertek PTL # P20210775

Dear M/M Patel:

Enclosed you will find the results of the testing you requested.

If you have any questions regarding the data, please do not hesitate to contact me.

Yours sincerely,



Kevin E. Schuman
Quality Manager

KES/sk
Enclosures

Unless inherent in the requested specification or standard, Intertek laboratories apply the "Simple Acceptance" rule, also called "Shared Risk approach" of ILAC-G8:09/2019 guide where the statements of conformity are reported as either: Passed (or in-tolerance) when measured values are within the specified limits, or Failed (or out-of-tolerance) if one or more measured values are outside the specified limits. If the Client requests a statement of conformity other than one inherent in the specification or standard or the "Simple Acceptance" rule, the decision rule shall be clearly defined by the Client, communicated to Intertek, and agreed by all related parties.



Testing : **Standard Test Method for Chipping Resistance of Coatings**
Test Method : ASTM D3170/D3170M-14
Project Number : P20210775
Customer : EonCoat LLC
Attention : Sameer Patel
Analyst : D. Loehr
Date : July 19, 2021



Substrate : **Steel panel**
Coating : **Gray Coating Material**
Panel Dimensions : 4" x 6" plaque
Test Temperature : Room Temperature
Conditioning : 1+ hour at test temperature
Impact Angle : 90°
Comparison Standards : SAE J400 chip rating standard transparencies
Adhesive Tape Used : 3M Strapping Tape

Sample #	Chipping Rating(s)
1	9A
2	8A
3	9A

Chipping ratings consist of a number which describes the number of chips in a 4" x 4" area, and a letter which describes the size of the chips area. The numbers range from 10 (no chips) to 0 (>250 chips). The letters range from A (< 1mm) to D (> 6mm). A tested sample will often exhibit multiple chip sizes, and the most numerous will be listed first - for example **5B-6A-8C**.

Chipping Ratings

10 - no chips	A - chips <1mm diameter
9 - 1 chip	B - chips 1mm to 3mm dia.
8 - 2 to 4 chips	C - chips 3mm to 6mm dia
7 - 5 to 9 chips	D - chips >6mm
6 - 10 to 24 chips	
5 - 25 to 49 chips	
4 - 50 to 74 chips	
3 - 75 to 99 chips	
2 - 100 to 149 chips	
1 - 150 to 250 chips	
0 - over 250 chips	

March 16, 2018

Sameer Patel
EonCoat LLC
551 Pylon Drive Unit D
Raleigh, NC 27606
USA

Intertek PTL # P20181093

Dear M/M Patel:

Enclosed you will find the results of the testing you requested.

If you have any questions regarding the data, please do not hesitate to contact me.


Yours sincerely,



Kevin E. Schuman
Quality Manager

KES/jh
Enclosures



Testing	: Standard Test Method for Chipping Resistance of Coatings	 <p>ACCREDITED Cert. No. 0619.01 TESTING LABORATORY</p>
Test Method	: ASTM D3170/D3170M-14	
Project Number	: P20181093	
Customer	: EonCoat LLC	
Attention	: Sameer Patel	
Analyst	: D. Loehr	
Date	: March 15, 2018	
Sample ID	: EC-PU	
Substrate	: Unknown	
Coating	: Unknown	
Panel Dimensions	: 4" x 6" plaque	
Test Temperature	: Room Temperature	
Conditioning	: 1+ hour at test temperature	
Impact Angle	: 90°	
Comparison Standards	: SAE J400 chip rating standard transparencies	
Adhesive Tape Used	: 3M Strapping Tape	

Sample #	Chipping Rating(s)
1	10
2	10
3	10

Note: Samples tested on the Black side. Samples exhibit surface blemishes but no chips

Chipping ratings consist of a number which describes the number of chips in a 4" x 4" area, and a letter which describes the size of the chips area. The numbers range from 10 (no chips) to 0 (>250 chips). The letters range from A (< 1mm) to D (> 6mm). A tested sample will often exhibit multiple chip sizes, and the most numerous will be listed first - for example **5B-6A-8C**.

Chipping Ratings	
10 - no chips	A - chips <1mm diameter
9 - 1 chip	B - chips 1mm to 3mm dia.
8 - 2 to 4 chips	C - chips 3mm to 6mm dia
7 - 5 to 9 chips	D - chips >6mm
6 - 10 to 24 chips	
5 - 25 to 49 chips	
4 - 50 to 74 chips	
3 - 75 to 99 chips	
2 - 100 to 149 chips	
1 - 150 to 250 chips	
0 - over 250 chips	

Intertek reports are issued for the exclusive use of the clients to whom they are addressed. No quotations from reports or use of the Intertek name is permitted except as expressly authorized in writing. Letters and reports apply only to the specific materials, products or processes tested, examined or surveyed and are not necessarily indicative of the qualities of identical or similar materials, products or processes. The liability of Intertek with respect to services rendered shall be limited to the amount of consideration paid for such services and not include any consequential damages.

www.assuredtestingservices.com
198 River Road
Ridgway, PA 15853 USA
P: +1 814-773-3224
F: +1 814-773-3225

TEST REPORT -20446

CLIENT BILL TO

BILL TO: NCRALEIGH--2300	OurSupplier ID
Eoncoat LLC	
551 Pylon Drive	
Unit D	
Raleigh NC 27606	
USA	Supplier Code
Ph: 919-244-3790	

CLIENT SENT TO

SENT TO: NCRALEIGH--2300	OurSupplier ID
Eoncoat LLC	
551 Pylon Drive	
Unit D	
Raleigh NC 27606	
USA	Supplier Code
Ph: 919-244-3790	

Test Report#:20446 **Entry Date:** Friday, March 24, 2017

Contact: Sameer Patel

PO#: 2357 QN022417EONCT

*Cyclic Salt Fog / UV Testing IAW ASTM D5894 for Painted Panels.
Panels were scribed and masked by Assured Testing Services.
See attached photos.*

Part Test Information										
PART NUMBER	LOT NUMBER	S#	SAMPLE DESCRIPTION							
TEST SPECIFICATION NAME		TEST PROCEDURE NAME				HOURS		CYCLES		
TEST RESULTS		INDATE	OUTDATE	TESTCNT	RETCNT	REQ	COMP	REQ	HOURS	COMP
3" x 5" Steel Panels										
Unspecified										
		06-Apr-17	02-Nov-17	1	1				15	15
		Cyclic Salt Fog / UV Exposure ASTM D5894-16								
		Dark Green - PSX Panel								
<i>Results: See attached data table.</i>										
<i>Requirements: Evaluate every 3 cycles (1000 hours).</i>										

Michele Singer

Revision# 1.11 Date 11/18/16

End Of Report



Assistant Laboratory Manager

The results stated above relate only to the specific items tested. Information and statements in this report are derived from material, information, and/or specifications furnished by the client and exclude any expressed or implied warranties as to the fitness of material tested or analyzed for any particular purpose or use. This report is confidential property of our client and may not be used for advertising purposes. This report shall not be reproduced except in full, without written approval of this laboratory. This recording of false, fictitious or fraudulent statements or entries on this document may be punished as a felony. Sample remnants are retained for a minimum of 6 months following issuance of test results, at which point they will be discarded unless notified in writing by the client. Any client claims or damages arising from the use of information in this report are strictly limited to the cost of the services provided by Assured Testing Services.

Photos: Testing IAW ASTM D5894-16 Cyclic Salt Fog / UV Exposure



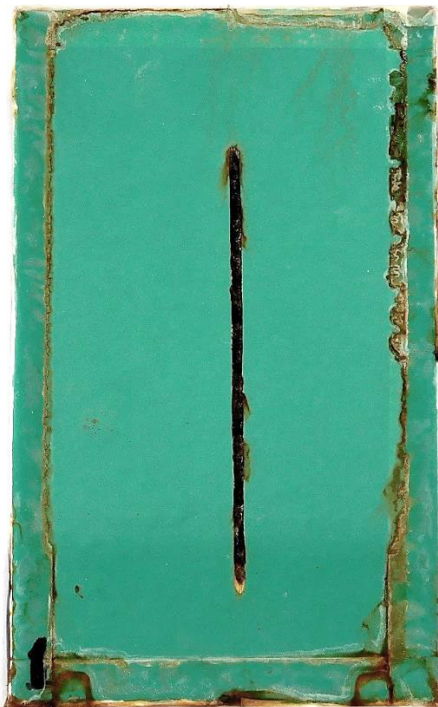
Sample Panel #1 - Dark Green prior to Exposure IAW ASTM D5894-16 (see above)



Sample Panel #1 - Dark Green at 3 Cycles Exposure IAW ASTM D5894-16 (see above)



Sample Panel #1 - Dark Green at 6 Cycles Exposure IAW ASTM D5894-16 (see above)



Sample Panel #1 - Dark Green at 9 Cycles Exposure IAW ASTM D5894-16 (see above)



Sample Panel #1 - Dark Green at 12 Cycles Exposure IAW ASTM D5894-16 (see above)



Sample Panel #1 - Dark Green at 15 Cycles Exposure IAW ASTM D5894-16 (see above)

End of photo pages

EONCOAT

Federal Railroad
Administration (FRA) Rail
Base Corrosion & Cracking
Prevention Study

Testing performed by EWI – Final report July 2014

Overview:

This is a synopsis of the Federal Railroad Administration (FRA) Rail Base Corrosion & Cracking Prevention Study. The summary outlines the background reasons for the study, a brief overview of the testing performed and the results of the tests, along with references to pages within the study itself (the full study is included behind this summary).

6 pieces of 20 foot rail were tested, each at a specific load range put through stress cycles, until the specimen failed. As the report below will explain, of the 3 specimens of rail that were coated, only the rail coated with EonCoat™ lasted so long (without failure) that the test was eventually stopped, after almost 3.5 million cycles.

In addition to the stress/fatigue testing, a steel panel coated with EonCoat™ was tested in an ASTM B117 chamber, against a coating specified by the US Navy for use inside ballast tanks. At 5,000 hours in the chamber, the test was stopped as the EonCoated plate had less damage at 5,000 hours than the competition did after only 500.

Background:

Rail base corrosion combined with fatigue or damage can significantly reduce rail life. Reducing or mitigating corrosion is directly tied to safety in operations and infrastructure for the rail industry.

Efforts to control corrosion on track segments are not new. Most of these efforts involve protecting the rail with a coating or paint-like material. This can provide a barrier to corrosive elements, but are temporary measures as the barrier will ultimately be breached, due to wear and/or abrasion.

In this study, EWI tested rail treatments that were created not to act as a barrier between the elements and rail, but that are designed to alter the surface chemistry of the rail in such a way that mitigates or rejects corrosion.

This study was funded by the **US Department of Transportation Federal Railroad Administration**. The attached report is publicly available through the FRA website at www.fra.dot.gov or by calling 202-493-1300.

The testing and reporting was performed by **EWI** (previously known as Edison Welding Institute) headquartered in Columbus, Ohio. EWI is considered one of the leading engineering and technology innovators for advanced manufacturing in North America.

It is important to note that EonCoat, LLC was contacted by EWI and did not solicit involvement in this research.

Summary:

(Note: the 2 paragraphs below are taken directly from the executive summary on page 1 of the report. Italics are ours):

Rail is subjected to fatigue loads in normal service. These loads can lead to small cracks in the rail. When corrosion forms in these cracks, the rail loses significant strength. For this program, the combined effects of damage, corrosion, and fatigue were examined under controlled conditions to ascertain the relative contribution of each to the overall condition of the rail and to measure the performance of anti-corrosion treatments in limiting corrosion and extending the rail's fatigue life.

An organometallic conversion type coating system, investigated at EWI for use in high pressure liquid water systems, was useful in retarding the effects of corrosion on fatigue rail life. However, it did not prevent corrosion in the presence of saltwater or condensing humidity. *A commercially available inorganic conversion coating (EonCoat®) was effective in preventing corrosion in saltwater environments. It enabled extended fatigue life, even on damaged rail subjected to aggressive corrosive environments, and gave performance similar to that for undamaged, uncorroded rail.*

Testing:

- To simulate small cracks produced by load fatigue, notches were carved in the rail
- To promote corrosion, pieces of rail were placed in an ASTM B117 salt fog chamber (the standard industry corrosion test)
- Fatigue was measured by cycles to failure, with a specific load range

Six specimens were tested (see page 7 for more detail on specimens; See page 15 for results chart):

- 1) **Specimen 1:** Negative Control
 - **Rail Condition:** Undamaged rail, no protection, no exposure to corrosion (this was the control specimen using just a flange base).
 - **Result:** An undamaged, unprotected 20 foot piece of rail will not fail until 5,000,000 cycles.

2) **Specimen 2:** Positive Control (no corrosion)

- **Rail Condition:** Untreated rail, damaged (notched), but without corrosion.
- **Result:** An untreated piece of rail that is damaged, but not subject to corrosion, will fail much faster at roughly 305,000 cycles.

3) **Specimen 3:** Positive Control

- **Rail Condition:** Untreated rail, notched, with corrosion exposure (this represents the baseline field installation in a corrosive environment such as a rail tunnel or drip zone).
- **Results:** An untreated piece of rail that is damaged AND exposed to corrosion (placed in the salt fog chamber) will fail even faster at 61,000 cycles.

Untreated

4) **Specimen 4:** ALL Method

- **Rail Condition:** A three-step treated rail section, notched, with corrosion exposure.
- **Results:** A piece of rail that is damaged AND exposed to corrosion, but coated with the EWI 3 part coating systems greatly extended the life of the rail to roughly 218,000 cycles. A vast improvement over the untreated specimen 3. (See pages 4 & 5 for more information about the EWI coating system).

5) **Specimen 5:** DP Method

- **Rail Condition:** A two-step treated rail section, notched, with corrosion exposure.
- **Result:** A piece of rail that is damaged AND exposed to corrosion, coated with the EWI 2 part coating system failed at 226,000 cycles. Again, a vast improvement over specimen 3. (See pages 4 & 5 for more information about the EWI coating system).

6) **Specimen 6:** EonCoat™

- **Rail Condition:** An EonCoat™ treated rail section, notched, with corrosion exposure.
- **Result:** A piece of rail that is damaged and exposed to corrosion with only one coat of EonCoat™ ran for 3,460,278 cycles, at which point the test was stopped because the rail had not yet failed.
- *With EonCoat, damaged rail in a corrosive environment was performing as well as brand new, undamaged rail that was not subjected to corrosion.*

EONCOAT

Additional Salt-Fog Testing on Standard Panels:

In addition to the stress corrosion testing, EWI tested EonCoat™ in the ASTM B117 Salt Fog Chamber, on their standard steel panels. The test was conducted pitting EonCoat against a high-build anti-corrosion coating that meets the US Navy specification for use inside ballast tanks (see page 49 for more details about comparison coating and test).

The purpose of this test was to examine the absolute corrosion protection ability of the two systems, regardless of their application to rail.

Results:

- After 500 hours, the EWI sample showed attack on the scribe line (Figure B-1, page 50), while the EonCoat system showed very little attack.
- After 5,000 hours, the EWI system showed significant damage including undercutting and blistering (figure B-2, page 50), while the EonCoat system was still offering good protection.
- Refer to pictures on page 50 for visual comparison.

This additional testing was performed unsolicited by EonCoat, LLC.

ASTM B117 Conclusion:

5,000 hours of ASTM B117 requires roughly 7 months to conclude. This test result, performed unsolicited by a highly reputable industry leading testing and research facility proves, indisputably, that EonCoat corrosion resistant coating can easily withstand 5,000 hours in a salt fog.

For companies needing ASTM B117 testing prior to specifying a coating, these test results can save the cost and time involved. However, we will be happy to coordinate sending coated steel plates for any company wishing for validate test results on their own.

Note of clarification about actual EonCoat product used:

At the time this testing was performed from 2013 through 2014, EonCoat had one coating available for market. For the purposes of this report, where ever the report refers to “EonCoat” as the product, the actual coating used in the test is now called “EonCoat CR”, the corrosion resistant coating produced by the manufacturer, EonCoat, LLC.



U.S. Department of
Transportation
**Federal Railroad
Administration**

Rail Base Corrosion and Cracking Prevention

Office of Research
and Development
Washington, DC 20590



NOTICE

This document is disseminated under the sponsorship of the Department of Transportation in the interest of information exchange. The United States Government assumes no liability for its contents or use thereof. Any opinions, findings and conclusions, or recommendations expressed in this material do not necessarily reflect the views or policies of the United States Government, nor does mention of trade names, commercial products, or organizations imply endorsement by the United States Government. The United States Government assumes no liability for the content or use of the material contained in this document.

NOTICE

The United States Government does not endorse products or manufacturers. Trade or manufacturers' names appear herein solely because they are considered essential to the objective of this report.

REPORT DOCUMENTATION PAGE			<i>Form Approved</i> <i>OMB No. 0704-0188</i>
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.			
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE July 2014	3. REPORT TYPE AND DATES COVERED Final (September 2012 – June 2013)	
4. TITLE AND SUBTITLE Rail Base Corrosion and Cracking Prevention		5. FUNDING NUMBERS DTFR-12-C-0024	
6. AUTHOR(S) Dr. George W. Ritter, EWI; Dr. William C. Mohr, EWI; Dr. David Y. Jeong, Volpe NTS Center; Yim A. Tang, Volpe NTS Center; Cameron Stuart, Federal Railroad Administration, US DOT			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) EWI 1250 Arthur E. Adams Drive Columbus, OH 43221		8. PERFORMING ORGANIZATION REPORT NUMBER EWI Project No. 53331GTH (Rev. 2)	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Department of Transportation Federal Railroad Administration Office of Research and Development Washington, DC 20590		10. SPONSORING/MONITORING AGENCY REPORT NUMBER DOT/FRA/ORD-14/27	
11. SUPPLEMENTARY NOTES COTR: Cameron D. Stuart			
12a. DISTRIBUTION/AVAILABILITY STATEMENT This document is available to the public through the FRA Web site at http://www.fra.dot.gov or by calling (202) 493-1300.		12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Rail base corrosion combined with fatigue or damage can significantly reduce rail life. Studies were done to examine the relative contribution of damage, corrosion, and fatigue on rail life. The combined effects can be separated into constituent factors. Anti-corrosion treatments based on surface passivation of steel have been shown to extend rail fatigue life in the presence of damage and corrosion.			
14. SUBJECT TERMS Rail base corrosion, corrosion, corrosion treatments, fatigue, rail life		15. NUMBER OF PAGES 60	
		16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT SAR

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

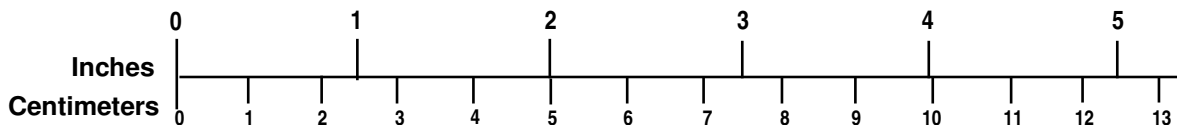
METRIC/ENGLISH CONVERSION FACTORS

ENGLISH TO METRIC

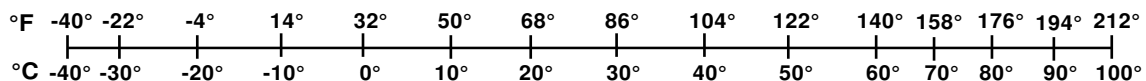
METRIC TO ENGLISH

<p>LENGTH (APPROXIMATE)</p> <p>1 inch (in) = 2.5 centimeters (cm)</p> <p>1 foot (ft) = 30 centimeters (cm)</p> <p>1 yard (yd) = 0.9 meter (m)</p> <p>1 mile (mi) = 1.6 kilometers (km)</p>	<p>LENGTH (APPROXIMATE)</p> <p>1 millimeter (mm) = 0.04 inch (in)</p> <p>1 centimeter (cm) = 0.4 inch (in)</p> <p>1 meter (m) = 3.3 feet (ft)</p> <p>1 meter (m) = 1.1 yards (yd)</p> <p>1 kilometer (km) = 0.6 mile (mi)</p>
<p>AREA (APPROXIMATE)</p> <p>1 square inch (sq in, in²) = 6.5 square centimeters (cm²)</p> <p>1 square foot (sq ft, ft²) = 0.09 square meter (m²)</p> <p>1 square yard (sq yd, yd²) = 0.8 square meter (m²)</p> <p>1 square mile (sq mi, mi²) = 2.6 square kilometers (km²)</p> <p>1 acre = 0.4 hectare (he) = 4,000 square meters (m²)</p>	<p>AREA (APPROXIMATE)</p> <p>1 square centimeter (cm²) = 0.16 square inch (sq in, in²)</p> <p>1 square meter (m²) = 1.2 square yards (sq yd, yd²)</p> <p>1 square kilometer (km²) = 0.4 square mile (sq mi, mi²)</p> <p>10,000 square meters (m²) = 1 hectare (ha) = 2.5 acres</p>
<p>MASS - WEIGHT (APPROXIMATE)</p> <p>1 ounce (oz) = 28 grams (gm)</p> <p>1 pound (lb) = 0.45 kilogram (kg)</p> <p>1 short ton = 2,000 pounds (lb) = 0.9 tonne (t)</p>	<p>MASS - WEIGHT (APPROXIMATE)</p> <p>1 gram (gm) = 0.036 ounce (oz)</p> <p>1 kilogram (kg) = 2.2 pounds (lb)</p> <p>1 tonne (t) = 1,000 kilograms (kg) = 1.1 short tons</p>
<p>VOLUME (APPROXIMATE)</p> <p>1 teaspoon (tsp) = 5 milliliters (ml)</p> <p>1 tablespoon (tbsp) = 15 milliliters (ml)</p> <p>1 fluid ounce (fl oz) = 30 milliliters (ml)</p> <p>1 cup (c) = 0.24 liter (l)</p> <p>1 pint (pt) = 0.47 liter (l)</p> <p>1 quart (qt) = 0.96 liter (l)</p> <p>1 gallon (gal) = 3.8 liters (l)</p> <p>1 cubic foot (cu ft, ft³) = 0.03 cubic meter (m³)</p> <p>1 cubic yard (cu yd, yd³) = 0.76 cubic meter (m³)</p>	<p>VOLUME (APPROXIMATE)</p> <p>1 milliliter (ml) = 0.03 fluid ounce (fl oz)</p> <p>1 liter (l) = 2.1 pints (pt)</p> <p>1 liter (l) = 1.06 quarts (qt)</p> <p>1 liter (l) = 0.26 gallon (gal)</p> <p>1 cubic meter (m³) = 36 cubic feet (cu ft, ft³)</p> <p>1 cubic meter (m³) = 1.3 cubic yards (cu yd, yd³)</p>
<p>TEMPERATURE (EXACT)</p> <p>$[(x-32)(5/9)] \quad [= y \quad] \quad \square C$</p>	<p>TEMPERATURE (EXACT)</p> <p>$[(9/5) y + 32] \quad [= x \quad] \quad \square F$</p>

QUICK INCH - CENTIMETER LENGTH CONVERSION



QUICK FAHRENHEIT - CELSIUS TEMPERATURE CONVERSION



For more exact and or other conversion factors, see NIST Miscellaneous Publication 286, Units of Weights and Measures. Price \$2.50 SD Catalog No. C13 10286

Updated 6/17/98

Contents

Executive Summary	1
1. Introduction.....	2
1.1 Background.....	2
1.2 Objectives	3
1.3 Technical Approach.....	3
2. Experimental Activities	4
2.1 Task 1 – Analyze Surface Chemistry on Rail (Small Scale)	4
2.1.1 Sample Corrosion Exposures.....	4
2.1.2 SEM and EDS Analyses	6
2.2 Task 2 – Treatments on Rail Stock (Medium-Scale) and Tie Plates	6
2.2.1 Preparation and Treatment of Medium-Scale Specimens.....	8
2.2.2 Corrosive Environment Exposures	11
2.2.3 Sample for Drip Zone Protection.....	13
2.2.4 Salt Fog Testing	13
2.3 Task 3 – Crack Fatigue Propagation and Mitigation (Medium-Scale)	14
2.4 Task 4 – Resonant Fatigue Testing (Rail Sections)	15
2.4.1 Resonant Fatigue Methodology	15
2.4.2 Preparation of Rail Sections.....	16
2.4.3 Corrosion Exposure Cycling for Large Rail Segments.....	18
3.0 Results and Discussion	19
3.1 Task 1 – Small-Scale Corrosion Exposure - Results and Discussion.....	19
3.1.1 Surface Studies.....	19
3.1.2 Results from SEM/EDS Analyses	22
3.1.3 Crosscut Samples.....	25
3.1.5 Top Surface Analyses – Elemental Content.....	28
3.1.6 Implications for Protecting Rail Steel.....	29
3.2 Task 2 – Treatments on Rail Stock (Medium-Scale).....	29
3.2.1 Drip Zone Samples	32
3.2.2 Salt Fog Testing	33
3.3 Task 3 – Crack Fatigue Propagation and Mitigation (Medium-Scale)	33
3.4 Task 4 – Resonant Fatigue Testing (Rail Sections).....	36
4.0 Conclusions and Recommendations	41
4.1 Conclusions	41
4.2 Recommendations for Future Work	41
5.0 References	42
Appendix A. Pictures of Corrosion Samples from Task 2.....	43
Appendix B. Salt Fog Testing.....	49
Abbreviations and Acronyms	53

Illustrations

Figure 1. High-Level Work Breakdown Structure	3
Figure 2. Flange Detail Showing Bottom Cut Notch (Right)	7
Figure 3. (ALL) Specimens in Bake Furnace	8
Figure 4. Bake Schedule for (ALL) Specimens.....	9
Figure 5. PD Two-Step Treated Sample Set.....	9
Figure 6. ALL Three-Step Treated Sample Set	10
Figure 7. EonCoat® Treated Sample Set.....	10
Figure 8. No Treatment Flange Base	11
Figure 9. PD Samples on First Exposure to Saltwater.....	12
Figure 10. Placement of Samples in Humidity Cabinet.....	12
Figure 11. Four-Point Load Fatigue Test Setup.....	14
Figure 12. Rail Suspended in Resonant Fatigue Apparatus.....	16
Figure 13. Resistance Heating Blankets used to Heat Cure Zirconia Sol-Gel Sealer.....	17
Figure 14. Technician Readies EonCoat® Applicator System.....	17
Figure 15. EonCoat® Sprayed Rail Segment	18
Figure 16. Bottoms of Samples after 2-Week Corrosion Cycle	20
Figure 17. Bottoms of Samples after 3-Week Corrosion Cycle	20
Figure 18. Tops of Samples after 2-Week Corrosion Cycles	21
Figure 19. Tops of Samples after 3-Week Corrosion Cycle.....	21
Figure 20. Photograph Summary of Samples for SEM/EDS Examination	23
Figure 21. Close-Up of PD3/4 Series	23
Figure 22. Close-Up of PDZ3/4 Series.....	24
Figure 23. Close-Up of ALL1/ALL2 Series	24
Figure 24. SEM Photographs of Cross Cuts for PD3 (Left) and PD4 (Right).....	25
Figure 25. SEM Photographs of Cross Cuts for PDZ3 (Left) and PDZ4 (Right).....	25
Figure 26. SEM Photographs of Cross Cuts for ALL1 (Left) and ALL2 (Right)	26
Figure 27. SEM Photographs of Top Surfaces for PD3 (Left) and PD4 (Right).....	27
Figure 28. SEM Photographs of Top Surfaces for PDZ3 (Left) and PDZ4 (Right).....	28
Figure 29. SEM Photographs of Top Surfaces for ALL1 (Left) and ALL2 (Right)	28
Figure 30. PD Treated Samples after 3-Week Exposure (Tie Plate and Flange Bottom)	30
Figure 31. ALL Samples after 3-Week Exposure.....	30
Figure 32. Untreated Samples after 3-Week Exposure.....	31
Figure 33. EonCoat® Samples after 3-Week Exposure	31
Figure 34. Drip Zone Top Surface after 3 Weeks.....	32
Figure 35. Drip Zone Bottom Surface after 3 Weeks	33
Figure 36. Fracture Surface of Corroded Sample with No Protection.....	35
Figure 37. Fracture Surface of Corroded Sample with ALL Treatment.....	35
Figure 38. Resonant Fatigue Test Lifetime Results.....	37
Figure 39. Resonant Fatigue Fracture Surface of Unprotected, Corroded Rail (Sample RS3) Arrow Shows Crack Zone.....	38
Figure 40. Fracture Surface of Corroded Sample with No Protection (Crack – corrosion area is circled)	39
Figure 41. Fracture Surface of Corroded Sample with ALL Treatment.....	40

Tables

Table 1. Sample Disposition for Treatments	4
Table 2. Corrosion Cycles for Even-Numbered Specimens	6
Table 3. Corrosion Exposure Schedule.....	11
Table 4. Drip Zone Corrosion Cycles	13
Table 5. Four-Point Load Fatigue Testing.....	15
Table 6. Visual Interpretation of Corrosion Results	22
Table 7. Summary of EDS Analyses for Cross Cut Sample Coatings.....	26
Table 8. Summary of EDS Analyses for Sample Coating Surfaces	29
Table 9. Fatigue on Medium-Scale Samples	34
Table 10. Summary of Resonant Fatigue Testing.....	36

Executive Summary

Rail base corrosion can take place when water gets trapped between the flange base and the tie plate. Corrosion is accelerated by the presence of salt in the water and electrical current transmitted through the rail base and the tie plate path to earth ground. This corrosion results in the loss of rail base material and strength. Ultimately, it is possible for the rail to fail. Rail base corrosion has been suspected as a cause for derailments in the recent past. This program was initiated to examine treatment methods to prevent or forestall rail base corrosion. The program was funded by the Federal Railroad Administration (FRA) and executed by Edison Welding Institute (EWI).

The most obvious way to protect steel against corrosion is to paint it with a barrier-type coating. Barrier-type coatings, like paint, have a limited life due to loss of adhesion and/or abrasive wear. Another approach is to treat the rail surface chemically to passivate (protect) the surface against rusting or slow the progress of rusting substantially. This was the overall approach used in this work.

Rail is subjected to fatigue loads in normal service. These loads can lead to small cracks in the rail. When corrosion forms in these cracks, the rail loses significant strength. For this program, the combined effects of damage, corrosion, and fatigue were examined under controlled conditions to ascertain the relative contribution of each to the overall condition of the rail and to measure the performance of anti-corrosion treatments in limiting corrosion and extending the rail's fatigue life.

An organometallic conversion type coating system, investigated at EWI for use in high pressure liquid water systems, was useful in retarding the effects of corrosion on fatigue rail life. However, it did not prevent corrosion in the presence of saltwater or condensing humidity. A commercially available inorganic conversion coating (EonCoat®) was effective in preventing corrosion in saltwater environments. It enabled extended fatigue life, even on damaged rail subjected to aggressive corrosive environments, and gave performance similar to that for undamaged, uncorroded rail.

In this work, it was found that the interactions between rail damage, corrosive attack, and overall rail fatigue life can be examined by methodical application of traditional four-point fatigue testing using manageable sample sizes. The influences of corrosion and damage in the presence of fatigue stress are believed to be separable based on this limited testing. The presence of damage alone showed reduced rail fatigue life by as much as one order of magnitude. Rail fatigue life is reduced by as much as an additional order of magnitude when these damaged areas are corroded. The combined effects on rail life of damage, corrosion, and fatigue can be severe. Use of corrosion protection systems, applied to the base of the rail flange, can extend rail fatigue life.

A resonant fatigue test method was adapted for use with 20-foot long rail segments. It was shown that asymmetrical and heavy cross sections could, in fact, be induced into self-oscillation, developing roughly 60–80 ksi peak stress. In this work, the method was not found to be predictive of fatigue life for damaged, corroded rail, with or without corrosion protection. However, rail was taken to failure by this method, suggesting that a more advanced test methodology might be useful for screening rail fatigue phenomena.

Based on the results of this preliminary work, the researchers recommend continued analysis of these corrosion inhibiting treatments. Field trails of these treatments to support the laboratory results is a necessary step towards in-service deployment.

1. Introduction

Rail base corrosion has been observed by several rail operators in tunnels where water is periodically present at the base of the rail. The Transit Cooperative Research Program/Transportation Technology Center, Inc. (TCRP/TTCI) review⁽¹⁾ of this behavior found that the most severe corrosion was reported in tunnels in the New York City area. Some corrosion was invasive and found to advance as much as one-fourth inch up from the area where the rail base crosses tie plates. References containing this information also warned of the possibility that the edge of the corroded area could act as a stress concentrator that would allow fatigue crack growth up the rail.^(2,3) Further, the effects of corrosive antagonists can be amplified by mechanical abuse and fatigue, exposure to road deicing materials, and leakage currents for electrified rail. This program was initiated to investigate rail treatments that can mitigate the effects of humidity, saline exposure, and fatigue on crack growth in rail steels.

A recent failure in a tunnel in the New York City area was found to be a fatigue-type failure initiating from the rail base at the edge of the corroded area. Plans are underway for rail replacement in and beyond the area of the derailment. However, there is significant interest from operators such as Amtrak, Long Island Rail Road (LIRR), and Port Authority Trans-Hudson (PATH) in extending the life of rails in tunnels by minimizing or eliminating the issue of rail corrosion and associated fatigue cracking. Further, there are many areas throughout the country where similar corrosion, or corrosion caused by condensing or dripping water, occurs. Treatment of the rail bottom surface to limit the effect of corrosion mechanisms may help increase rail life, reliability, and overall rail safety.^(4,5)

Reducing or mitigating rail corrosion speaks directly to safety in operations and reliability of infrastructure. The technology under study is expected to be applicable to new rail and possibly to installed rail as a remedial measure. It is believed that the treated rail can be installed following established procedures. It may also be beneficial to use treated rail and tie plates in combination on electrified rail lines. Potential users with tunnels in the New York City area (Amtrak, PATH, LIRR, etc.) have been made aware of this technology development by representatives from the Volpe National Transportation Systems Center (Volpe).

1.1 Background

Efforts to control corrosion on track segments are not new. Most approaches involve protecting the rail with paint-like materials or coatings. These materials provide a barrier to corrosive elements, but they do not prevent their ultimate intrusion. Wear resistance is also an important factor in the lifetime of protection. In this research program, EWI tested rail treatments that are designed to alter the surface chemistry of the rail such that it impedes or rejects corrosion. Two of the treatments were based on work previously done at EWI for corrosion protection in nuclear piping.⁽⁶⁾ The third treatment is commercially available.⁽⁷⁾

When iron rusts, it oxidizes first to ferrous ion (Fe^{+2}). This process can be initiated by the influence of chloride salts found in road deicing mixtures. Further reactions between iron, oxygen, and water result in the formation of ferric ions (Fe^{+3}). All these species exist as oxides on the metal surface.

Interestingly, ferrous oxide (FeO – black rust) is rather stable in water, and the mixed oxide, Fe₃O₄, a mixture of FeO and Fe₂O₃, is also fairly stable. It is the red rust form, Fe₂O₃, that is commonly referred to as “rust” or “bleeding rust” – that is invasive and destructive of the iron underneath. If the surface oxides can be driven back to the more stable forms, such as FeO or Fe₃O₄ mixed oxide, “rusting” can be abated and controlled.

Corrosion prevention methods include creating a barrier (i.e. paint) to prevent corrosive elements from reaching the base metal, adding a sacrificial material to the steel (i.e., galvanizing), using electrical charge to reverse oxidation (cathodic protection), or using a chemical conversion to discourage the formation of ferric oxide rust. This research project examined surface conversion methods for rust mitigation or prevention.

1.2 Objectives

The primary objectives of this project were to:

- 1) Examine selected anti-corrosion treatments that render the rail surface immune to corrosion or mitigate its spread should initial corrosion occur.
- 2) Introduce elements of flexural fatigue to the rail to examine the combined effects of corrosion and fatigue in crack propagation.
- 3) Delineate, if possible, the relative contributions of damage, corrosion, and fatigue level to the reduction of rail fatigue life.
- 4) Examine the use of a resonant fatigue methodology for its applicability to rail, and specifically, as a predictive tool for this investigation. Compare this method with four-point load fatigue methods.

1.3 Technical Approach

A summarized Work Breakdown Structure is shown below.

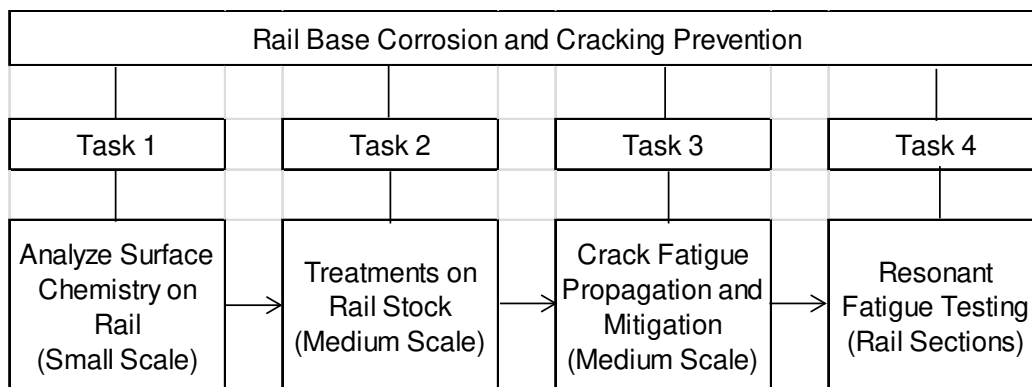


Figure 1. High-Level Work Breakdown Structure

2. Experimental Activities

2.1 Task 1 – Analyze Surface Chemistry on Rail (Small Scale)

Task 1 of this program examined protective treatments on small samples of rail steel and the effects of corrosive attack on surface chemistries. The objective was to document the effect of these treatments on rail steel and to determine if there was a need to heat-treat the ceramic precursor coating.

One-inch wide test specimens were cut from the web of standard 136-pound rail (common rail steel), degreased in an aqueous cleaner and wiped with isopropyl alcohol, and subsequently treated with EWI-developed combinations of phosphoric acid etch, an iron conversion coating, and a zirconium oxide sol-gel.⁽⁸⁾ Following these varied treatments, some samples were subjected to 3 weeks of cyclic corrosion, while other similarly treated counterparts were kept aside as controls. Those exposed to corrosion were then examined visually for their general performance and also analyzed using scanning electron microscopy (SEM) and elemental x-ray dispersive analysis (EDS) to examine surface condition and chemistry.

They were then treated according to the outline presented in Table 1.

2.1.1 Sample Corrosion Exposures

Table 1. Sample Disposition for Treatments

Sample Preparation							
	No Induced Corrosion	Induced Corrosion	P	D	Z	Bake	Total
2-step	PD1	PD2	X	X			6
	PD3	PD4	X	X			
	PD5	PD6	X	X			
	PZ1	PZ2	X		X	X	2
3-step	PDZ1	PDZ2	X	X	X		6
	PDZ3	PDZ4	X	X	X		
	PDZ5	PDZ6	X	X	X		
	ALL1	ALL2	X	X	X	X	2

P = phosphoric acid; D = di-phenolic converting agent; Z = zirconium-oxide sol-gel sealer

The EWI system can be applied using two or three treatment steps, as described below. Their designations are:

1. PD (two-step) – Consists of a treatment with phosphoric acid (P), followed by treatment with a di-phenolic converting agent (D).

2. PZ (two-step) – Consists of a treatment with phosphoric acid, followed by application of a zirconium-oxide producing sol-gel sealer (Z), followed by a post bake at 550°C with a 3-hour hold at temperature.
3. PDZ (three-step, no post bake) – The PD process is followed by application of a zirconium-oxide producing sol-gel sealer. There is no post bake.
4. ALL (three-step, with post bake) – The PDZ process, followed by a post bake at 550°C with a 3-hour hold at temperature.

P, D, and Z may be applied sequentially by spray or brush.

All rail specimens were treated with a 10 percent phosphoric acid solution (P) (by weight in water). Grit-blasting was used to remove surface rust to get down to base metal.

Air drying following the phosphoric acid application produced a white, powdery material on the surface. The surface was then wiped with water to remove the excess crystal formation.

For the D treatment, the phosphated rail was treated with the di-phenolic reducing agent, also brush applied. A deep blue-black color emerged, indicating the formation of the mixed metal oxide of $\text{FeO} \cdot \text{Fe}_2\text{O}_3$. After drying for 4–6 hours, the sample was washed with water to remove excess reducing agent.

After the P and any applied D treatment, the Z step consisted of applying a zirconia sol-gel. A sol-gel is a ceramic precursor solution derived from an organometallic precursor in alcohol solvent. The precursor hydrolyzes to form an oxy-hydroxide zirconate that can be heated to form zirconium oxide ceramic. The preparation of this material was taken from the literature.⁽⁸⁾ Heating the coated rail to a temperature of 550°C for 3 hours converted the sol-gel to a form of zirconium oxide. This temperature is well below the transition temperature between ferrite and austenite for common rail steels (~700°C), thus avoiding martensite formation upon cooling. The hardness of these treated steels never dropped below 325 Brinell.

The three-step process (ALL) used a muffle furnace for the bake step. There were two two-step variants. One involved using only the phosphate and the di-phenol, with no zirconia seal or bake (PD). The other used the phosphate treat and zirconia seal with a bake (PZ).

The bake for PZ1, PZ2, ALL1, and ALL2 specimens consisted of heating them in air at 550°C for 3 hours to set the zirconia ceramic seal coat. (Testing showed that this heat soak cycle of rail steel in air gave a Brinell hardness of 329.) PZ1 and PZ2 were not treated with the iron conversion coating, but were baked to produce possible iron-zirconium phosphate complexes. ALL1 and ALL2 were also baked.

The “even numbered” specimens were subjected to a 3-week cycle of corrosion, while the “odd numbered” specimens were set aside as controls. The corrosion cycle is given in Table 2. The saline solution was made up of a 5 percent by weight addition of road deicing salt in tap water. Road deicing salt also contains calcium chloride and magnesium salts in addition to the predominant sodium chloride (rock salt). The hot and wet conditions were 50°C with 98–100 percent relative humidity in a closed-temperature humidity chamber. This type of corrosion cycle was designed to be exceptionally severe compared with normal operating conditions for rail, so that differences in corrosion protection or attack could be shown rapidly.

Table 2. Corrosion Cycles for Even-Numbered Specimens

Corrosion Cycles							
Week 1	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	1	2	3	4	5	6	7
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
Week 2	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	8	9	10	11	12	13	14
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
Week 3	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	15	16	17	18	19	20	21
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
* Samples removed and pictures taken							

2.1.2 SEM and EDS Analyses

Sample bars representing the different treatments and corrosion exposure histories were taken from the series and examined with a Zeiss EVO Model 60 scanning electron microscope equipped with an Oxford Instruments X-Max Energy Dispersive Spectroscopy (EDS) for elemental analysis. The sample selection was based on visual appearance and apparent resistance to corrosion attack.

2.2 Task 2 – Treatments on Rail Stock (Medium-Scale) and Tie Plates

Treatments that showed the best corrosion resistance in Task 1 were selected for further study in Task 2 and Task 3 of the program. In Task 2, larger rail segments (~6- × 18-in. segments of flange bases) were notched (Figure 2), treated, and exposed for a period of 3 weeks to an aggressive corrosive environment consisting of a saltwater soak and condensing humidity.

Based on the results from Task 1, the PD two-step variant and the ALL system three-step variant were selected to be treated in Task 2 and tested in Task 3. EonCoat®, a commercially-available material, was also tested.

EonCoat® is a commercially spray-applied ceramic coating.⁽⁷⁾ The overall formulation is proprietary. It is applied using standard two-component spray equipment with a mixer-head nozzle. It dries within minutes of application. A second coat can be applied in approximately 10–15 minutes, which was the practice used here. EonCoat® was applied by the vendor, and the plate sets were returned to EWI.

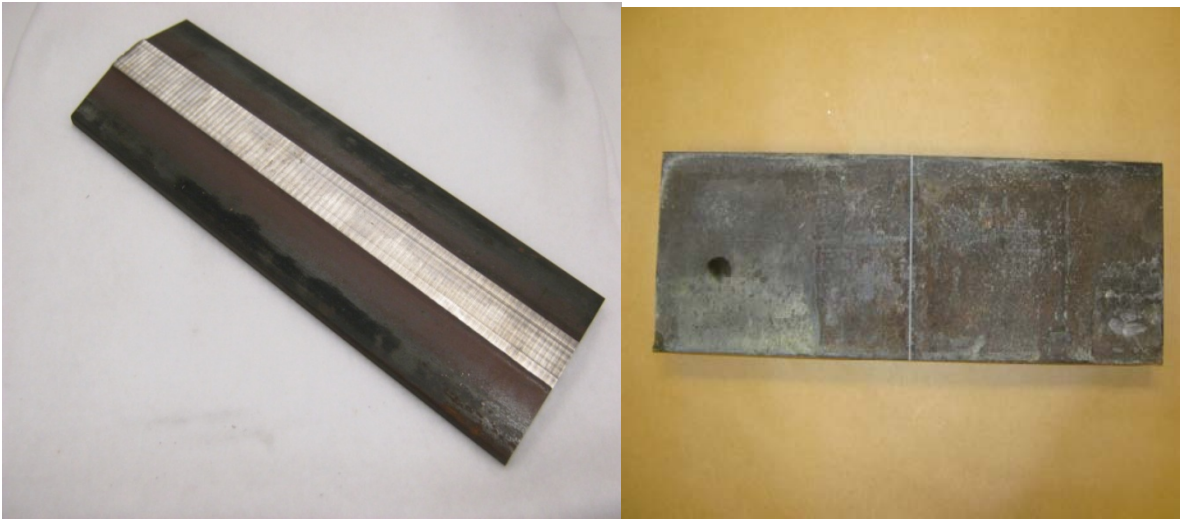


Figure 2. Flange Detail Showing Bottom Cut Notch (Right)

Six specimens were tested:

1. Undamaged rail, no protection, no corrosion (control using just a flange base);
2. Untreated rail, damaged (notched), but with no corrosion. This represents a baseline field installation that has been in use but is not in a corrosive environment ordinarily;
3. Untreated rail, notched, with corrosion exposure. This was resting on an untreated tie plate throughout the corrosion cycling. This represents the baseline field installation in a corrosive environment, such as a rail tunnel or drip zone;
4. The three-step treated rail section (ALL), notched, with corrosion exposure. This was resting on an ALL-treated tie plate throughout the corrosion cycling. This shows the effect of trapped corrosive elements between the bottom of the rail and the tie plate having protection by the ALL method;
5. The two-step treated rail section (DP), notched, with corrosion exposure. This was resting on a DP-treated tie plate throughout the corrosion exposure. This shows the effect of trapped corrosive elements between the bottom of the rail and the tie plate having protection by the DP method; and
6. An EonCoat® treated rail section, notched, with corrosion exposure. This was resting on an EonCoat® treated tie plate throughout the corrosion exposure. This shows the effect of trapped corrosive elements between the bottom of the rail and the tie plate having protection by the EonCoat® material.

Notching, if used, was done *before* any treatments were applied and/or before corrosion exposure.

2.2.1 Preparation and Treatment of Medium-Scale Specimens

All the flange pieces and tie plates were grit-blasted to remove excess scale and rust. The appropriate treatments were then applied using the same approaches as those for Task 1. The ALL three-step samples were baked at 550°C for 3 hours to set the zirconium oxide sol-gel sealer. The samples, as placed into the bake furnace, are shown in Figure 3, and the thermocouple trace for the bake is shown in Figure 4.



Figure 3. (ALL) Specimens in Bake Furnace

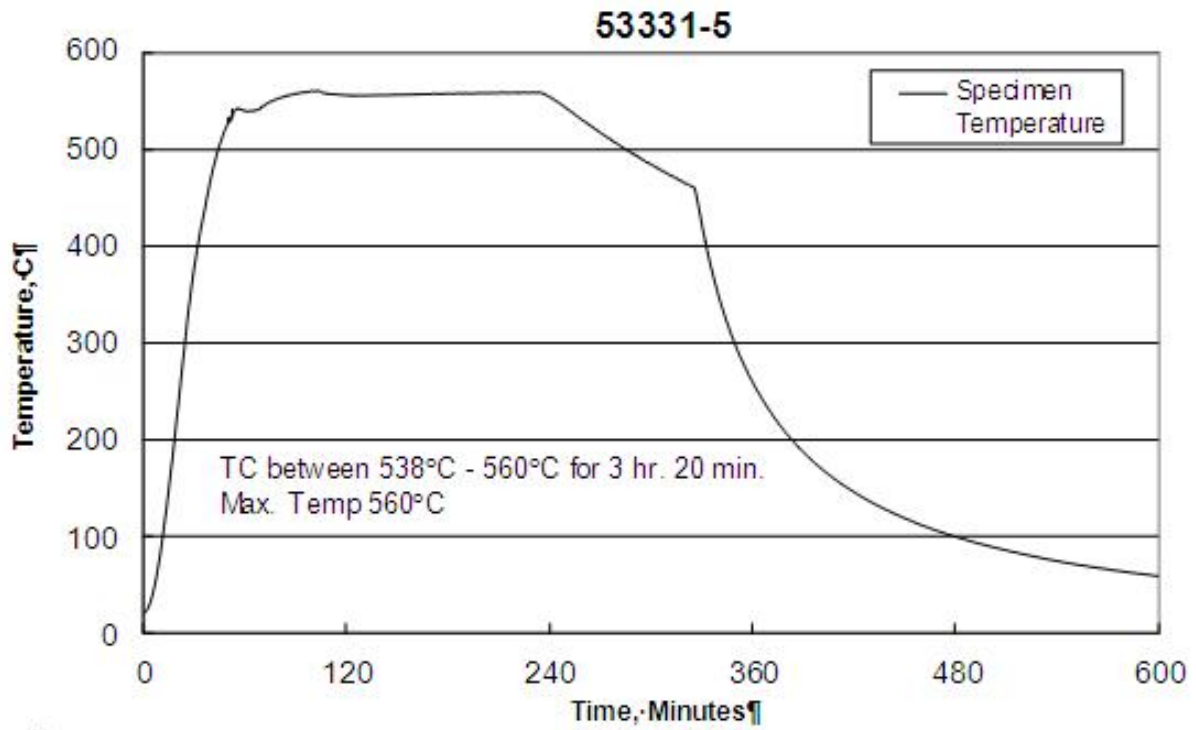


Figure 4. Bake Schedule for (ALL) Specimens

Figure 5 through Figure 8 show the specimens just prior to corrosion exposure.



Figure 5. PD Two-Step Treated Sample Set



Figure 6. ALL Three-Step Treated Sample Set

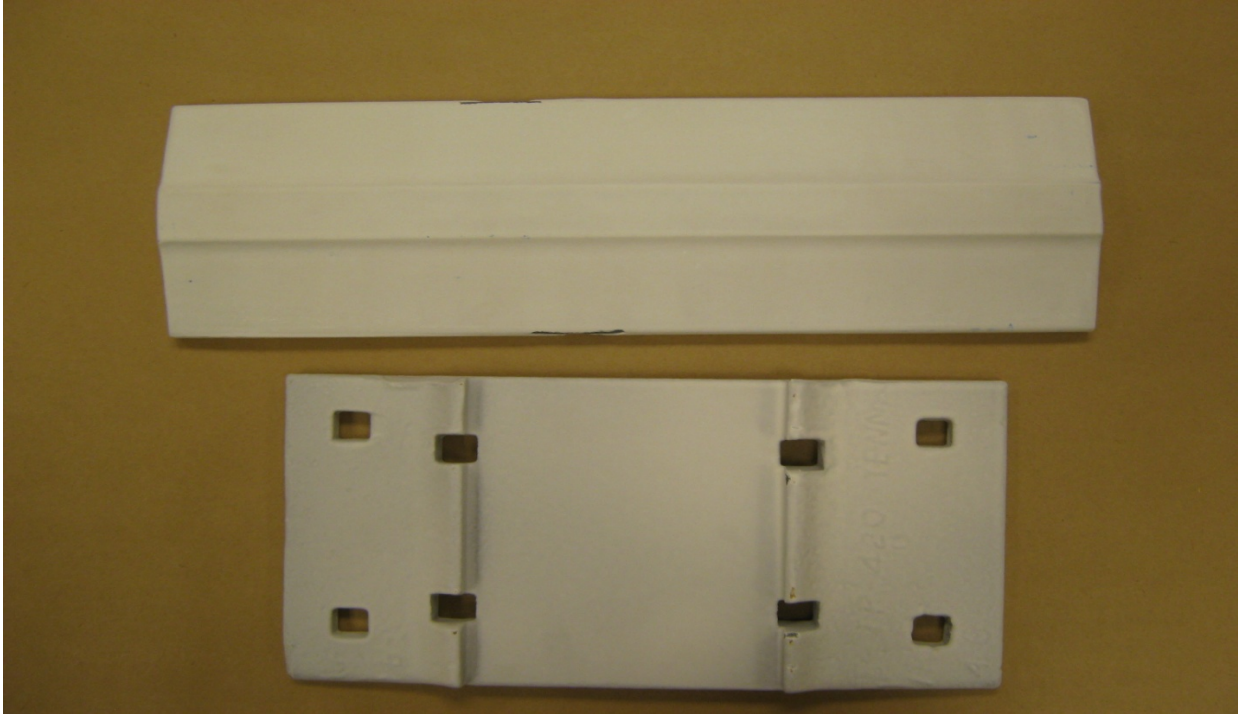


Figure 7. EonCoat® Treated Sample Set



Figure 8. No Treatment Flange Base

2.2.2 Corrosive Environment Exposures

The schedule for exposure is given in Table 3.

Table 3. Corrosion Exposure Schedule

Corrosion Cycles							
Week 1	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	8-Jan	9-Jan	10-Jan	11-Jan	12-Jan	13-Jan	14-Jan
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
Week 2	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	15-Jan	16-Jan	17-Jan	18-Jan	19-Jan	20-Jan	21-Jan
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
Week 3	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	22-Jan	23-Jan	24-Jan	25-Jan	26-Jan	27-Jan	28-Jan
Exposure	Salt water	Salt water	Dry (out)	Humidity	Humidity	Humidity	Dry (out)
Bold days-samples get moved							
*Thursdays and Mondays, take pictures of interfacial regions							

The saltwater solution was fixed at 5 percent by weight in a water solution of road deicing salt in tap water. Deicing salt contains magnesium chloride and calcium chloride in addition to the predominant sodium chloride. When the samples were first placed in saltwater, the PD system began to bleed blue-black color. (This also happened with the small-scale test bars in Task 1.) It was found later that this only happened when first immersed. In subsequent weeks of exposure, there was no additional bleeding (see Figure 9).



Figure 9. PD Samples on First Exposure to Saltwater

The hot-wet exposure took place in a large temperature and humidity controlled cabinet set at 50°C with saturated humidity. For the humidity exposures, the stacked specimens were placed into the controlled temperature-humidity cabinet, as shown in Figure 10. Notice it is possible for condensate from the upper samples to drip down onto the lower samples.



Figure 10. Placement of Samples in Humidity Cabinet

2.2.3 Sample for Drip Zone Protection

An additional test was added during Task 2 to examine the ability of the treatments to inhibit corrosion due to drip condensation. EWI prepared an additional specimen with the ALL treatment. For the humidity exposure, a “drip tent” was fashioned inside the chamber to collect and direct condensate onto the top surface. The saltwater soak was replaced by an additional 2 days of humidity exposure. The ALL treatment was applied to one half of the top and bottom surfaces for this exposure. The exposure schedule for the drip zone test is given in Table 4.

Table 4. Drip Zone Corrosion Cycles

Corrosion Cycles - Drip							
Week 1	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	4-Feb	5-Feb	6-Feb	7-Feb	8-Feb	9-Feb	10-Feb
Exposure	Humidity	Humidity	Out	Humidity	Humidity	Humidity	Dry (out)
Week 2	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	11-Feb	12-Feb	13-Feb	14-Feb	15-Feb	16-Feb	17-Feb
Exposure	Humidity	Humidity	Out	Humidity	Humidity	Humidity	Dry (out)
Week 3	Tues	Weds	Thurs*	Fri	Sat	Sun	Mon*
Day	18-Feb	19-Feb	20-Feb	21-Feb	22-Feb	23-Feb	24-Feb
Exposure	Humidity	Humidity	Out	Humidity	Humidity	Humidity	Dry (out)
Bold days-samples get moved							
Only one sample. Place flange down on bottom shelf.							
Make inverted-Vee foil tent to drip condensate onto the top.							
*Thursdays and Mondays, take pictures of interfacial regions							

2.2.4 Salt Fog Testing

EWI performed salt fog testing (ASTM B117) on a separate set of treated specimens. Specimens were produced from 4×12-inch plates of 1018 steel, approximately ³/₁₆-inch thick to accommodate the ASTM B117 test chamber. These specimens were treated using the EWI ALL processing as a primer and a subsequent overcoating of PPG® PSX-700 epoxy-silicate paint. PSX-700 is approved by the U.S. Navy for use in ballast tank lining for ships and may stay in service for years. Salt fog testing was included to benchmark the EWI ALL system as a primer with a high-performance corrosion protection barrier coating, such as the PSX-700. The EonCoat® was included to rank its performance against the latter. The samples were sent to an outside vendor for exposure to 5,000 hours of salt fog exposure following ASTM B117.

2.3 Task 3 – Crack Fatigue Propagation and Mitigation (Medium-Scale)

The purpose of this task was to test the combined effects of damage (notch), surface protection, and corrosion on the fatigue performance of lower flange sections of rail. Fatigue loading was applied with rollers in a four-point bending arrangement. The center rollers were spaced at 8 inches and contacted the top surface adjacent to the radius of the rail. The outside rollers contacted the lower surface of the flange at 16-inch spacing (see Figure 11). Testing was performed at about 7 Hz.

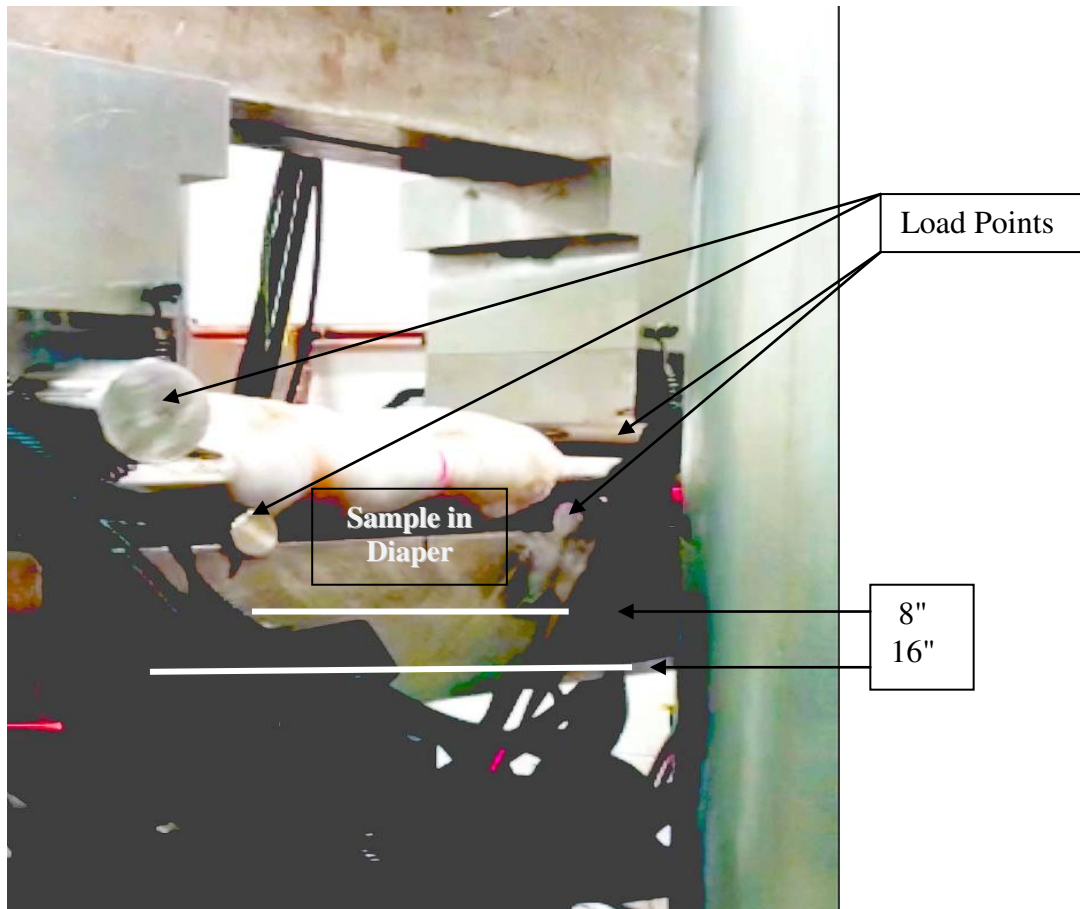


Figure 11. Four-Point Load Fatigue Test Setup

Table 5 lists the sample descriptions, applicable load ranges, and cycles to failure. The differing load ranges were required because of slight differences in sample geometries. When the heads and webs were removed from the rail stock, the position of the cut surface relative to the upturning radius of the flange-to-web junction differed from sample to sample. Some had cut faces leaving more residual web riser and others had been cut more deeply into the flange top surface. To provide the same nominal stress on the *lower* base flange surface during fatigue testing, different load levels were chosen for the top side loading to account for the differences in specimen size.

Corroded specimens were tested in the presence of topical, trapped saltwater (Samples #2-6). A saltwater “diaper” was emplaced to surround the notch region and was held in place with shrink wrap.

Table 5. Four-Point Load Fatigue Testing

Sample	Description	Corrosion Cycles	Load Range for R=0.1 (lbf)	Cycles to Failure	Comments
1	Un-notched, unprotected	N	15,755 - 1576	5,000,000	Run-out
2	Notched, unprotected	N	11,467 - 1147	305, 198	Failed at notch
3	Notched, unprotected, corrosion	Y	13,789 - 1379	61, 839	Failed at notch
4	Notched, 3-step, corrosion	Y	12,422 - 1242	218,514	Failed at notch
5	Notched, 2-step, corrosion	Y	12,422 - 1242	226,479	Failed at notch
6	Notched, EonCoat, corrosion	Y	13,322 - 1332	3,460,278	Stopped test (run-out)

2.4 Task 4 – Resonant Fatigue Testing (Rail Sections)

The purpose of this task was to look at the combined effects on full-scale rail segments and to determine if there was correlation between the medium-scale fatigue results and the fatigue results for longer rail sections. Twenty-foot rail sections of 136-pound rail were used for testing with a fatigue method called *resonant fatigue*.

2.4.1 Resonant Fatigue Methodology

Resonant fatigue methodology has been most commonly applied to testing welds in pipe and drill pipe used in oil exploration. It has been used on symmetrical, round cross sections. In the EWI adaptation, it was used on the asymmetrical cross section of a rail segment.

As applied here, a 20-foot section of rail was suspended between, and strapped to, two support points (Figure 12). Counterweights of calculated size were then attached to the ends. The cyclic excitation at close to the resonant frequency, provided by an eccentric drive cam at one end, forces the rail into self-resonance around the center line, creating what amounts to a standing wave on the rail with maximum stress in bending at the center of the length. The rail becomes an oscillator element.

Strain gauges monitor the cyclic strains at several locations on the part, but especially near the center. At some point, a crack will grow to sufficient size by fatigue to cause the rail to fracture. This is the failure point at which the machine stops and the cycles to failure are noted. In this case, the rail went into self-resonance at approximately 21 Hz. The applied stress level was determined by the difference between the resonant and excitation frequencies. The excitation and self-resonance induced a stress on the rail head of 28–40 ksi and a stress on the rail base of 25–26 ksi, for notched rail, not accounting for the additional stress concentration at the notch.



Figure 12. Rail Suspended in Resonant Fatigue Apparatus

Resonant fatigue of the rail provides reversed loading, represented as $R=-1$, where R is the ratio of minimum stress to maximum stress. Thus, the applied stress range is double the maximum stress of the cycle. The maximum stress is listed in tables and descriptions of this task. There were five test specimens for the resonant fatigue test.

- 1) RS1 – Plain rail segment, no notch or corrosion
- 2) RS2 – Notched, untreated, no corrosion
- 3) RS3 – Notched, untreated, corrosion
- 4) RS4 – Notched, treated with EWI three-step system, corrosion exposure
- 5) RS5 – Notched, treated with EonCoat®, corrosion exposure

2.4.2 Preparation of Rail Sections

Surface preparation methods and corrosion cycling for the 20-foot rail sections were modified from those used for partial rail segments to account for size. Surface rust was ground off to a distance of about 2 feet to either side of the center line on the bottom of the flange only. If notched, the notch was cut across the flange at the 10-foot point using a ceramic saw.

(Unfortunately, this method does not allow for consistent notch dimensioning, which may have become a factor in skewing the results expected from the testing.)

Figure 13 shows a rail segment that has been treated with the EWI ALL three-step process and is wrapped with heater blankets prior to the bake cycle. An insulating layer of ceramic fiber blanket was then placed over the heating area. The rail section outside the insulated heating zone stayed quite cool.

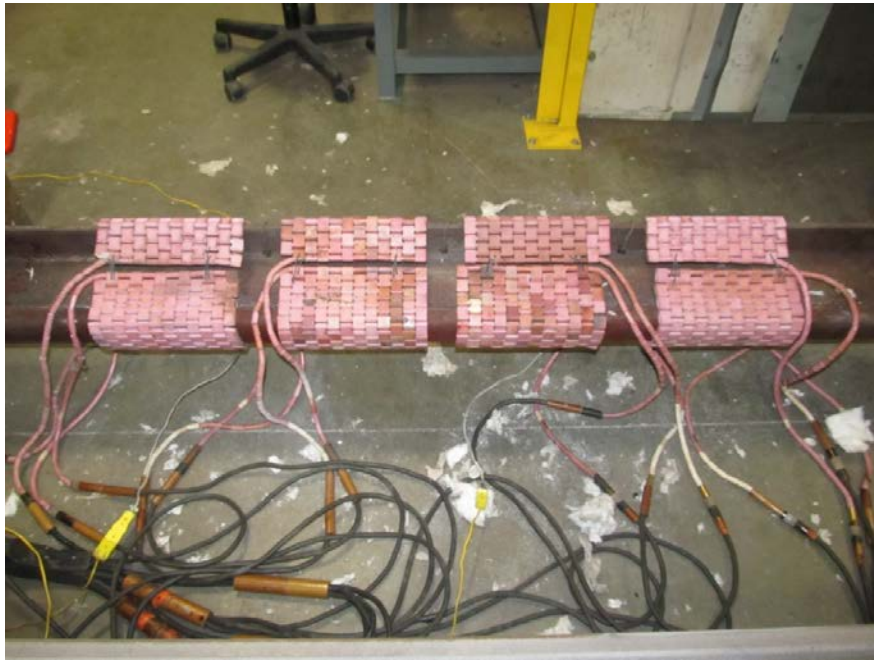


Figure 13. Resistance Heating Blankets used to Heat Cure Zirconia Sol-Gel Sealer

The vendor applied EonCoat® to the rail segment at EWI. Standard two-component spray equipment was used with a mixer-head nozzle (Figure 14). It dried within minutes of application. A second coat was then applied after approximately 10–15 minutes. That rail segment is shown in Figure 15. There was evidence of some debonding of the spray at the edges (circled in the figure). These locations were outside the prepared regions.



Figure 14. Technician Readies EonCoat® Applicator System



Figure 15. EonCoat® Sprayed Rail Segment

2.4.3 Corrosion Exposure Cycling for Large Rail Segments

The corrosion cycle followed that shown in Table 3 above. It was necessary to modify the corrosion cycling to adapt it to the large sample size. Soaking in a tank was impossible, for example, and the humidity exposure method required a change.

For the saltwater exposure, the rails were brushed with saltwater solution. Paper towels soaked in saltwater were then placed over the surface. A lofted fabric mat, also soaked in saltwater, was then overwrapped. That whole assembly was wrapped in place with shrink wrap to retain the solutions in contact with the rail and the notch region. This became the “diaper” to keep the saltwater solution in contact with the rail. For air drying, the diaper was cut off and discarded. To mimic the humidity chamber, the same basic diapering procedure was used, substituting water for salt water, to emulate the required 3-day humidity exposure. The cycle time lengths were the same as before and, overall, the exposure lasted 3 weeks.

No tie plates were used to trap liquid in the flange-to-plate gap during this corrosion cycle exposure.

3. Results and Discussion

3.1 Task 1 – Small-Scale Corrosion Exposure – Results and Discussion

3.1.1 Surface Studies

The purpose of Task 1 was to make a preliminary determination of which EWI two-step or three-step treatment systems might be effective in preventing or retarding surface corrosion on rail steel. The corrosive environment used in this program was highly aggressive and might not represent the effects of slower corrosion attack, as might be found in service. Therefore, the fact that aggressive corrosion exposure resulted in attack was not taken as an indication that the treatments were of no value.

At the end of each exposure week, photographs were taken of the bar samples to record their performance. All the samples exposed to corrosion showed rusting. This was not surprising given the severity of the exposure, especially the hot-wet portion, which is very aggressive when combined with a saline soak and interim drying.

Particular attention was paid to the condition of the bottom surface of the samples. This would be the area of most concern in a rail tunnel since it would not easily dry. Figure 16 and Figure 17 show the views of the bottoms of the samples after 2-week and 3-week exposure, respectively. Figure 18 and Figure 19 show the same samples, but for the top surfaces.

The samples were rated visually based on overall rust amount and severity, including blistering or flaking. The “PD” series and the “PDZ” series had replicates, so only one rating is given for each of those. The rating is from best to worst (1 to 4), as shown in Table 6.

One of the more striking observations was the degree of overall corrosion attack after 3 weeks compared with 2 weeks. The corrosion became quite aggressive during the third week. The results show that the ALL variant, which had the three-step application system with final bake, performed best in protecting the bottoms. However, the best performance for protecting the tops was the PD variant, which is a two-step process with no bake.



Figure 16. Bottoms of Samples after 2-Week Corrosion Cycle



Figure 17. Bottoms of Samples after 3-Week Corrosion Cycle

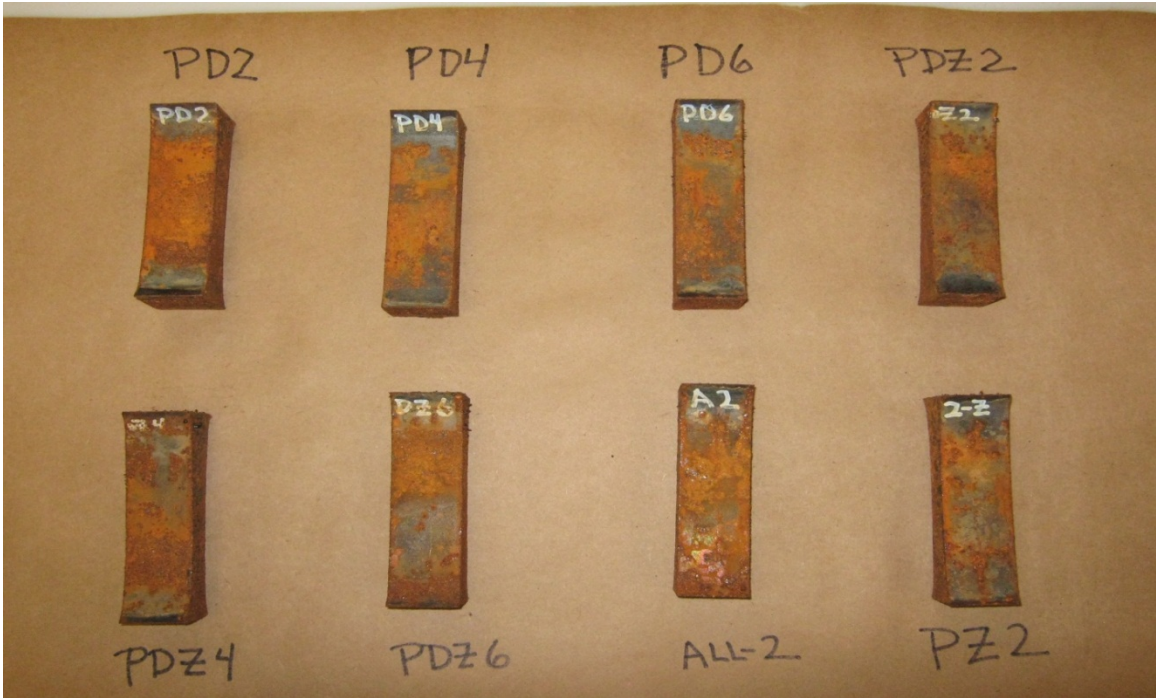


Figure 18. Tops of Samples after 2-Week Corrosion Cycles

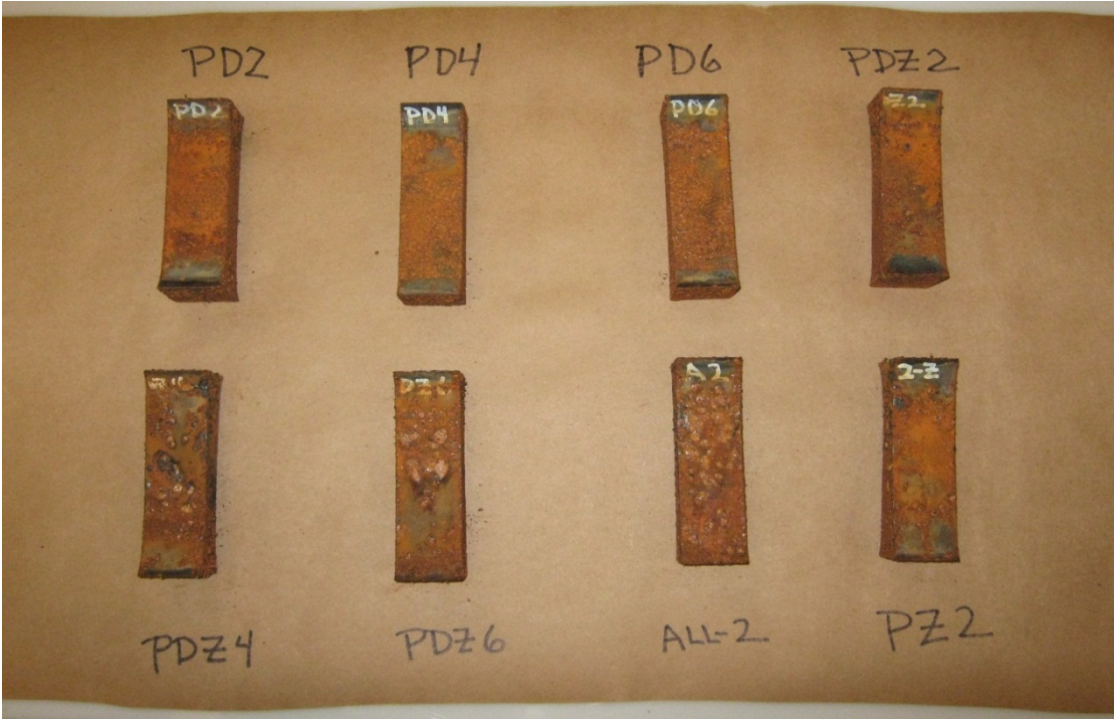


Figure 19. Tops of Samples after 3-Week Corrosion Cycle

Table 6. Visual Interpretation of Corrosion Results

Sample Preparation						Tops						Bottoms		
						Ranking - after Salt Water			Ranking - after Hot/Wet			Ranking		
Corrosion	Phos	Dopa	Zrc	Bake	Total	Week 1	Week 2	Week 3	Week 1	Week 2	Week 3	Week 1	Week 2	Week 3
PD2	X	X			6	1	1	1	1	2	1	NR	3	2
PD4	X	X				3	3	2	3	3	2	NR	2	3
PD6	X	X												
PZ2	X		X	X	2									
Corrosion														
PDZ2	X	X	X		6	2	2	3	2	1	3	NR	4	4
PDZ4	X	X	X			4	4	4	4	4	4	NR	1	1
PDZ6	X	X	X											
ALL2	X	X	X	X	2									

Overall, the diphenol treatment (D) was more effective than diphenol (D) and zirconia (Z) combined (PD versus ALL2). The Z treatment in the absence of D was not as good when baked (PZ) compared with ALL. Therefore, some initial conversion treatment using D is important.

The specimens exposed to corrosion cycles (even numbered) were compared with their unexposed, treated counterparts (odd numbered) from the same sets. Three treated sets were chosen for the SEM/EDS analyses on unexposed and exposed samples.

- 1) PD series, which gave the best results on top surface corrosion resistance
- 2) ALL series, which gave the best results on bottom surface corrosion
- 3) PDZ series, which had the worst performance of all the combinations tried

3.1.2 Results from SEM/EDS Analyses

Samples were cut to provide test articles for scanning electron microscopy (SEM) and elemental x-ray dispersive (EDS) analyses. These were pieces taken from the center portions, as shown below (Figure 20 through Figure 23). The top sides were marked for orientation in the SEM instrument. The samples were analyzed using SEM to examine morphology. Integrated EDS capability was used to examine the coatings for chemical composition. The samples were examined both as cross cuts through the coatings (“sideways”) and also on the tops themselves.



Figure 20. Photograph Summary of Samples for SEM/EDS Examination

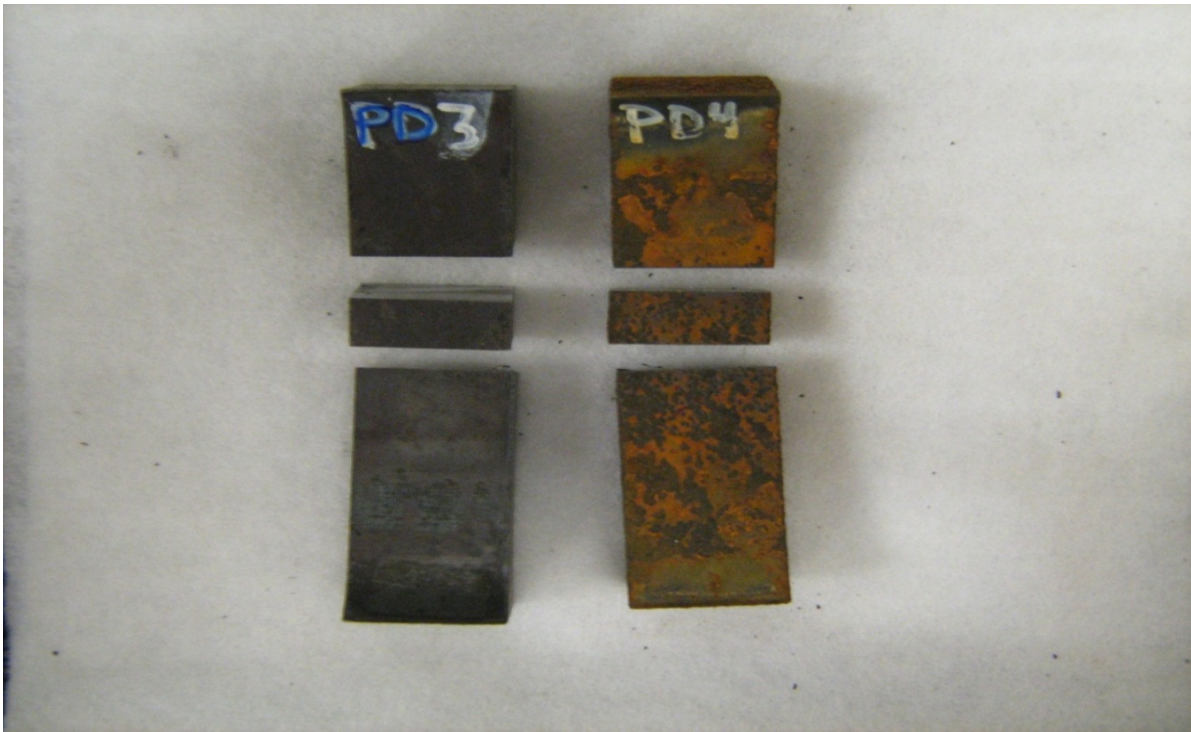


Figure 21. Close-Up of PD3/4 Series

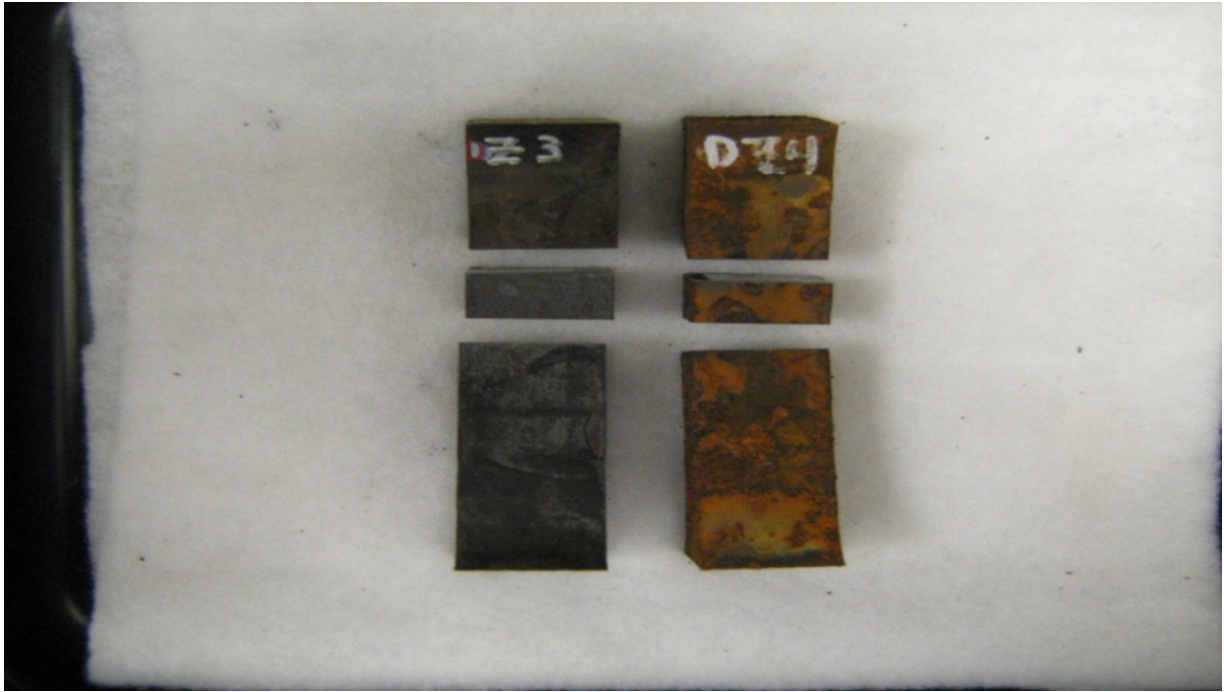


Figure 22. Close-Up of PDZ3/4 Series



Figure 23. Close-Up of ALL1/ALL2 Series

3.1.3 Crosscut Samples

For the following discussion, recall that odd-numbered sample types were treated, but not exposed to corrosion. Even-numbered sample types were treated and exposed to corrosion. Representative SEM photographs are shown for PD3 and PD4 (Figure 24), PDZ3 and PDZ4 (Figure 25), and ALL1 and ALL2 (Figure 26). These were taken at 1000× to accentuate the interfacial zones and the coatings themselves. The areas marked “Spectrum” show the scan locations for the EDS analysis.

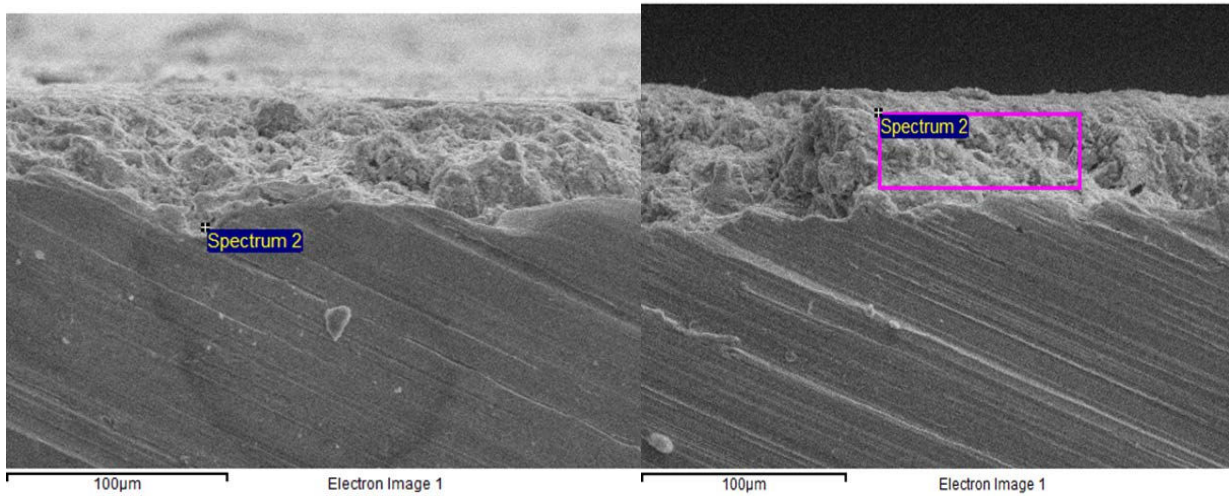


Figure 24. SEM Photographs of Cross Cuts for PD3 (Left) and PD4 (Right)

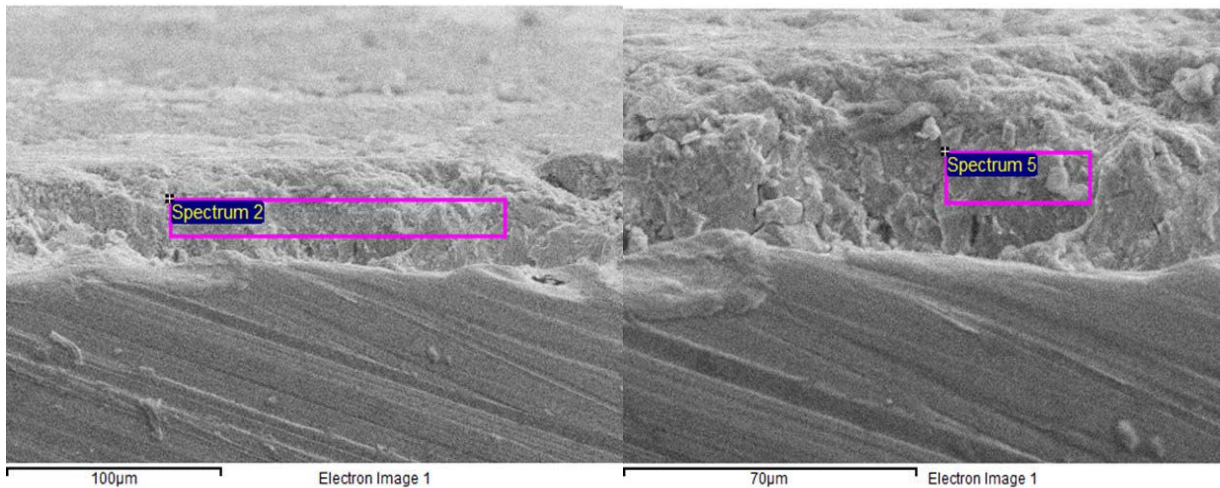


Figure 25. SEM Photographs of Cross Cuts for PDZ3 (Left) and PDZ4 (Right)

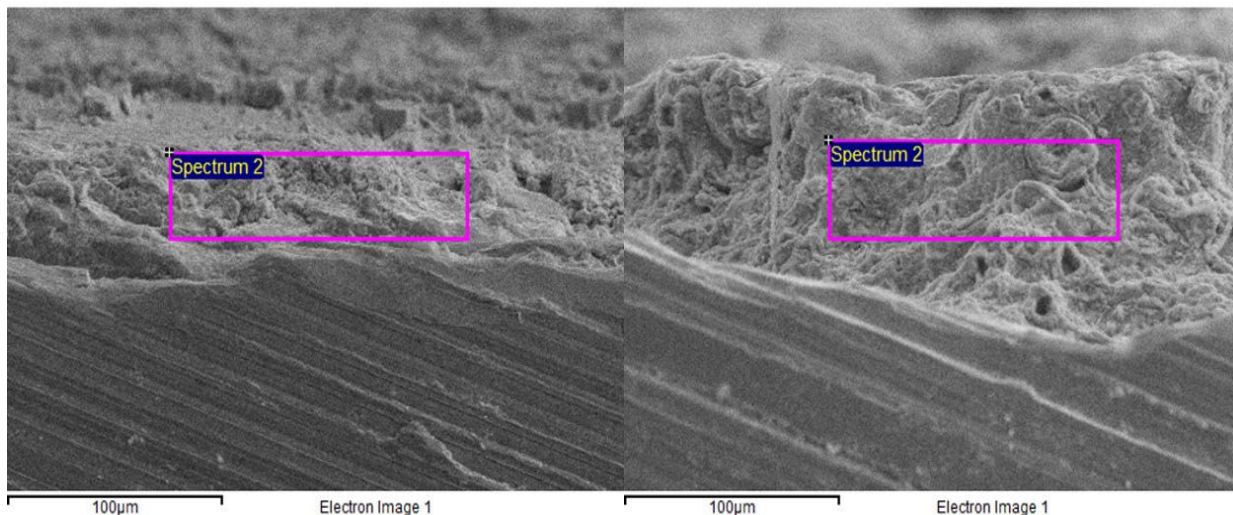


Figure 26. SEM Photographs of Cross Cuts for ALL1 (Left) and ALL2 (Right)

All samples showed some porosity in the coatings. The EDS elemental analyses taken from cross cuts of the samples are provided in Table 7. There is significant iron content indicative of the ferrous/ferric material in the coatings and surface oxides. The predominance of iron (and also aluminum and silicon from the steel) is reflected in the low weight-percent of the other elements. The iron in these samples was 90–95 weight-percent of the total, meaning the other peaks were dwarfed.

The main interest was in finding evidence that the treatments used had changed the surface chemistry predictably, since the presence of phosphorous and zirconium would be expected. Other elements of potential interest were found in some cases, so they are reported below.

Table 7. Summary of EDS Analyses for Cross Cut Sample Coatings

Sample Preparation							EDS Analysis - Cross Section (Wt-%)			
	Sample [Thickness (µ)]	Corrosion	Phos	Dopa	Zirc	Bake	P	Zr	Ca	Mn
2-step	PD3 (44)	N	X	X						0.88
	PD4 (45)	Y	X	X						0.91
3-step	PDZ3 (41)	N	X	X	X			5.34		0.94
	PDZ4 (62)	Y	X	X	X		0.1	0.58	1.13	0.87
	ALL1 (48)	N	X	X	X	X	0.1	0.84		
	ALL2 (82)	Y	X	X	X	X	0.1	0.39		0.78

(P = Phosphorous; Zr = Zirconium; Ca = Calcium, Mn = Manganese)

The source of manganese is perplexing. It may have come from the bulk of the steel and been lifted into the coating during the phosphoric acid etch. Phosphorous and zirconium came from the treatments and were expected. The high level of zirconium in PDZ3 seemed anomalous given the

levels found elsewhere. The calcium was likely from the road deicing salt used in the saltwater exposure.

It is possible the calcium that appeared in PDZ4 was replacing the zirconium in the coating through ion exchange, resulting in depletion of the zirconium. The calcium was found mostly in nodules that appeared near and on the coating surface. Since PDZ3 and PDZ4 were not baked, the coatings could change in composition if chemical substitutions of other ions were possible. The ALL1 and ALL2 series were baked at 550°C, and the findings of P and Zr were not out of keeping with each other. No calcium appeared in the interior of that coating, suggesting possibly that ion exchange was discouraged by a more densified zirconium oxide coating formation.

Analyses of the cross cut samples indicate the treatments were effective in converting the surfaces, as hoped. However, ion exchange of calcium for zirconium appears to have taken place, rendering the protection of the zirconia seal coat ineffective. Calcium oxides are not particularly resistant to water attack or preventing chloride from reaching the iron surface.

3.1.4 Top Surface Analyses – Topology

The top surfaces were also examined using SEM. Representative SEM photographs of those samples are shown for PD3 and PD4 (Figure 27), PDZ3 and PDZ4 (Figure 28), and ALL1 and ALL2 (Figure 29). These were taken at 200× to give a larger view of the surfaces.

In general, the physical topologies were unremarkable. Cracking found in PDZ3 might be from the zirconia sol-gel. Sol-gels are susceptible to mud-cracking if dried or condensed too rapidly. Localized thickness variations leading to drying stress can also cause sol-gels to mud-crack as they dry and react. One might expect the same topology for PDZ4 as for PDZ3, since it was not baked, but that was not evident in the sample examined. As shown in Table 8, the amount of surface zirconium dropped in PDZ4 after the corrosive exposure. The mud-cracked zones could be expected to be more sensitive to “etching” so they may have been attacked preferentially during corrosion.

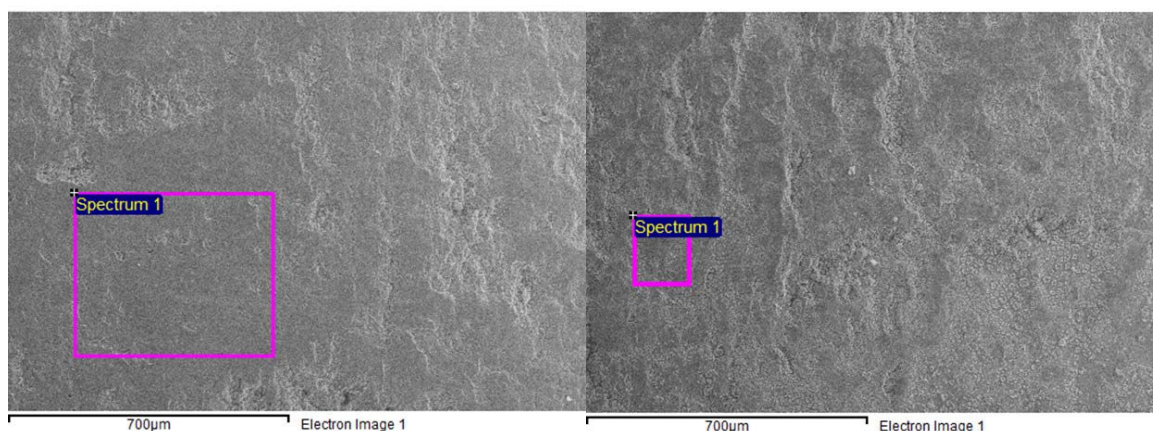


Figure 27. SEM Photographs of Top Surfaces for PD3 (Left) and PD4 (Right)

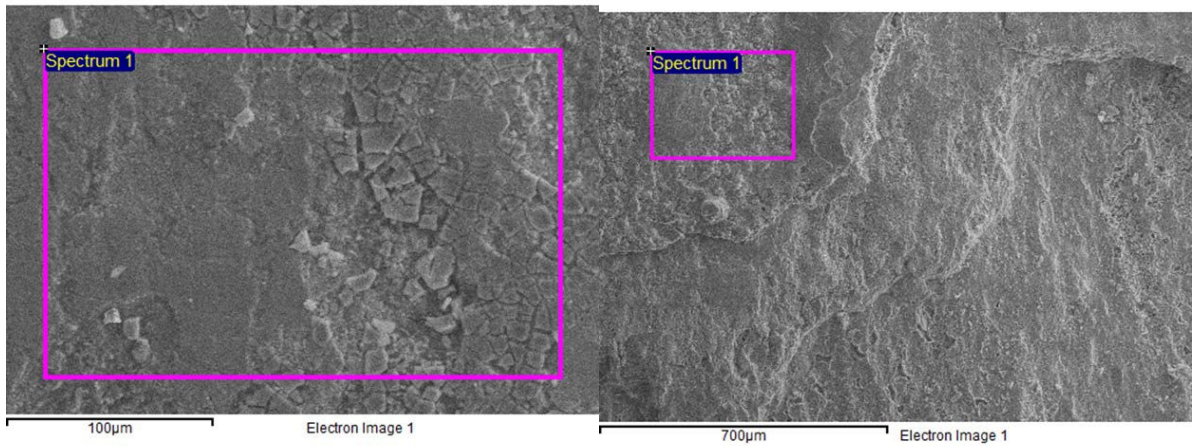


Figure 28. SEM Photographs of Top Surfaces for PDZ3 (Left) and PDZ4 (Right)

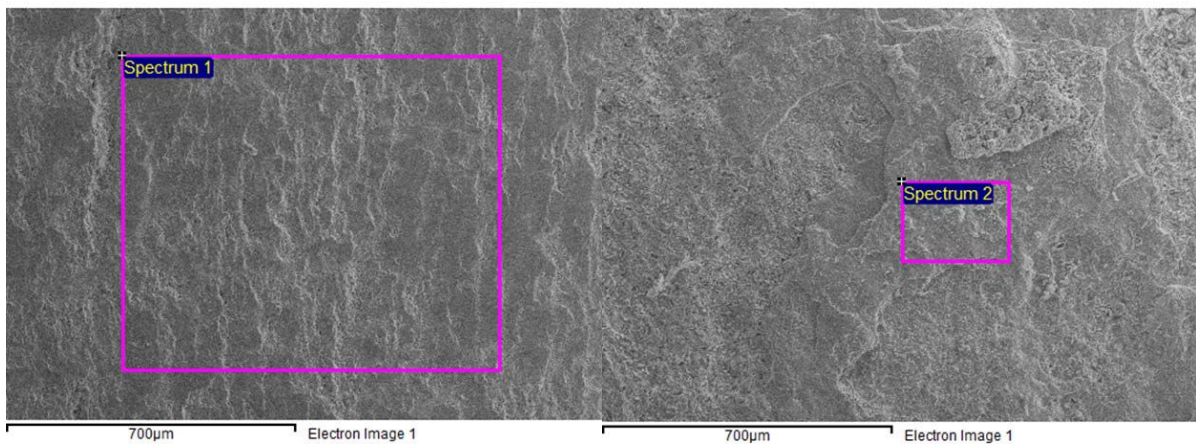


Figure 29. SEM Photographs of Top Surfaces for ALL1 (Left) and ALL2 (Right)

3.1.5 Top Surface Analyses – Elemental Content

The surface chemistry of the coatings is important because that is the area affected by corrosive attack. The EDS elemental analyses for the top surfaces are summarized in Table 8.

Table 8. Summary of EDS Analyses for Sample Coating Surfaces

Sample Preparation							EDS Analysis - Top (Wt-%)			
	Sample	Corrosion	Phos	Dopa	Zirc	Bake	P	Zr	Ca	Mn
2-step	PD3	N	X	X			1.13		[0.55]	
	PD4	Y	X	X			1.52	[3.89]		
3-step	PDZ3	N	X	X	X		1.58	1.37		
	PDZ4	Y	X	X	X		0.42	0.42		
	ALL1	N	X	X	X	X	0.44	1.05		
	ALL2	Y	X	X	X	X			1.28	1.41

(P = Phosphorous; Zr = Zirconium; Ca = Calcium, Mn = Manganese)

There was a seemingly large peak for zirconium in PD4 which was unexpected since the PD-series was not treated with zirconia sol-gel. It is possible the zirconium leached from the PDZ3 and PDZ4 sample bars, which were unbaked. The unbaked sol-gel could have allowed leaching of Zr into the soak baths during the early soaking phases, possibly the first cycle of saltwater soak. The presence of calcium in PD3 was also unexpected since it was not exposed to deicing salt. The presence of calcium found in the top surface of ALL2 does not correlate with the analysis of ALL2 in the cross sections where none was found. Likely, it is topical from exposure to the saltwater and found only on the surface, again possibly due to ion exchange with zirconium, which was not found on the surface, but was found internally.

3.1.6 Implications for Protecting Rail Steel

In the previous EWI work,⁽⁶⁾ the ALL system was used. It was very robust in ion-free water and survived 300°C water in an autoclave, exposed to approximately 1250 psi internal pressure. No rust formed. In this work, the availability of ionic species from the saltwater clearly had a different effect on the corrosion protection of the system. The significant finding of these analyses is that the topical treatments for the steel were effective in changing the surface chemistry, which was the desired intent. However, the combined protection of the ALL system and variants, while a partial hindrance to corrosion, did not protect against it. The overall effectiveness in protecting against corrosion varied, depending on the treatment.

Based on these findings, it was decided to proceed with the testing of some of the variants for coating larger samples. These were the PD two-step process and the ALL three-step process. The PD system was chosen because of its simplicity, requiring only two treatment materials and not requiring a bake. The ALL system was chosen because it represented the best EWI approach, which had borne out well in the previous studies involving water only.

3.2 Task 2 – Treatments on Rail Stock (Medium-Scale)

The sequential progress of corrosion is shown pictorially in Appendix A. A synopsis follows. At the end of the 3-week corrosion exposure, the PD, ALL, and untreated samples were corroded in varying degrees while the EonCoat® system showed virtually no corrosion (Figure 30 through Figure 33). The bottoms of the flanges and the tops of the tie plates are shown.



Figure 30. PD Treated Samples after 3-Week Exposure (Tie Plate and Flange Bottom)



Figure 31. ALL Samples after 3-Week Exposure



Figure 32. Untreated Samples after 3-Week Exposure



Figure 33. EonCoat® Samples after 3-Week Exposure

Based on visual examination, the EonCoat®-treated sample showed considerable resistance to the combined corrosion cycles. The rust spots on the EonCoat® surface were caused by condensate from the specimen on the shelf above it in the humidity cabinet. A beneficial observation was that areas of the EonCoat® that had been chipped or physically damaged in handling still did *not* succumb to corrosion attack.

In general, the time spent in humidity was very aggressive and most likely to produce the most sequential damage. Of interest concerning the samples treated with the ALL conversion system was the considerable protection between the flange bottom and the tie plate nested surface even in the presence of aggressive corrosion attack (Figure 31). The outer exposed portions of the samples were severely corroded.

The notches cut into flange bases before treatment and corrosion exposure showed varying degrees of rusting within the defect. The untreated samples were the most attacked, being almost rusted shut, while the sample with EonCoat® system was unaffected. The samples treated with the PD system and ALL system showed about the same level of corrosion within the defect zones, based on visual examination at 4×. There was little bridging rust in the latter samples where the rusting had progressed to where it was closing the gap for the untreated flange.

3.2.1 Drip Zone Samples

The results after 3 weeks for the top and bottom of the drip zone sample are shown in Figure 34 and Figure 35, respectively. In both cases, the protected portions were on the bottom half of the photographs. The protection on the bottom half of the top surface was fairly good. The protection for the trapped portion on the flange bottom was fair, but noticeable. There was possibly some benefit from the ALL treatment, and it might prolong rail life in actual service, but the protection was not dramatic.



Figure 34. Drip Zone Top Surface after 3 Weeks



Figure 35. Drip Zone Bottom Surface after 3 Weeks

3.2.2 Salt Fog Testing

Additional plate specimens had been prepared prior to this work using the EWI ALL processing as a primer on 1018 steel and a subsequent overcoating of PPG® PSX-700 epoxy-silicate paint. Samples coated with EonCoat® were also included to rank its performance against the latter. A total of six sample plates were exposed to ASTM B117 salt fog testing. The salt fog testing and specimen photographs are detailed in Appendix B.

The results indicate that the EWI three-step (ALL) process may be useful as a primer underneath a barrier-type coating; but, the EonCoat® system itself provided better overall corrosion protection.

3.3 Task 3 – Crack Fatigue Propagation and Mitigation (Medium-Scale)

The samples prepared and exposed in Task 2 were subjected to four-point load fatigue testing. The results for the four-point load fatigue testing are given in Table 9.

With the first three samples, a flange segment having no damage or corrosion (#1) did not fail after 5,000,000 cycles. Testing was stopped and this was considered run-out. Sample #2 was notched on the base (damaged), but had no corrosion cycles and failed at ~300,000 cycles. Thus, the damage itself reduced fatigue life by approximately one order of magnitude. The added effect of corrosion (#3) dropped the result almost another order of magnitude to ~60,000 cycles. This suggests the combined effects of damage, corrosion, and fatigue have a severe impact on rail fatigue life.

Table 9. Fatigue on Medium-Scale Samples

Sample	Description	Corrosion Cycles	Load Range for R=0.1 (lbf)	Cycles to Failure	Comments
1	Un-notched, unprotected	N	15,755 - 1576	5,000,000	Run-out
2	Notched, unprotected	N	11,467 - 1147	305, 198	Failed at notch
3	Notched, unprotected, corrosion	Y	13,789 - 1379	61, 839	Failed at notch
4	Notched, 3-step, corrosion	Y	12,422 - 1242	218,514	Failed at notch
5	Notched, 2-step, corrosion	Y	12,422 - 1242	226,479	Failed at notch
6	Notched, EonCoat, corrosion	Y	13,322 - 1332	3,460,278	Stopped test (run-out)

For the corrosion-protected samples, the ALL (three-step) and DP (two-step) treatment processes both showed the ability to offer some benefit in fatigue life from protection in severe corrosive environments. Samples #4 and #5 showed a reduction in fatigue life of approximately 30 percent compared with Sample #2, but a significant improvement in fatigue life over Sample #3 (no protection). Of the three protected samples, the EonCoat® system (#6), effectively gave run-out under fatigue, meaning it showed very good ability to prevent corrosive damage and allow a longer fatigue life.

In comparing Sample #6 with Sample #2, the question may arise as to why Sample #6 lasted so much longer than Sample #2, which had no corrosion protection. As mentioned, the test samples were fatigued in the presence of saltwater. Visual examination showed the presence of rust formation in the crack zone of Sample #2. It is postulated that the unprotected Sample #2 actually experienced an unintended corrosive attack during the test, resulting in reduced fatigue life.

All the corrosion protection methods showed some benefit in extending fatigue performance on notched samples exposed to corrosion. The largest improvement came from the EonCoat®, while the other two treatment methods improved fatigue life, but not as dramatically. Both those treatments showed similar performance and life extension.

The fracture surfaces for Sample #2 (NONE) and Sample #4 (ALL) are shown in Figure 36 and Figure 37, respectively. Notice the advance of corrosion in the damage zone is greater for the sample with no protection than for the sample with corrosion protection.



Figure 36. Fracture Surface of Corroded Sample with No Protection



Figure 37. Fracture Surface of Corroded Sample with ALL Treatment

The results of the four-point load fatigue tests were in keeping with expectations. For this work, only one sample of each type was tested. A traditional S/N curve was not developed. The goal was to look for trends. Future work with more focus on sample size and statistical performance could be appropriate for a subsequent phase. From this work, it appears the influence of damage on fatigue life is to reduce it about one order of magnitude. Added corrosion can reduce fatigue life by roughly an additional order of magnitude. In other words, pernicious corrosion combined with damage and fatigue can significantly reduce rail life.

3.4 Task 4 – Resonant Fatigue Testing (Rail Sections)

The resonant fatigue test can be compared with three-point bend and four-point bend fatigue tests, as well as with rolling load tests, since all these tests can be performed on scales similar to those used in this work. Fatigue tests can be performed on samples ranging from small coupons to a test of rail in place, as at the Facility for Accelerated Service Testing (FAST) loop. Because of its high cycle rate, resonant fatigue testing has the potential to perform the testing more quickly than similar scale tests and places peak bending stresses at both the top and bottom surfaces of the rail instead of only at one or the other. Resonant fatigue uses a longer sample of rail than similar scale tests, but also stresses a longer area with a nearly uniform stress distribution. Like three- and four-point bend tests, resonant fatigue does not allow for the contact of the rail wheel with the rail head. It is potentially useful for checking behavior where contact forces are not an issue. If contact forces are an issue, then a rolling load test is more appropriate.

The results of the resonant fatigue testing are summarized in Table 10 and Figure 38.

Table 10. Summary of Resonant Fatigue Testing

Sample	Description	Corrosion Cycles	Rail Head Stress (ksi)	Rail Base Stress (ksi)	Cycles to Failure	Comments
RS1	Un-notched, unprotected	N	39.6	32.9	840,613	No failure
RS2	Notched, unprotected	N	28.6	25	109,888	Fracture from fatigue crack at notch
RS3	Notched, unprotected, corrosion	Y	30	26.4	133,291	Fracture from fatigue crack at notch
RS4	Notched, 3-step, corrosion	Y	29.3	26.3	967,968	No failure, but rusted
RS5	Notched, EonCoat, corrosion	Y	29.8	25.7	94,745	Fracture from fatigue crack at notch

Resonant Fatigue Testing of Full Rail

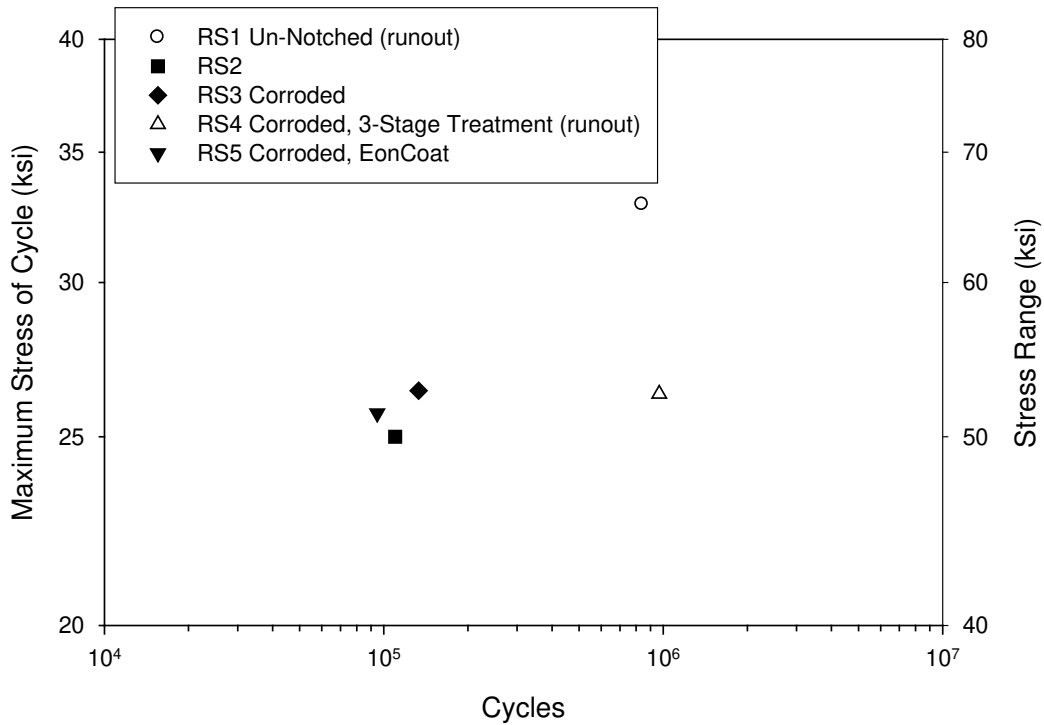


Figure 38. Resonant Fatigue Test Lifetime Results

One of the more significant findings of this test was that asymmetrical cross sections, such as rail, could be induced to self-resonate by this method. Previously, only circular cross sections (i.e., pipe and girth welds) had been tested with resonant fatigue, and the ability to put rail segments into oscillation was noteworthy.

Fatigue results are judged partly based on the range of stress loading. Since the cycles provide reversed loading, the stress ranges will be twice the values given in Table 10, or roughly 60–80 ksi. Sample RS1 was tested without a notch and at a higher stress range than the notched rails. Failure of that sample was not induced in the testing. Samples RS2–RS5 were pre-notched before treatments, corrosion cycles, or testing. RS2 was not protected from corrosion, but it also did not see purposeful corrosion cycles. Sample RS3 was unprotected, but *did* undergo corrosion cycling and had a saltwater solution “diaper” applied during resonant fatigue. It could represent older rail in service that had somehow been damaged and suffered corrosion attack. It failed in the crack zone, but showed slightly higher fatigue life despite the corrosion and lack of protection. Rust formation is clearly evident in the failure zone of RS3 (Figure 39).



**Figure 39. Resonant Fatigue Fracture Surface of Unprotected, Corroded Rail (Sample RS3)
Arrow Shows Crack Zone**

The EonCoat®-treated sample (RS5) showed approximately the same number of cycles to failure as the notched untreated sample that had not been corroded (RS2) and the notched untreated sample that had been corroded (RS3). This comparison suggests corrosion has *no* effect on rail fatigue life, which is in conflict with the four-point load results found in Task 3. The differences in fatigue life found for the notched samples RS2, RS3, and RS5 are not statistically significant. This suggests that damage, corrosion, and corrosion protection are *not* interconnected in fatigue-related phenomena, a conclusion which sharply contrasts with the fatigue results found for the four-point load testing used on the medium-scale samples. These samples also do not correlate intuitively with anticipated fatigue life behavior.

Because sample RS4 did not fail within expected test duration, that test run was suspended. This was also surprising and unexpected. Sample RS4 was notched and had the EWI three-step corrosion protection applied prior to being subjected to corrosion cycles. It also had the saltwater diaper applied during resonant fatigue. It did not fail during testing, reaching nearly one million cycles without failure, which conflicted with expectations and did not mirror the results found with the four-point load testing. The notch in RS4 showed minimal corrosion, with the least amount of corrosion in the center of the flange width and more at the tips of the flange.

In comparing the character of the fracture surfaces with those for the medium-scale samples, the resonant fatigue samples showed much smaller regions of fatigue cracking than those visible on the medium-scale samples (Figure 40 and Figure 41, tested in Task 3). This difference is attributed to the larger cross-section of the full rail inducing a higher stress intensity factor in the rail at the fatigue crack tip, allowing brittle fracture to take over from fatigue at a smaller crack size.



Figure 40. Fracture Surface of Corroded Sample with No Protection (Crack – corrosion area is circled)



Figure 41. Fracture Surface of Corroded Sample with ALL Treatment

It is disappointing that resonant fatigue testing did not provide predictable correlation. It was hoped this technique might be useful as a general, and fairly inexpensive, screening tool for examining rail fatigue behavior prior to submitting to more time-consuming field trials. It was not meant to replace those trials, but to enable reducing the number of samples needed in trial by first culling those destined to give poor results. This was not the case; therefore, the testing was of no predictive value.

If the reliability of the technique is governed by notch dimension and quality, a more thorough study would be needed to determine the notch dimensions and even where it should be placed. The technique may ultimately be of value. Further testing using statistically significant numbers of samples would be useful in making a final determination about the potential for this technique.

4. Conclusions and Recommendations

4.1 Conclusions

An organometallic conversion type coating system, investigated at EWI for use in high pressure liquid water systems, was useful in retarding the effects of corrosion on fatigue rail life. However, it did not prevent corrosion in the presence of saltwater or condensing humidity. A commercially available inorganic conversion coating (EonCoat®) was effective in preventing corrosion in saltwater environments. It enabled extended fatigue life, even on damaged rail subjected to aggressive corrosive environments, and gave performance similar to that for undamaged, uncorroded rail.

In this work, it was found that the interactions between rail damage, corrosive attack, and overall rail fatigue life can be examined by methodical application of traditional four-point fatigue testing using manageable sample sizes. The influences of corrosion and damage in the presence of fatigue stress are believed to be separable based on this limited testing. The presence of damage alone reduces rail fatigue life by as much as one order of magnitude. Added corrosion attack reduced fatigue life by as much as an additional order of magnitude. The combined effects on rail life of damage, corrosion, and fatigue can be severe. Use of corrosion protection systems, applied to the base of the rail flange, can extend rail fatigue life.

A resonant fatigue test method was adapted for use with 20-foot long rail segments. It was shown that asymmetrical and heavy cross sections could, in fact, be induced into self-oscillation, developing approximately 60–80 ksi peak stress. In this work, the method was not found to be predictive regarding fatigue life for damaged, corroded rail, with or without corrosion protection. However, rail was taken to failure by this method, suggesting a more advanced test methodology might be useful for screening rail fatigue phenomena.

4.2 Recommendations for Future Work

This program was initiated in response to the specific issue of track damage and derailment resulting from corrosive underside attack to the rail. The intent of the program has always been to proceed to on-track evaluation of the potential anti-corrosion system. Therefore, a monitored field trial of the EonCoat® system is highly recommended. A substantive test should involve five to seven select trial installations representing different levels of potential corrosion attack, based on track location and traffic load.

Further, the four-point fatigue data gathered, while indicative of the potential for success, is minimal. Now that a preliminary recommendation has been achieved, continued four-point load fatigue testing involving a more statistically significant sample population should be undertaken, focusing on the EonCoat® system as the protective material. This testing would include continued use of corrosion exposure. Further attention must be paid to the dimensions and machining accuracy of the notches. Essentially, this would replicate Task 2 and Task 3 with a larger population of samples, arriving at an actual stress range versus number of cycles (S/N) plot for each condition.

Finally, it is still believed that the resonant fatigue testing method has the potential to be a cost-effective test tool. First, the methodology should be improved to be consistently predictive of fatigue behavior within a given sample set featuring different kinds of abuse. Particular attention should be paid to notch dimensions and placement. Once the methodology is internally self-consistent, a method should be found to correlate those results with those found in the field; for example, placing rail having similar abusive history on fast track, which would enable the resonant fatigue method to become truly predictive.

5. References

- (1) Robles-Hernandez, F.C.; Koch, Kevin; Plascencia-Barrera, Gabriel. Rail Base Corrosion Detection and Prevention. *TCRP Web Only Document 37*, March 2007.
- (2) Robles-Hernandez, F.C., Plascencia, G., Koch, K. “Rail Base Corrosion Problem for North American Transit Systems,” *Engineering Failure Analysis* 16(1), 2009, p. 281–294.
- (3) Robles-Hernandez, F.C., Koch, K. “Rail-base Corrosion Analysis: A Major Factor that Shortens Service Life of Rail in North American Transit,” *TMS Annual Meeting*, p. 39–54, 2007.
- (4) Akhtar, Muhammad. Guidelines for Rail Base Inspection and Rail Condemnation Limits for Corrosion Induced Material Loss. *TCRP Web Only Document 47*, March 2009.
- (5) Panda, Bijayani; Balasubramaniam, R.; Dwivedi, Gopal. On the Corrosion Behavior of Novel High Carbon Rail Steels in Simulated Cyclic Wet-Dry Salt Fog Conditions. *Corrosion Science*, 50(6), 1684–1692, June 2008.
- (6) EWI Research report for Project No. 52684GTH.
- (7) EonCoat LLC, 4000 Airport Drive, Wilson, NC, 27896.
- (8) H. Li, K. Liang, L. Mei and S. Gu, “Oxidation Resistance of Mild Steel by Zirconia Sol-Gel Coatings,” *Mater. Sci. and Eng. A*, 341, 2003, p. 87–90.

Appendix A. Pictures of Corrosion Samples from Task 2



Fig. A-1. Week 1 – PD after Salt Water



Fig. A-2. Week 1 – PD after Humidity



Fig. A-3. Week 2 – PD after Salt Water



Fig. A-4. Week 2 – PD after Humidity



Fig. A-5. Week 3 – PD after Salt Water



Fig. A-6. Week 3 – PD after Humidity

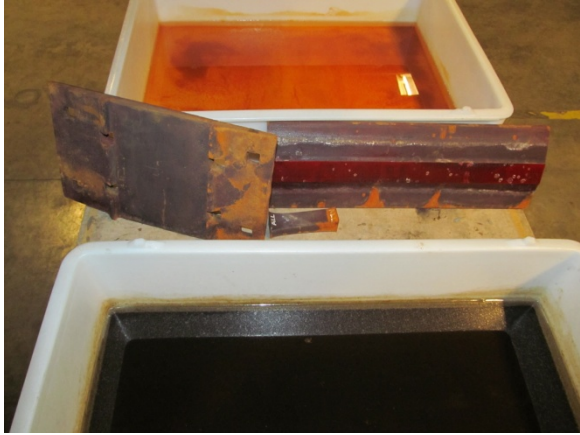


Fig. A-7. Week 1 – ALL after Salt Water



Fig. A-8. Week 1 – ALL after Humidity



Fig. A-9. Week 2 – ALL after Salt Water



Fig. A-10. Week 2 – ALL after Humidity



Fig. A-11. Week 3 – ALL after Salt Water



Fig. A-12. Week 3 – ALL after Humidity



Fig. A-13. Week 1 – EC after Salt Water



Fig. A-14. Week 1 – EC after Humidity



Fig. A-15. Week 2 – EC after Salt Water



Fig. A-16. Week 2 – EC after Humidity



Fig. A-17. Week 3 – EC after Salt Water



Fig. A-18. Week 3 – EC after Humidity



Fig. A-19 Week 1 – No Treat, Salt Water



Fig. A-20. Week 1 – No Treat, Humidity



Fig. A-21 Week 2 – No Treat, Salt Water



Fig. A-22. Week 2 – No Treat, Humidity



Fig. A-23. Week 3 – No Treat, Salt Water



Fig. A-24. Week 3 – No Treat, Humidity



Figure A-25. ALL after Cleaning

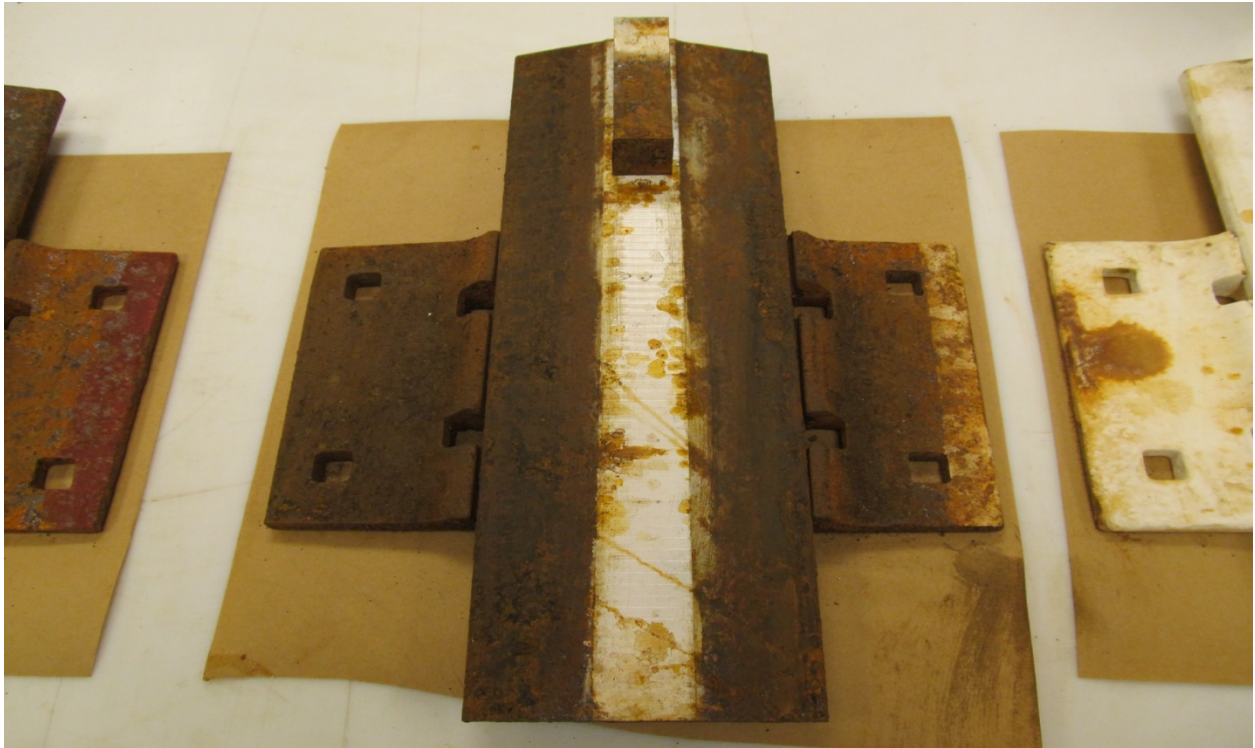


Figure A-26. PD after Cleaning



Figure A-27. EonCoat® after Cleaning



Figure A-28. No Treatment, after Cleaning

Appendix B. Salt Fog Testing

Salt fog testing (ASTM B117) is a highly aggressive procedure for examining the relative corrosion resistance of treatments on metals. Previously, EWI had prepared four 4x12-inch sample plates of 1018 steel, treated with the EWI ALL three-step process and coated with PPG® PSX-700 epoxy-silicate paint. This is a high-build material which meets U.S. Navy specification for use inside ballast tanks. Two additional plates were treated with EonCoat® only by the vendor.

This testing can be performed in two ways. All the plates are first completely coated, including the edges. The first test consists simply of placing them in the test chamber for the requested number of exposure hours. The second method is to purposely scribe through the coating to get to bare metal. In the first case, the absolute barrier protection of the coating can be visually examined. In the second case, the amount of developed rust and its undercutting of the metal underneath the coating are examined. The second method enables a ranking based on how well the coating protects the surrounding area from creeping damage once initial damage has taken place. For this testing, both methods were used. One face was left coated and the other was scribed. The same plates therefore allowed for a total of twelve exposure types: eight for the EWI system and four for the EonCoat®.

The purpose for including this testing was to examine the absolute corrosion protection ability of the two systems, regardless of whether or not they were applied to rail. The specified time increments were 500 hours, 1,500 hours, 3,000 hours, and 5,000 hours for the EWI system with PPG® PSX-700. The EonCoat® samples were exposed for 500 and 5,000 hours. A pictorial summary follows.

After 500 hours, the EWI sample showed attack on the scribed side (Figure B-1), while the EonCoat® system showed very little attack. After 5,000-hour exposure (Figure B-2) the EWI system sustained significant damage with undercutting and blistering, whereas the EonCoat® system was still holding up very well and offering good protection.



Figure B-1. EWI (left) and EonCoat® (right) after 500 Hours, Scribed to Metal.



Figure B-2. EWI (left) and EonCoat® (right) after 5000 Hours, Scribed to Metal

After 5,000 hours, the back sides (Figures B-3) were showing delamination for the EWI system, but there was still residual protection on the exposed metal. This suggests the EWI system as a primer can continue to provide protection even if the top coat is dislodged, as long as the primer is intact (not scribed or damaged).

The progression of front, scribed side attack on the EWI system is shown in Figure B-4. Once the primer is destroyed, it does not function effectively. The back side for EonCoat® shown at 500 hours and 5,000 hours in Figure B-5 shows the possible onset of progressive loss in effectiveness. However, the overall protection appears to be quite good.



Figure B-3. EWI (left) and EonCoat® (right) after 5,000 Hours, Back Side



Figure B-4. EWI, Scribed to Metal. L→R: 500, 1500, 3000, 5000 Hours



Figure B-5. EonCoat®, Back Side. L→R: 500, 1500 Hours

Abbreviations and Acronyms

ALL	EWI 3-step coating process, with post bake
ASTM	American Society for Testing and Materials
EC	EonCoat® ceramic coating
EDS	Elemental X-ray Dispersive Analysis
FAST	Facility for Accelerated Service Testing
FRA	Federal Railroad Administration
LIRR	Long Island Rail Road
PATH	Port Authority Trans-Hudson
PD	EWI 2-step coating process
PDZ	EWI 3-step coating process, without post bake
PZ	EWI 2-step process, not having the di-phenolic conversion, but with post bake
SEM	Scanning Electron Microscopy
TCRP/TTCI	Transit Cooperative Research Program/Transportation Technology Center, Inc.

Chevron's Use of EonCoat® To Prevent Corrosion On Steel

Benjamin Chaloner-Gill, Ph.D.

Chevron U.S.A. 100 Chevron Way, Richmond, California 94801

bchalonergill@chevron.com

Martin Quintero

Chevron U.S.A. 1400 Smith Street, Houston, Texas 77002

mquintero@chevron.com

Michael Melancon

Chevron U.S.A. 100 Chevron Way, Richmond, California 94801

mmelancon@chevron.com

Jim Glowniak

Chevron U.S.A. 1400 Smith Street Houston, Texas 77002

jjgl@chevron.com

Mark Smith

Chevron U.S.A. 1400 Smith Street Houston, Texas 77002

smithme@chevron.com

Raoul Martin-Blouin

Chevron U.S.A. 1400 Smith Street Houston, Texas 77002

raoul@chevron.com

Syed M Taha Hussain

Chevron U.S.A. 1400 Smith Street Houston, Texas 77002

hussains@chevron.com

Brock Genter

Chevron U.S.A. 1564 China Grade Loop, Bakersfield, California 93308

brockgenter@chevron.com

Michael Davenport

Chevron U.S.A. 100 Chevron Way, Richmond, California 94801

mdvn@chevron.com

Sameer Patel

EonCoat, LLC 551 Pylon Drive Suite D Raleigh, North Carolina 27606

sameer.patel@eoncoat.com

Merrick Alpert

EonCoat, LLC 551 Pylon Drive Suite D Raleigh, North Carolina 27606

merrick.alpert@eoncoat.com

Amal Al-Borno

Charter Coating Service (2000) Ltd. #6, 4604 - 13 St. NE Calgary, AB T2E 6P1

aalborn@chartercoating.com

Abstract: The paper will discuss how Chevron U.S.A. has been successful in using EonCoat® to prevent atmospheric corrosion and using a high temperature version of the coating to prevent corrosion under insulation (CUI). Chevron's Benjamin Chaloner-Gill, Ph.D., Senior Advisor, Materials and Corrosion, R&D, is the senior author of the paper and will deliver the lecture at EUROCORR 2018. The resulting alloy forms a magnesium iron phosphate alloy layer to prevent atmospheric oxygen and moisture from reaching the substrate.

Third party laboratory testing (carried out at Charter Coating Service (2000) Ltd.) and field-test results will be presented to the audience. Commercial use case history will be shared with the presentation of a case study involving Chevron's use of the technology at their refinery in Pascagoula, Mississippi, USA.

Usage detail will be revealed, including surface preparation (ISO Sa 2), and the coating's unique characteristic that allow for the coating to be applied over a flash rusted surface.

Keywords: Chevron; corrosion protection; refinery best practice; CUI; corrosion under insulation

Introduction

In a recent paper delivered to the Society of Petroleum Engineers in Aberdeen, Scotland on 18 June 2018 (SPE – 190893 – MS), the chemistry of EonCoat was fully explained. In short, when EonCoat chemically reacts with carbon steel, a protective passivated layer over the iron is produced. This protective layer has two important attributes that are very attractive – (1) it is insoluble and (2) it is chemically bonded to the substrate. By understanding the chemistry of EonCoat and these features, this non-traditional (i.e., inorganic) coating system can be utilized in the protective coating realm.

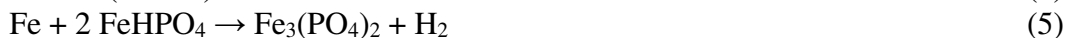
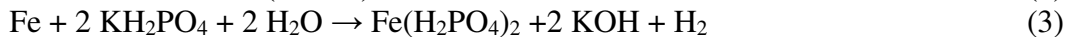
Chemistry of EonCoat

EonCoat is a two-component waterborne spray system. Part A is based on potassium monophosphate, KH_2PO_4 , and Part B contains magnesium hydroxide, $\text{Mg}(\text{OH})_2$. Minor amounts of proprietary components are added to the formulation to control the rate of the reaction, rheology of the system, stability (shelf life) of the product and some other properties. After spraying, the coating sets in minutes by acid-base reaction:



The product that forms in reaction (1) is a solid material with appearance and mechanical properties that resemble those of traditional ceramics.

As EonCoat is spray-applied onto carbon steel, the acid in EonCoat converts the top layer of steel into iron phosphate and/or iron magnesium phosphate so that the steel can no longer corrode. Passivation layer formation reaction tentatively can be represented by the following equations:



Since the above reactions occur in the presence of a metal oxide/hydroxide that is a major component of Part B, the following reactions can occur at the same time:



The second mechanism for corrosion protection is the cementitious layer. This results from the reaction (1) between the acid/acidic salt and the metal oxide. This cementitious layer which is based on a binder, $\text{MgKPO}_4 \cdot 6 \text{H}_2\text{O}$ which is a spatially soluble metal phosphate with solubility product constant $K_{\text{SP}} = 2.1 \cdot 10^{-12}$. The cementitious layer works as a phosphate reservoir providing phosphate ions to the steel surface for the whole lifetime of the coating.

In essence, EonCoat works by passivating the surface of the substrate so it can no longer react with corrosive substances such as oxygen and moisture. Unlike paint, EonCoat chemically bonds to the surface of the steel. And unlike paint, it is not a barrier coating.

Testing of The Technology

To fully understand this coating system, a thorough investigation has been undertaken. Testing has been conducted both in the field and at a third-party laboratory over a three-year period. Through this testing, we have learned how to use EonCoat as a coating system in two different regimes. The two regimes that have been tested are atmospheric corrosion below 100 °C and corrosion under insulation (CUI) both at low temperature (less than 100 °C) and at higher temperatures (up to 350 °C). Given our understanding of how EonCoat works, this coating system has been utilized as a protective coating at our facilities, in both applications.

To gain a full understanding of EonCoat as a protective coating system, atmospheric corrosion testing has been conducted in the following environments: salt fog testing (ASTM B117), cyclic corrosion testing (ASTM 5894), and CUI testing (NACE 2014, Paper 4193). Furthermore, testing also included various surface preparations Sa 2 ½ | SSPC SP 10, Sa 2 | SSPC SP6, SSPC SP 7 and SSPC SP 5. A third variable was also included in the surface preparation experimental matrix, soluble salts. While this test matrix could be a paper in itself, the results of the testing will be simplified for brevity.

The long-term laboratory test results revealed that the preferred surface preparation is Sa 2 | SSPC SP 6. The laboratory test results were consistent with field-testing. Field testing consisted of coating very large panels (5 feet x 7 feet) with EonCoat with the various surface preparations. Panels were placed at two different geographic locations in the United States – Pascagoula, Mississippi and Richmond, California. While these two locations differ greatly in both temperature and humidity, the performance results were the same. Salt levels made no difference in performance. Whether the steel was contaminated with salt or the steel was clean, no performance difference was observed.

Both of these conclusions have major implications in understanding the cost of EonCoat application. Additionally, as was detailed at the SPE conference, Florida International University's Department of Mechanical and Materials Engineering conducted extensive cyclic polarization studies (Reference – Unpublished data?) that reached the same conclusion. Internal EonCoat test data, reviewed by Chevron, also resulted in the same findings. EonCoat can be applied on a flash rusted or rusted surface. These results, a simplified surface preparation and being able to coat over a rusted surface, will lower the overall cost of application when compared with a standard three coat system. EonCoat projects this coating system to last 30+ years in atmospheric service. With a lower total cost of application and a simpler surface preparation prior to coating, EonCoat provides an attractive solution that facility operators are seeking.

Current coating products in the market place for CUI provide a very wide temperature range. CUI occurs due to damage to the coating system. If the coating is damaged and insulated, then CUI becomes a very costly inspection and maintenance problem. When damage to the coating system occurs, the damage is not intentional. But it leaves behind an asset integrity problem. What if the tables were turned? What if mechanical integrity is built into the protection layer? What if this layer is not susceptible to mechanical damage? If so, the solution would provide a large number of benefits. First and foremost, the failure mode has changed. It would no longer be the coating failure leading to a corrosion and inspection problem. This is the second attribute of EonCoat, the mechanical integrity of the protective layer, which changes the game for CUI.

To test the mechanical integrity of EonCoat, panels were intentionally damaged. Damage was inflicted in a number of different ways – (i) Impact damage with a ball being dropped on the panel, (ii) The panel was dropped from a height of 3-4 meters onto the cement, (iii) A saw blade was used to make a cut like mark in the protective layer (but not cut through the steel itself), (iv) Various hardness tests (designed to inflict mechanical damage) were done on the panel, and (v) A mechanical polisher was used in some areas. In the most extreme case, one of the panels was bent at a 90° angle. This bend did bring new steel to the surface. Figure 1 shows the panels after damage and before B117 exposure.



Figure 1 – EonCoat panels with intentional mechanical damage.

Then the panels were subjected to ASTM B117 conditions; the figures show the duration time for the panels. In the pictures below, we show the panels before B117 testing and then after 335 hours (Figure 2) and after 1,870 hours (Figure 3). In all cases, there is not rust in the areas where the panels were intentionally damaged, aside from the 90° bend in the panel. Again, the panels show no sign of rust. Organic coating would have failed to control the corrosion in these conditions.



Figure 2 – EonCoat panels with intentional damage after 335 hours of B117 exposure.



Figure 3 – EonCoat panels with intentional damage after 1,870 hours of B117 exposure.

Where new steel is exposed, that section of the panel rusted, as expected. But notice on the portion of the panel that was bent – at the edge of the EonCoat, there is no rust along the entire line. Meaning, when the EonCoat forms a chemical bond with the surface of the steel, the protective layer is formed. When this occurs, the mechanical integrity is maintained. (It should be noted that in the field, if a section of tank or pipe incurred mechanical damage as severe as a 90° bend, that steel would be taken out of service as it would be permanently deformed.)

Field-testing of EonCoat in CUI conditions continues. Figure 4 (below) shows cross-cut adhesion into the coating. The cross-cut adhesion was performed in February 2018 and then re-examined in June 2018, 4 months later. The picture in Figure 4 shows the cross-cut and subsequent removal of adjacent phosphate ceramic. This picture was taken in June 2018 after the pipe had been placed back in service. The pipe service, insulated with blankets, is running at temperatures ranging from 120 to 138 °C.



Figure 4 – Cross-cut adhesion of EonCoat – June 2018 after 4 months of being insulated and in service.

Like the intentionally damaged panels above, again, the EonCoat shows no rust. Organic coatings would have begun to show corrosion.

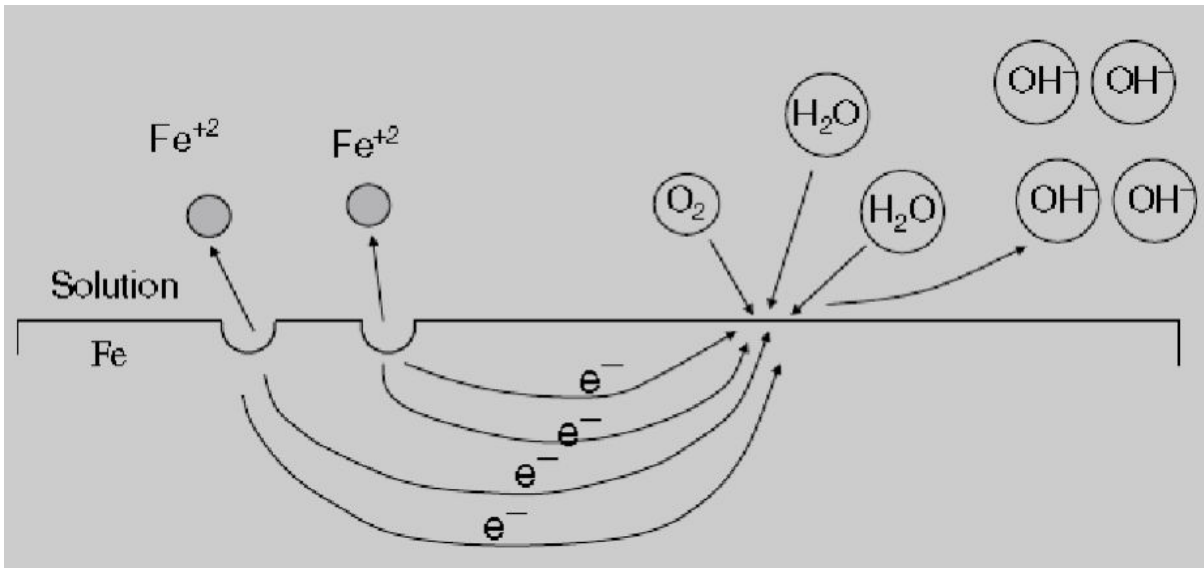
Conclusion

Through years of extensive lab and field tests, EonCoat is proving to be an effective anti-corrosion coating technology. In 2018, EonCoat was incorporated into the Chevron Engineering Standards.

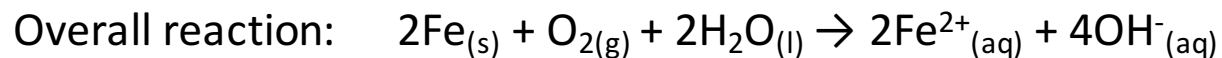
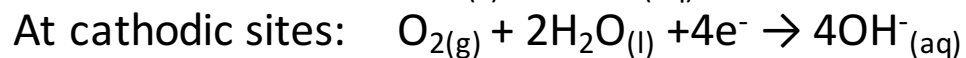
Corrosion is an electrochemical process.

There are four requirements for electrochemical corrosion cell:

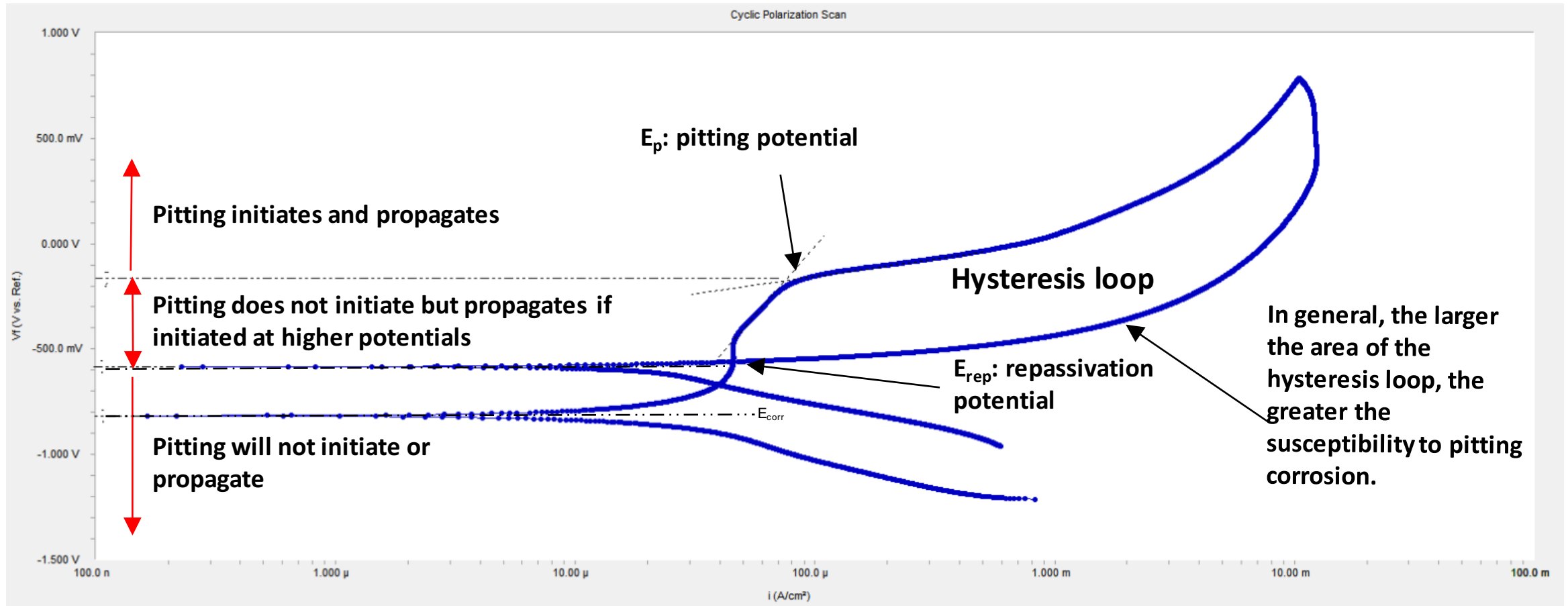
1. **cathodic site** (where electrons are consumed);
2. **anodic sites** (usually where corrosion occurs). Both sites can be on the same piece of metal;
3. cathode and anode are connected through the solution by **an ionic current pass**;
4. and they are connected through the metal by **an electronic path**.

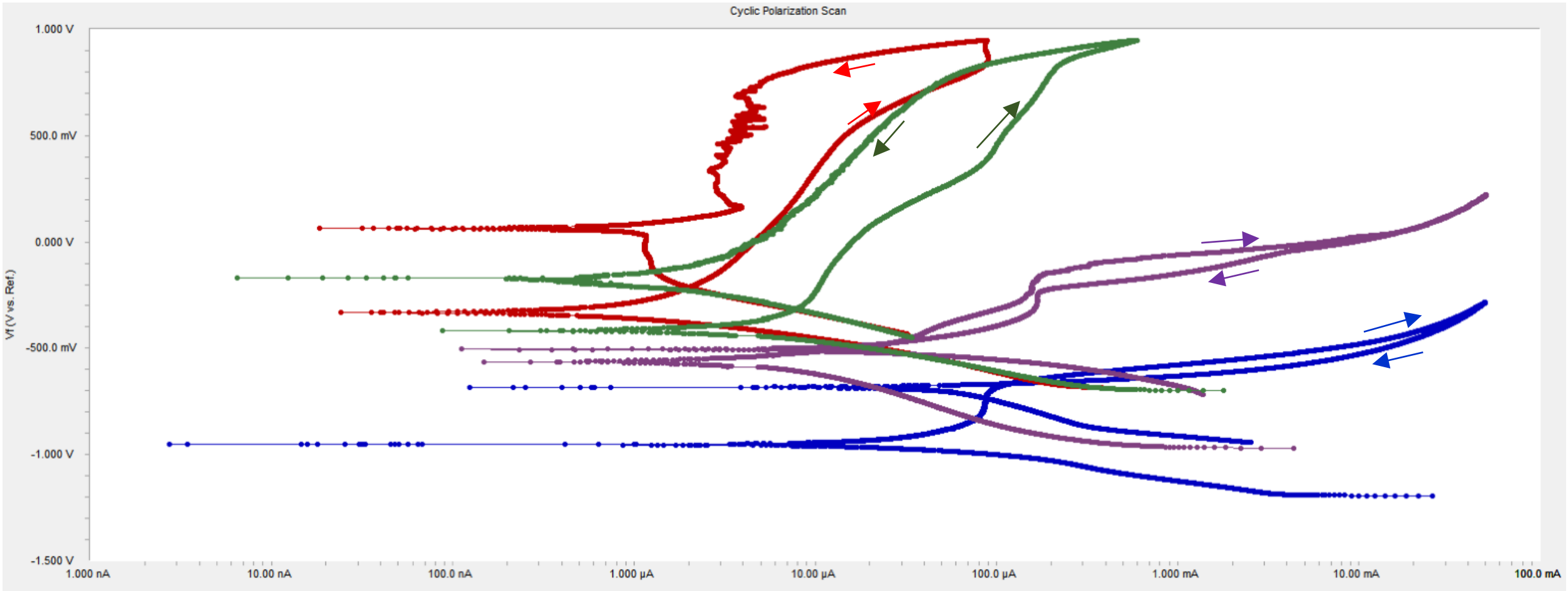


Because corrosion occurs via electrochemical reactions, electrochemical techniques are ideal for the study of the corrosion processes.



HOW TO INTERPRET A CYCLIC POLARIZATION CURVE





Cyclic polarization curve for Eoncoat (green) in comparison with mild steel (blue), stainless steel 304 (magenta) and Hastelloy (red). Arrows show directions of forward and backward sweeps.