

#### DEPARTMENT OF THE NAVY COMMANDER NAVY REGION HAWAII 850 TICONDEROGA ST STE 110 JBPHH, HAWAII 96860-5101

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CERTIFIED NO: 7015 0640 0003 8076 1341 Mr. Omer Shalev U.S. Environmental Protection Agency, Region IX 75 Hawthorne Street San Francisco, CA 94105

# CERTIFIED NO: 7015 0640 0003 8076 1358

Ms. Roxanne Kwan State of Hawaii Department of Health Solid and Hazardous Waste Branch 2827 Waimano Home Road Pearl City, HI 96782

Dear Mr. Shalev and Ms. Kwan:

SUBJECT: DESTRUCTIVE ANALYSIS OF 10 STEEL COUPONS REMOVED FROM RED HILL FUEL STORAGE TANK NO. 14, PURSUANT TO SECTION 5.3 OF THE ADMINISTRATIVE ORDER ON CONSENT STATEMENT OF WORK, RED HILL BULK FUEL STORAGE FACILITY, JOINT BASE PEARL HARBOR-HICKAM, OAHU, HAWAII

Please find enclosed the Destructive Analysis of 10 Steel Coupons Removed from Red Hill Fuel Storage Tank No. 14. This interim report was produced to support ongoing analysis pursuant to the Administrative Order on Consent (AOC) Statement of Work (SOW), Section 5.3. as described in the Red Hill Bulk Fuel Storage Facility Scope of Work for Destructive Testing Supplement, Destructive Testing Plan, 1 June 2018.

The U.S. Department of the Navy (the Navy) and Defense Logistics Agency (DLA) developed this report based on the concurrence with the U.S. Environmental Protection Agency (EPA) and the State of Hawaii Department of Health (DOH) of the objectives and requirements outlined in Section 5.3 of the SOW and related correspondence between the AOC parties.

If you have any questions, please contact Mark S. Manfredi, the Red Hill Regional Program Director/Project Coordinator at (808) 473-4148 or via email at mark.manfredi@navy.mil.

Sincerely. M. R. DELAO

Captain, CEC, U.S. Navy Regional Engineer By direction of the Commander

Enclosure: (1) Destructive Analysis of 10 Steel Coupons Removed from Red Hill Fuel Storage Tank #14



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# Destructive Analysis of 10 Steel Coupons Removed from Red Hill Fuel Storage Tank #14

Solomon Resources, LLC 1001 Kamokila Blvd., Suite 125 Kapolei Bldg. Kapolei, HI 96707

**Attention: Michelle Mercado** 

# **REPORT No. 201801967**

December 17, 2018

Report By:

Thomas N. Ackerson, P.E. and Jennifer Breetz





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Solomon Resources, LLC 1001 Kamokila Blvd., Suite 125 Kapolei Bldg. Kapolei, HI 96707 December 17, 2018

Attention: Michelle Mercado

# Report No. 201801967

# Destructive Analysis of 10 Steel Coupons Removed from Red Hill Fuel Storage Tank #14

# INTRODUCTION

The United States Navy maintains a set of underground fuel storage tanks at Red Hill approximately five miles northwest of Honolulu, Hawaii. To ensure tank integrity, the Navy has contractors perform non-destructive evaluation (NDE) on the operational tanks at periodic intervals. NDE processes include identifying locations of possible excessive back-side thinning, back-side pitting, and weld defects, as well as the minimum wall thickness of each section of the tank walls.

The primary purpose of this study was to provide in-depth chemical and physical analysis of coupon samples provided by the customer from Red Hill Tank #14, in an effort to assist them in validating NDE results. Ten coupons removed from the tank were submitted for analysis, and were approximately 12" X 12" in size. They were labeled as 1, 2, 3, 5, 6, 7, 8, 10, A1 and A2 (there was no Sample #4 or #9). Poly bags of concrete powder samples and corrosion product were also submitted with the coupons. The coupons and bagged samples were evaluated using a combination of laboratory techniques. It was reported that the specified tank wall thickness was 250 mils.

An initial evaluation was performed on the coupons in the as-received condition (i.e., prior to the removal of loose corrosion product), which included visual inspection and computerized tomography scans. This was followed by a step-wise process of excising corrosion product for analysis, CO<sub>2</sub> blast cleaning, and determining microstructural, chemical and mechanical properties.

The work was performed over a 4-month period, from August to November 2018. All analytical work was performed by IMR Test Labs – Louisville, under the direction of the authors, unless otherwise indicated. IMR Test Labs – Louisville is accredited to ISO 17025 by the American Associate for Laboratory Accreditation (A2LA), Certificate #1140-03 and 1140-04.

# ANALYTICAL PROCEDURES

#### I. Visual Examination and Optical Microscopic Examination

- A. Visual Observations
- B. Photography (digital)
- C. Digital Optical Microscopy
- D. Dimensional Measurements

#### II. Non-Destructive Inspection

A. Computer Tomography (CT), subcontracted to an approved vendor using personnel certified and qualified in accordance with ASNT Recommended Practice No. SNT-TC-1A: *Personnel Qualification and Certification in Nondestructive Testing.* 

#### III. Chemical Analysis: Base Metal

- A. Optical Emission Spectroscopy, ASTM E415-17
- B. Carbon and Sulfur Content by Combustion Analysis, ASTM E1019-18

#### **IV. Scanning Electron Microscopic Examination**

A. Scanning Electron Microscopy (SEM)

#### V. Microanalysis: Deposits, Corrosion Products, etc.

- A. Energy Dispersive X-ray Spectroscopy (EDS) in conjunction with SEM, permits detection and guantification of all elements greater than beryllium in atomic weight, ASTM E1508-12a.
- B. Ion Chromatography for water soluble anions (IC), ASTM D4327-17 and pH measurement per ASTM D1293-18
- C. Fourier Transform Infrared Spectroscopy (FTIR) FTIR is a nondestructive microanalytical spectroscopic technique that involves the study of molecular vibrations. Reference ASTM E1252-98(13)e1
- D. X-Ray Diffraction Spectroscopy Analysis (XRD) This technique generates a diffraction pattern and matches unique peaks using a computer-based library in order to identify compounds by their crystalline structure, CLP-056A

#### VI. Mechanical Testing

- A. Tensile Testing, ASTM E8-16a
- B. Rockwell Hardness Testing, ASTM E18-17e1

#### VII. Metallography

- A. Microstructural Analysis using a Light Metallurgical Microscope, specimen preparation in accordance with ASTM E3-11 (17)
- B. Pit Depth Measurement per ASTM G46-94 (2005), as referenced by ASTM G1 03(2017)e1

# RESULTS

# **VISUAL EXAMINATION**

Overall images of the submitted coupons in the as-received condition are provided in Figures 1 through 30.

Computer Tomography (CT) scans were performed on each of the submitted coupons prior to cleaning or sectioning. This task was subcontracted to an accredited partner laboratory. Representative screenshots of the scans are provided in Figures 31 through 40. The full CT scan files and appropriate software will be provided to the customer on a separate disk. The CT scans were used to identify three regions on each coupon that exhibited the most severe wall thinning or any other density-based anomaly. The three regions of severe wall thinning identified were excised from the coupons for further analysis.

#### **CHEMICAL ANALYSIS**

Chemical analysis was performed on the base metal of specimens excised from each coupon using optical emission spectroscopy (OES). The results are summarized in Tables 1 through 4. The samples were consistent with ASTM A36 structural steel, or similar. No specification for the steel was provided.

# SCANNING ELECTRON MICROSCOPY

The three regions removed from each coupon were examined using a scanning electron microscope (SEM) with imaging in secondary electron mode. Representative images of the features observed are provided as Figures 41 through 70. When no specific pitting was observed, the nominal surface of the specimen was imaged and analyzed.

## **MICROANALYSIS**

The three regions removed from each coupon were analyzed using energy dispersive x-ray spectroscopy (EDS). Summaries of the results are provided in Tables 5 through 14. Representative spectra obtained are provided in Figures 71 through 100.

The concrete samples were analyzed by ion chromatography (IC) for common anions. Tables 15 and 16 summarize the ion chromatography results along with the pH values of the analyzed solution from the samples. The ion chromatograms generated are provided as Figures 101 through 110.

Fourier transform infrared (FTIR) spectroscopy was performed on paint samples removed from each coupon using Attenuated Total Reflectance (ATR) with a diamond crystal. All paint samples were identified as polyurethane enamel. The resulting FTIR spectra of the samples and the best matches from the spectral FTIR library of standards for polyurethane enamel are provided as Figures 111 and 112.

X-Ray Diffraction Spectroscopy (XRD) analysis of the concrete samples revealed the presence of the following crystalline phases: calcium oxide (lime); calcium carbonate (calcite); silicon dioxide (coesite); calcium sulfate; calcium sulfate hydrated (gypsum) and calcium hydroxide (portlandite). The results are summarized in Tables 17 and 18. The XRD diffractograms for the concrete samples are provided as Figures 113 through 122.

X-Ray Diffraction Spectroscopy (XRD) analysis of the corrosion product from the metal panels revealed the presence of the following crystalline phases: iron oxide (magnetite); iron hydroxide (goethite); calcium carbonate (calcite); silicon dioxide and hydrous calcium aluminum sulfate (ettringite). The results are summarized in Tables 19 and 20. The XRD diffractograms for the corrosion samples are provided as Figures 123 through 128.

### **CLEANING OF COUPONS AND 3-D IMAGING**

After analysis of the corrosion product and concrete, both sides of the coupons were cleaned using a CO<sub>2</sub> dry ice blast process by a subcontractor. Cleaning was witnessed by the report authors. The CO<sub>2</sub> technique was selected due to its ability to remove most of the residual concrete on the coupon surface, yet not be so aggressive as to remove base metal. Chemical cleaning as prescribed in ASTM G1-03(2011) would not have adequately cleaned the surfaces for analysis. Representative images of the panels after cleaning are provided as Figures 129 through 148. The majority of the residual concrete and loose corrosion product was removed, however the green paint could not be removed.

Digital optical 3D images acquired of the three specimens removed from each coupon are shown in Figures 149 through 179.

#### **MECHANICAL TESTING**

Full size flat tensile specimens were excised from each coupon. It could not be readily determined if the samples were removed from the rolling direction of the steel, or transverse to the rolling direction, as this was not marked on the coupons. The results of the tensile testing are summarized in Table 21.

Rockwell hardness specimens were excised from the coupons and the results are presented in Table 22.

### **METALLOGRAPHY**

A minimum of three metallographic specimens were prepared from each of the 10 coupons and were identified as #-1, #-2 and #-3, where "#" was the original coupon number. In some cases, additional areas of interest were identified during the course of the analysis. When this occurred, those samples were identified as #-4, #-5, etc. Some of these additional samples were determined to be worst-case areas for wall loss and are included in Table 23 as such.

In preparation for mounting and polishing, each specimen was sectioned as necessary using a water-cooled saw. The sectioned specimens were mounted in either glass-filled thermosetting epoxy (compression mount) or 2-part slow-cure epoxy (cold mount), depending on the specimen size. The mounted samples were ground and polished using standard metallographic techniques. The finished specimens were used for determining pitting depth, weld imaging, and paint characterization. All specimens were chemically etched with a 2% Nital solution to reveal the microstructure and facilitate imaging.

#### Pit Depth Measurement

The area exhibiting the maximum degree of wall thinning was measured for each prepared specimen, then the percent remaining wall thickness was calculated. The specified wall thickness of 250 mils, provided by the customer, was used for percent remaining wall thickness calculations. For

additional information, local wall thickness measurements were made in the least observed corroded location for each submitted coupon after the remaining paint was removed by grit-blasting. These readings were made using calibrated digital micrometers. The results are summarized in Table 23. Note that some of the NDE indications were likely due to surface anomalies other than pitting (i.e., welding or mechanical depressions on the painted surface). Representative images with calibrated markers are provided in Figures 180 through 207. Markers on the images are in metric units, but were converted to mils for the purposes of Table 23.

# Weld Characterization

Coupon 8 exhibited what is presumed to be a prior weld repair and/or addition of a backing plate, where a patch was made on the tank wall. Representative images of the welds are presented as Figures 208 through 213. A full weld evaluation is outside the scope of this effort. The results are thus provided for information only.

# Paint Characterization

A typical paint layer was examined microscopically and was determined to consist of two layers, as shown in Figure 214. This indicates that the exterior tank wall exhibits a primer layer with a top coat. The scraped paint samples that were excised from each coupon previously for FTIR analysis would likely have captured some of both layers. The FTIR results presented previously were a good match for polyurethane, with no observable secondary component. Although this indicates that both the primer and top coat were polyurethane-based, it cannot be ruled out that the primer exhibited a different composition.



Respectfully submitted

Thomas N. Ackerson PE Laboratory Director

Concurrence

Jennifer Breetz Metallurgy/Corrosion Manager Materials Engineer/Failure Analyst

All procedures were performed in accordance with the IMR Quality Manual, current revision, and related procedures; and the PWA MCL Manual F-23 and related procedures. The information contained in this test report represents only the material tested and may not be reproduced, except in full, without the written approval of IMR Test Labs ("IMR"). IMR maintains a quality system in compliance with the ISO/IEC 17025 and is accredited by A2LA, certificates #1140.03 and #1140.04. IMR will perform all testing in good faith using the proper procedures, trained personnel, and equipment to accomplish the testing required. Conformance will be based on results without measurement uncertainty applied, unless otherwise requested by the customer. IMR's liability to the customer or any third party is limited at all times to the amount charged for the services provided. All test samples will be retained for a minimum of 3 months and may be destroyed thereafter, unless otherwise specified by the customer. The recording of false, fictitious, or fraudulent statements or entries on this document may be punished as a felony under federal statutes. IMR Test Labs is a GEAE S-400 approved lab (Supplier Code T9334).

# TABLE 1 – CHEMICAL ANALYSIS TEST RESULTS, COUPONS 1, 2 AND 3

Element	#1	#2	#3	ASTM A36-14 Plates > 15" width and Plates up to <sup>3</sup> ⁄ <sub>4</sub> " Thick Requirements
Carbon	0.16	0.16	0.16	0.25 Maximum
Manganese	0.51	0.49	0.48	
Silicon	0.06	0.03	0.02	0.40 Maximum
Phosphorus	0.006	0.006	0.006	0.030 Maximum
Sulfur	0.019	0.019	0.019	0.030 Maximum
Chromium	0.04	0.02	0.01	
Nickel	0.04	0.01	0.01	
Molybdenum	0.01	< 0.01	< 0.01	
Aluminum	0.01	0.02	0.02	
Cobalt	0.01	0.01	0.01	
Copper	0.09	0.03	0.03	0.20 Minimum <sup>1</sup>
Niobium	< 0.01	< 0.01	< 0.01	
Titanium	< 0.01	< 0.01	< 0.01	
Vanadium	< 0.01	< 0.01	< 0.01	
Iron	Remainder	Remainder	Remainder	Remainder

Results in weight percent

<sup>1</sup>Copper minimum when copper steel is specified

# **METHODS** Optical Emission Spectroscopy, ASTM E415-17

# TABLE 2 – CHEMICAL ANALYSIS TEST RESULTS, COUPONS 5, 6 AND 7

Element	#5	#6	#7	ASTM A36-14 Plates > 15" width and Plates up to <sup>3</sup> ⁄ <sub>4</sub> " Thick Requirements
Carbon	0.11	0.20	0.16	0.25 Maximum
Manganese	0.44	0.49	0.52	
Silicon	0.05	0.05	0.06	0.40 Maximum
Phosphorus	0.014	0.007	0.024	0.030 Maximum
Sulfur	0.012	0.017	0.028	0.030 Maximum
Chromium	0.01	0.02	0.02	
Nickel	0.01	0.02	0.01	
Molybdenum	< 0.01	0.01	< 0.01	
Aluminum	0.02	0.01	0.01	
Cobalt	0.01	0.01	0.01	
Copper	0.03	0.07	0.04	0.20 Minimum <sup>1</sup>
Niobium	< 0.01	< 0.01	< 0.01	
Titanium	< 0.01	< 0.01	< 0.01	
Vanadium	< 0.01	< 0.01	< 0.01	
Iron	Remainder	Remainder	Remainder	Remainder

Results in weight percent

<sup>1</sup>Copper minimum when copper steel is specified

# METHODS

Optical Emission Spectroscopy, ASTM E415-17

# TABLE 3 - CHEMICAL ANALYSIS TEST RESULTS, COUPONS 8 AND 10

Element	#8	#10	ASTM A36-14 Plates > 15" width and Plates up to <sup>3</sup> ⁄4" Thick Requirements
Carbon	0.14	0.20	0.25 Maximum
Manganese	0.46	0.47	
Silicon	0.06	0.06	0.40 Maximum
Phosphorus	0.018	0.012	0.030 Maximum
Sulfur	0.014	0.040*	0.030 Maximum
Chromium	0.01	0.02	
Nickel	0.01	0.01	
Molybdenum	< 0.01	< 0.01	
Aluminum	0.01	0.01	
Cobalt	0.01	0.01	
Copper	0.05	0.02	0.20 Minimum <sup>1</sup>
Niobium	< 0.01	< 0.01	
Titanium	< 0.01	< 0.01	
Vanadium	< 0.01	< 0.01	
Iron	Remainder	Remainder	Remainder

Results in weight percent

\*Fails to meet requirements of ASTM A36, however, no specification was provided.

<sup>1</sup>Copper minimum when copper steel is specified

# **METHODS**

Optical Emission Spectroscopy, ASTM E415-17 Carbon and Sulfur by Combustion, ASTM E1019-18

# TABLE 4 – CHEMICAL ANALYSIS TEST RESULTS, COUPONS A1 AND A2

Element	#A1	#A2	ASTM A36-14 Plates > 15" width and Plates up to <sup>3</sup> ⁄4" Thick Requirements
Carbon	0.15	0.16	0.25 Maximum
Manganese	0.49	0.46	
Silicon	0.06	0.06	0.40 Maximum
Phosphorus	0.013	0.020	0.030 Maximum
Sulfur	0.018	0.022	0.030 Maximum
Chromium	0.01	0.01	
Nickel	0.01	0.01	
Molybdenum	< 0.01	< 0.01	
Aluminum	0.01	0.02	
Cobalt	0.01	0.01	
Copper	0.04	0.02	0.20 Minimum <sup>1</sup>
Niobium	< 0.01	< 0.01	
Titanium	< 0.01	< 0.01	
Vanadium	< 0.01	< 0.01	
Iron	Remainder	Remainder	Remainder

Results in weight percent

<sup>1</sup>Copper minimum when copper steel is specified

# METHODS

Optical Emission Spectroscopy, ASTM E415-17

## TABLE 5 - ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 1

Element	Location 1-1	Location 1-2	Location 1-3
Carbon	7.1	2.9	17.7
Oxygen	35.2	37.9	37.2
Sodium	1.5	1.2	0.8
Magnesium			
Aluminum	0.4		
Silicon	1.7	1.5	0.6
Phosphorus			
Sulfur			
Chlorine			
Potassium			
Calcium	4.8		12.0
Titanium			
Vanadium			
Chromium			
Manganese	0.3	0.4	0.4
Iron	48.8	55.9	31.3
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 6 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 2

Element	Location 2-1	Location 2-2	Location 2-3
Carbon	6.0	7.1	6.4
Oxygen	37.9	36.6	37.1
Sodium			
Magnesium			
Aluminum			
Silicon			
Phosphorus			
Sulfur	0.2		
Chlorine	0.3	0.6	0.2
Potassium			
Calcium			0.8
Titanium			
Vanadium			
Chromium			
Manganese	0.5	0.4	0.7
Iron	54.9	55.1	53.7
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 7 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 3

Element	Location 3-1	Location 3-2	Location 3-3
Carbon	6.8	6.6	3.71
Oxygen	38.5	42.7	37.4
Sodium		0.2	
Magnesium			
Aluminum	0.2		
Silicon	0.2		
Phosphorus			
Sulfur			0.4
Chlorine			0.3
Potassium			
Calcium	6.7	0.6	
Titanium			
Vanadium			
Chromium			
Manganese	0.5	0.3	0.4
Iron	46.8	49.4	57.7
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 8 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 5

Element	Location 5-1	Location 5-2	Location 5-3
Carbon	5.8	3.4	17.2
Oxygen	28.4	27.2	35.8
Sodium	0.4		0.5
Magnesium	0.2		0.3
Aluminum	0.3		1.2
Silicon	0.9		3.5
Phosphorus	0.2		
Sulfur			0.3
Chlorine			0.3
Potassium			
Calcium	2.5	0.6	15.2
Titanium			
Vanadium			
Chromium			
Manganese	0.2	0.3	
Iron	61.0	68.4	25.7
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 9 - ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 6

Element	Location 6-1	Location 6-2	Location 6-3
Carbon	2.1	3.8	3.1
Oxygen	34.1	34.6	33.3
Sodium			
Magnesium			
Aluminum		2.1	
Silicon		4.1	
Phosphorus			
Sulfur		0.3	0.2
Chlorine			0.3
Potassium			
Calcium	1.2	13.9	0.2
Titanium			
Vanadium			
Chromium			
Manganese	0.4		0.4
Iron	61.9	41.3	62.3
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 10 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 7

Element	Location 7-1	Location 7-2	Location 7-3
Carbon	3.6	5.0	3.6
Oxygen	28.5	38.7	28.5
Sodium	0.3		0.3
Magnesium			
Aluminum			
Silicon			
Phosphorus			
Sulfur			
Chlorine	0.6	0.3	0.6
Potassium			
Calcium	0.8	6.1	0.8
Titanium			
Vanadium			
Chromium			
Manganese	1.0	0.4	1.0
Iron	64.7	49.2	64.7
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations  $\geq 0.2\%$  are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 11 - ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 8

Element	Location 8-1	Location 8-2	Location 8-3
Carbon	4.7	4.5	5.1
Oxygen	31.8	33.9	33.2
Sodium	0.5	0.4	0.5
Magnesium			
Aluminum	0.3		
Silicon	1.0	0.3	0.4
Phosphorus			
Sulfur			
Chlorine			
Potassium			
Calcium	2.9	0.8	0.9
Titanium			
Vanadium			
Chromium			
Manganese	0.4	0.3	0.3
Iron	58.2	59.5	59.2
Nickel			
Copper			0.3
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 12 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON 10

Element	Location 10-1	Location 10-2	Location 10-3
Carbon	34.9	27.6	22.5
Oxygen	20.1	16.5	23.7
Sodium	0.3	0.2	0.3
Magnesium			
Aluminum	0.2	1.6	0.5
Silicon	0.5	0.4	0.8
Phosphorus			
Sulfur	0.3	0.4	0.4
Chlorine		0.3	
Potassium		0.2	
Calcium	1.2	0.5	2.2
Titanium			
Vanadium			
Chromium			
Manganese		0.2	
Iron	41.9	52.1	49.2
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 13 - ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON A1

Element	Location A1-1	Location A1-2	Location A1-3
Carbon	3.1	3.7	3.4
Oxygen	33.7	31.6	28.2
Sodium			
Magnesium			
Aluminum			0.3
Silicon			0.6
Phosphorus			
Sulfur		0.2	
Chlorine	1.7	0.7	0.5
Potassium			
Calcium			2.7
Titanium			
Vanadium			
Chromium			0.2
Manganese	0.4	0.6	0.3
Iron	61.0	63.2	63.6
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

## TABLE 14 – ENERGY DISPERSIVE X-RAY SPECTROMETRY RESULTS, COUPON A2

Element	Location A2-1	Location A2-2	Location A2-3
Carbon	2.7	2.3	1.9
Oxygen	32.6	32.9	31.1
Sodium		0.3	
Magnesium	0.3		
Aluminum	0.3		0.4
Silicon	1.0	0.6	1.4
Phosphorus			
Sulfur			
Chlorine			
Potassium			
Calcium	2.3	1.7	2.5
Titanium			
Vanadium			
Chromium			
Manganese	0.3	0.4	0.2
Iron	60.7	61.4	62.4
Nickel			
Copper			
Zinc			

• All reported values are in relative weight percent.

• Only elements present in concentrations ≥ 0.2% are reported.

• Results may not add up to 100%, due to rounding or lesser concentrations not being reported.

Anion	Concrete 1	Concrete 2	Concrete 3	Concrete 5	Concrete 6
Fluoride	11	11	27	13	13
Chloride	82	59	50	80	81
Nitrite	282	595	24	26	17
Nitrate	273	535	15	12	3
Sulfate	1518	684	1501	1979	1660
рН	9.86 (alkaline)	11.79 (alkaline)	11.03 (alkaline)	11.13 (alkaline)	10.27 (alkaline)

# TABLE 15 – ION CHROMATOGRAPHY AND pH TEST RESULTS

Results in parts per million (ppm) unless otherwise indicated

Method(s): Modified ASTM D 4327-11 (IC) and pH measurement per ASTM D1293-12.

# TABLE 16 – ION CHROMATOGRAPHY AND pH TEST RESULTS

Anion	Concrete 7	Concrete 8	Concrete 10	Concrete A1	Concrete A2
Fluoride	25	70	10	3	16
Chloride	78	171	62	53	80
Nitrite	20	18	6	15	17
Nitrate	5	4	3	6	8
Sulfate	2190	2392	2092	1266	1754
рН	10.65 (alkaline)	10.55 (alkaline)	10.37 (alkaline)	10.45 (alkaline)	10.10 (alkaline)

Results in parts per million (ppm) unless otherwise indicated

Method(s): Modified ASTM D 4327-11 (IC) and pH measurement per ASTM D1293-12.

Phase	Concrete 1	Concrete 2	Concrete 3	Concrete 5	Concrete 6
Calcium Oxide (CaO) (Lime)	4 (±0.3)	ND	4 (±0.2)	2 (±0.2)	11 (±1.5)
Calcium Carbonate (CaCO <sub>3</sub> ) (Calcite)	37 (±1.0)	5 (±0.5)	12 (±0.4)	53 (±1.2)	49 (±1.8)
Silicon Dioxide (SiO <sub>2</sub> ) (Coesite)	22 (±0.4)	8 (±0.5)	41 (±0.6)	10 (±0.3)	37 (±1.3)
Calcium Sulfate (CaSO <sub>4</sub> )	36 (±1.0)	ND	ND	ND	ND
Calcium Sulfate Hydrated (CaSO <sub>4</sub> 2H <sub>2</sub> O) (Gypsum)	1 (±0.8)	ND	16 (±0.2)	27 (±0.3)	3 (±0.4)
Calcium Hydroxide (Ca(HO) <sub>2</sub> ) (Portlandite)	ND	87 (±0.8)	27 (±0.6)	8 (±0.2)	ND

Values in relative weight percent ND=Not Detected

# TABLE 18 – X-RAY DIFFRACTION ANALYSIS TEST RESULTS FOR CONCRETE

Phase	Concrete 7	Concrete 8	Concrete 10	Concrete A1	Concrete A2
Calcium Oxide (CaO) (Lime)	13 (±0.7)	3 (±0.6)	2 (±0.2)	2 (±0.2)	12 (±1.0)
Calcium Carbonate (CaCO <sub>3</sub> ) (Calcite)	49 (±1.3)	32 (±0.2)	55 (±1.2)	69 (±1.1)	39 (±2.7)
Silicon Dioxide (SiO <sub>2</sub> )	30 (±1.0)	60 (±0.7)	32 (±0.7)	12 (±0.6)	4 (±0.7)
Calcium Sulfate (CaSO <sub>4</sub> )	ND	ND	ND	7 (±0.6)	38 (±2.9)
Calcium Sulfate Hydrated (CaSO <sub>4</sub> 2H <sub>2</sub> O) (Gypsum)	8 (±1.5)	3 (±0.9)	10 (±0.2)	10 (±0.3)	7 (±0.5)
Calcium Hydroxide (Ca(HO)₂) (Portlandite)	ND	2 (±0.4)	1 (±0.3)	ND	ND

Values in relative weight percent ND=Not Detected

# TABLE 19 - X-RAY DIFFRACTION ANALYSIS TEST RESULTS FOR CORROSION PRODUCT

Phase	Corrosion 1	Corrosion 2	Corrosion 3
Iron Oxide (Fe <sub>3</sub> O <sub>2</sub> )	60 (±0.3)	57 (±0.4)	48 (±1.1)
Iron Hydroxide (FeHO <sub>2</sub> )	40 (±1.2)	34 (±0.6)	46 (±1.2)
Calcium Carbonate (CaCO <sub>3</sub> )	ND	6 (±0.5)	6 (±0.6)
Silicon Dioxide (SiO <sub>2</sub> )	ND	3 (±0.3)	ND
Hydrous Calcium Aluminum Sulfate (Ca <sub>6</sub> Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> (OH) <sub>12</sub> 26 H <sub>2</sub> O)	ND	ND	ND

Values in relative weight percent ND=Not Detected

# TABLE 20 – X-RAY DIFFRACTION ANALYSIS TEST RESULTS FOR CORROSION PRODUCT

Phase	Corrosion 6	Corrosion 7	Corrosion 8
Iron Oxide (Fe3O2)	50 (±4.6)	84(±1.5)	17 (±0.3)
Iron Hydroxide (FeHO2)	33 (±1.3)	16(±0.7)	71 (±1.0)
Calcium Carbonate (CaCO3)	ND	ND	ND
Silicon Dioxide (SiO2)	4 (±0.9)	ND	12 (±1.1)
Hydrous Calcium Aluminum Sulfate (Ca <sub>6</sub> Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> (OH) <sub>12</sub> 26 H <sub>2</sub> O)	13 (±1.7)	ND	ND

Values in relative weight percent ND=Not Detected

# **TABLE 21 - TENSION TEST RESULTS**

Sample Identification	Tensile Strength (ksi)②	Yield Strength (ksi)①②	Elongation (%)②③
1	62.0	38.9	36
2	63.5	39.1	36
3	61.5	40.1	35
5	66.0	44.4	33
6	68.0	42.5	32
7	68.5	46.3	32
8	65.0	45.9	34
10	67.5	45.8	23
A1	66.5	44.8	35
A2	67.5	44.1	30

**NOTE:** ① Yield strength was determined using the 0.2% offset method.

② Mechanical properties were measured using a standard specimen with nominal gauge dimensions of 0.500" x 2.000".

③ Elongation was calculated using elongation-after-fracture measurements

# **METHODS**

Tensile Testing, ASTM E8/8M-16a

# TABLE 22 - HARDNESS TEST RESULTS

Sample Identification	Rockwell B Hardness, HRBW					
	Reading 1	Reading 2	Reading 3	Reading 4	Average	
1	68	68	68	68	68	
2	67	66	66	66	66	
3	66	66	66	66	66	
5	68	68	68	68	68	
6	72	73	73	73	73	
7	72	72	73	72	72	
8	71	71	71	71	71	
10	68	69	68	68	68	
A1	72	72	72	72	72	
A2	72	73	72	72	72	

# METHODS Rockwell Hardness, ASTM E18-17<sup>ε1</sup>

Solomon Resources, LLC

### **TABLE 23 – COUPON THICKNESS AND THINNING MEASUREMENT RESULTS**

Coupon #	Location	Sample ID	Specified Plate Thickness, mils	Measured Plate Thickness, mil①	Thinnest Measured Location, mil	Remaining Wall Thickness, %②
1	1	1-2		250	222.4	89.0
	2	1-3			218.5	87.4
	3	1-4			207.9	83.1
2	1	2-1		261	152.4	60.9
	2	2-2			159.8	63.9
	3	2-3			186.6	74.6
3	1	3-1		250	171.3	68.5
	2	3-2			145.3	58.1
	3	3-3			131.5	52.6
	4	3-4			233.9	93.5
	1	5-1		251	247.6	99.1
53	2	5-4			225.6	90.2
	3	5-5	250		224.0	89.6
6	1	6-1		245	157.9	63.1
	2	6-2			206.7	82.7
	3	6-3			224.0	89.6
7	1	7-1		252	163.8	65.5
	2	7-3			172.4	69.0
	3	7-4			166.1	66.5
8@	1	8-1		234		
	2	8-3			205.9	82.4
	3	8-4				
10⑤	1	10-1		245	241.7	96.7
	2	10-2			242.1	96.9
	3	10-4			N/A	N/A
A1	1	A1-1		252	193.7	77.5
	2	A1-4			122.4	49.0
	3	A1-5			192.9	77.2
A2©	1	A2-3			247.6	99.1
	2	A2-4		254	250.8	100.3
	3	N/A			N/A	N/A

① Local wall thickness, as measured with calibrated digital micrometers after removing paint layer. Measurement uncertainty available upon request.

© Calculated using specified tank wall thickness (250 mils). Measurement uncertainty available upon request.

③ Suspected NDT inspection indication was a depression on the painted face of Sample 5-4. No pits observed.

④ This is the weld-repaired plate section. Sample 8-3 exhibited a depression or pit on the painted surface.

© No pits observed. Suspected NDT inspection indication was a weld nugget on the interior surface of the coupon (thick area) on Sample 10-4.

© No pits observed. Suspected NDT inspection indications are areas of melting on the painted face, underneath the paint on Sample A2-4.

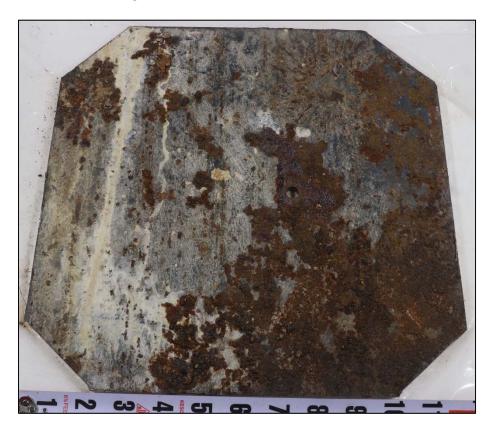


Figure 1. An image of the interior surface of Coupon 1 in the as-received condition.



Figure 2. An image of the exterior surface of Coupon 1 in the as-received condition.



**Figure 3.** An image of a representative edge of Coupon 1 in the as-received condition.



Figure 4. An image of the interior surface of Coupon 2 in the as-received condition.



Figure 5. An image of the exterior surface of Coupon 2 in the as-received condition.



Figure 6. An image of a representative edge of Coupon 2 in the as-received condition.



Figure 7. An image of the interior surface of Coupon 3 in the as-received condition.

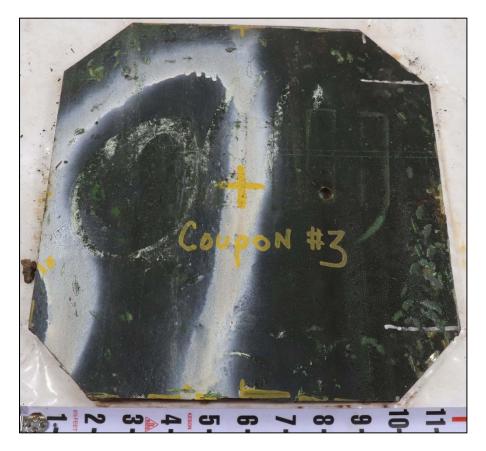


Figure 8. An image of the exterior surface of Coupon 3 in the as-received condition.



Figure 9. An image of a representative edge of Coupon 3 in the as-received condition.



Figure 10. An image of the interior surface of Coupon 5 in the as-received condition.



Figure 11. An image of the exterior surface of Coupon 5 in the as-received condition.



Figure 12. An image of a representative edge of Coupon 5 in the as-received condition.



Figure 13. An image of the interior surface of Coupon 6 in the as-received condition.



Figure 14. An image of the exterior surface of Coupon 6 in the as-received condition.



Figure 15. An image of a representative edge of Coupon 6 in the as-received condition.



Figure 16. An image of the interior surface of Coupon 7 in the as-received condition.



Figure 17. An image of the exterior surface of Coupon 7 in the as-received condition.

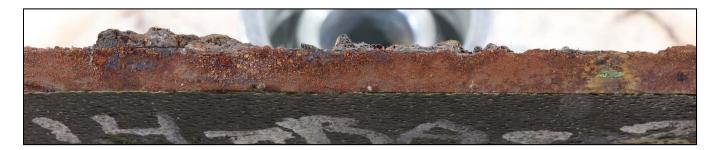


Figure 18. An image of a representative edge of Coupon 7 in the as-received condition.



Figure 19. An image of the interior surface of Coupon 8 in the as-received condition.



Figure 20. An image of the exterior surface of Coupon 8 in the as-received condition.

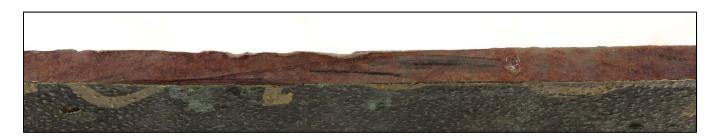


Figure 21. An image of a representative edge of Coupon 8 in the as-received condition.



Figure 22. An image of the interior surface of Coupon 10 in the as-received condition.



Figure 23. An image of the exterior surface of Coupon 10 in the as-received condition.



Figure 24. An image of a representative edge of Coupon 10 in the as-received condition.



Figure 25. An image of the interior surface of Coupon A1 in the as-received condition.



Figure 26. An image of the exterior surface of Coupon A1 in the as-received condition.



**Figure 27.** An image of a representative edge of Coupon A1 in the as-received condition.



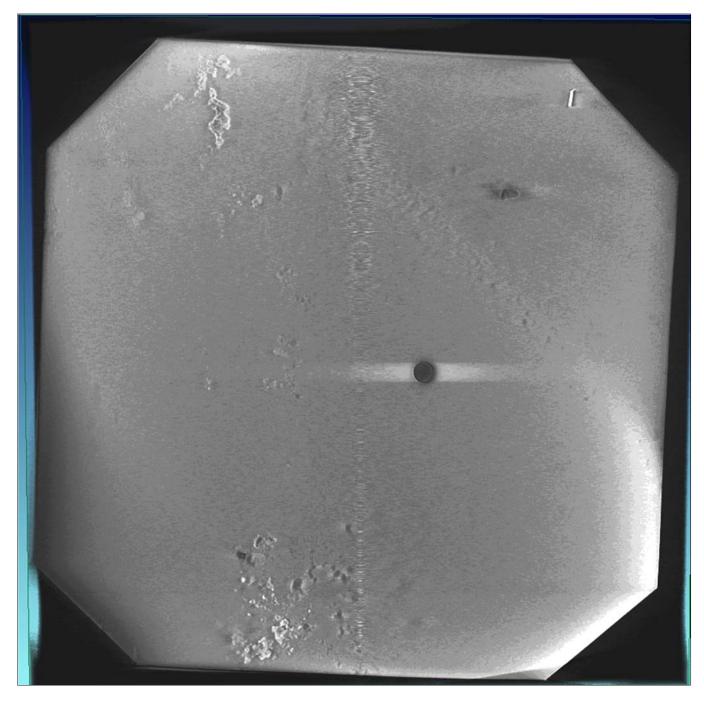
Figure 28. An image of the interior surface of Coupon A2 in the as-received condition.



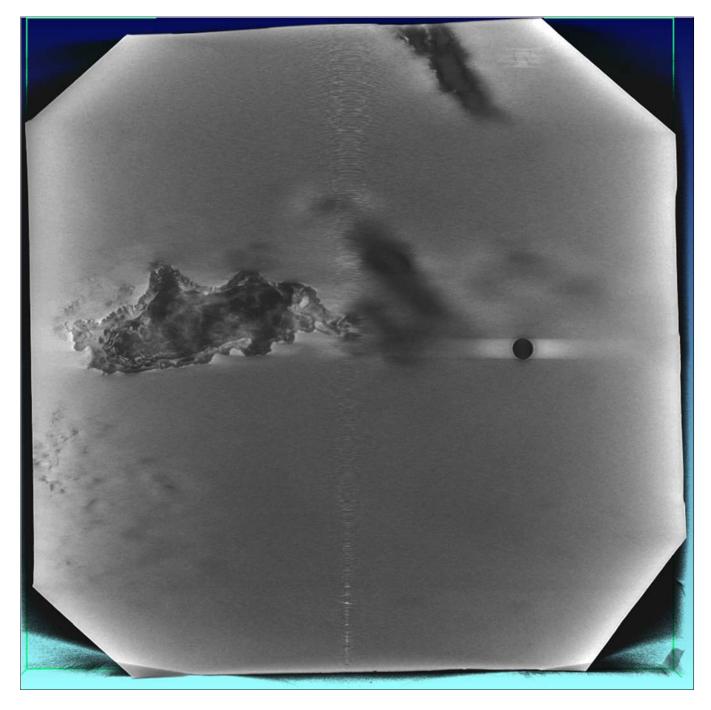
Figure 29. An image of the exterior surface of Coupon A2 in the as-received condition.



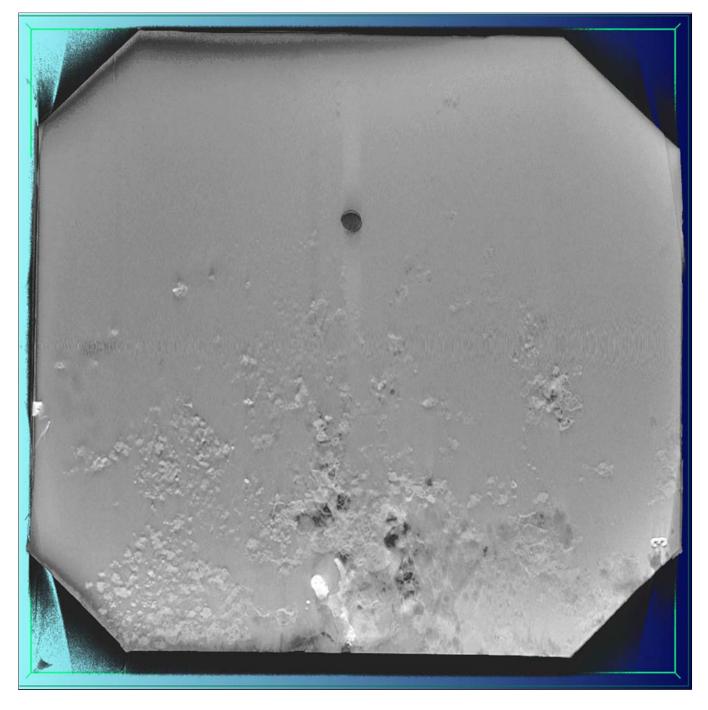
**Figure 30.** An image of a representative edge of Coupon A2 in the as-received condition.



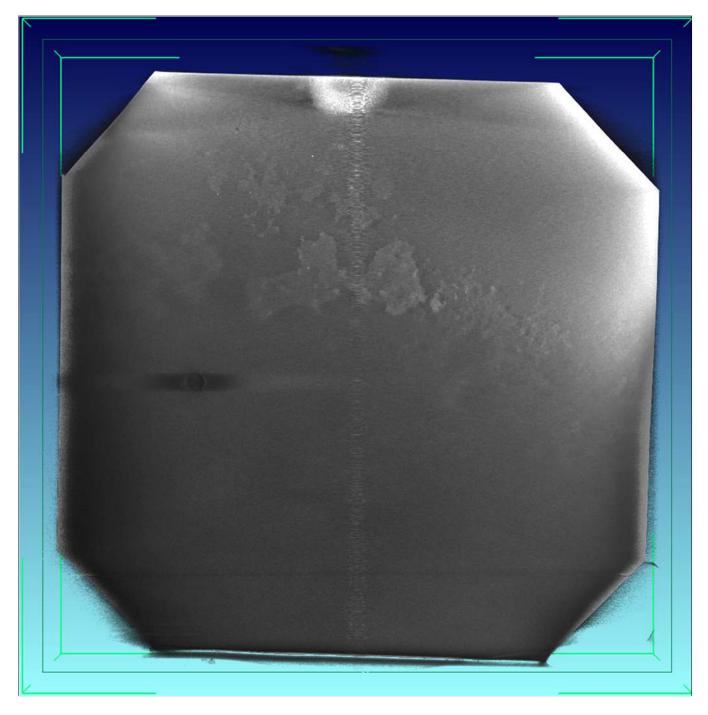
**Figure 31.** A representative screenshot of the CT scan performed on Coupon 1.



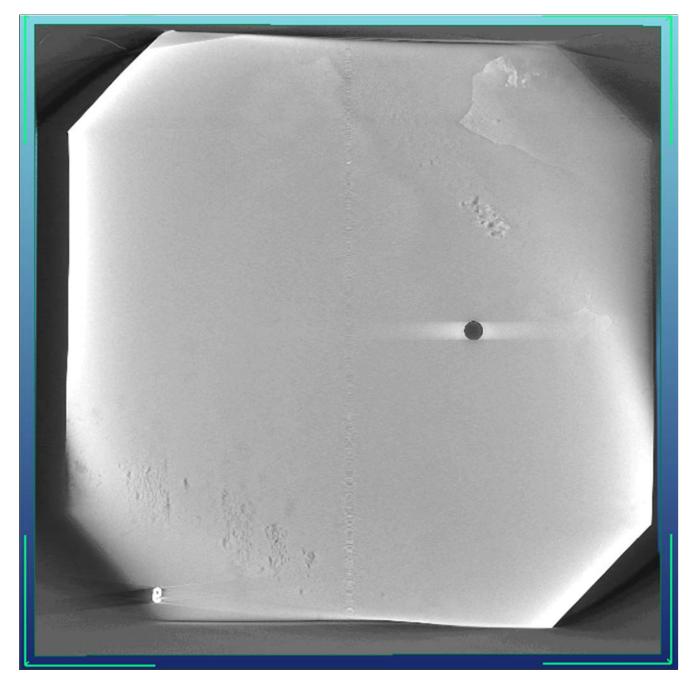
**Figure 32.** A representative screenshot of the CT scan performed on Coupon 2.



**Figure 33.** A representative screenshot of the CT scan performed on Coupon 3.



**Figure 34.** A representative screenshot of the CT scan performed on Coupon 5.



**Figure 35.** A representative screenshot of the CT scan performed on Coupon 6.

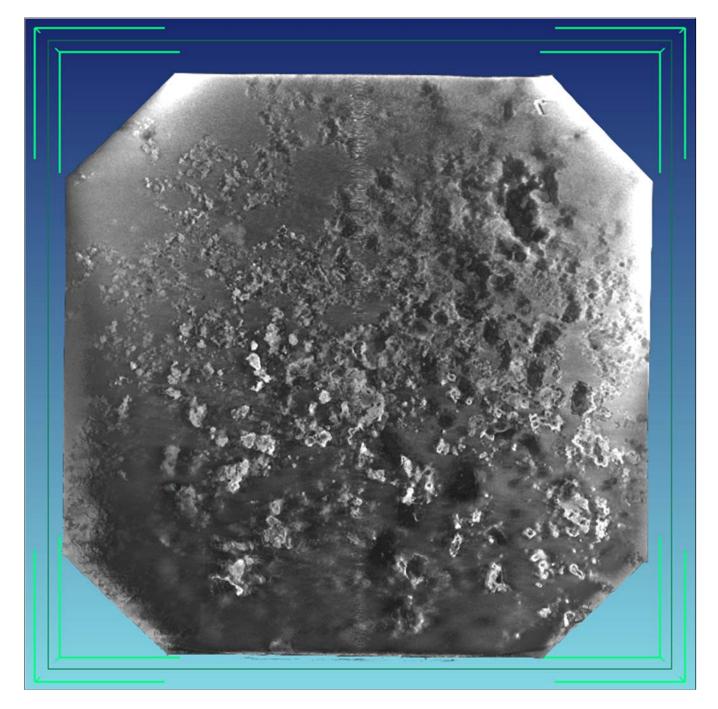
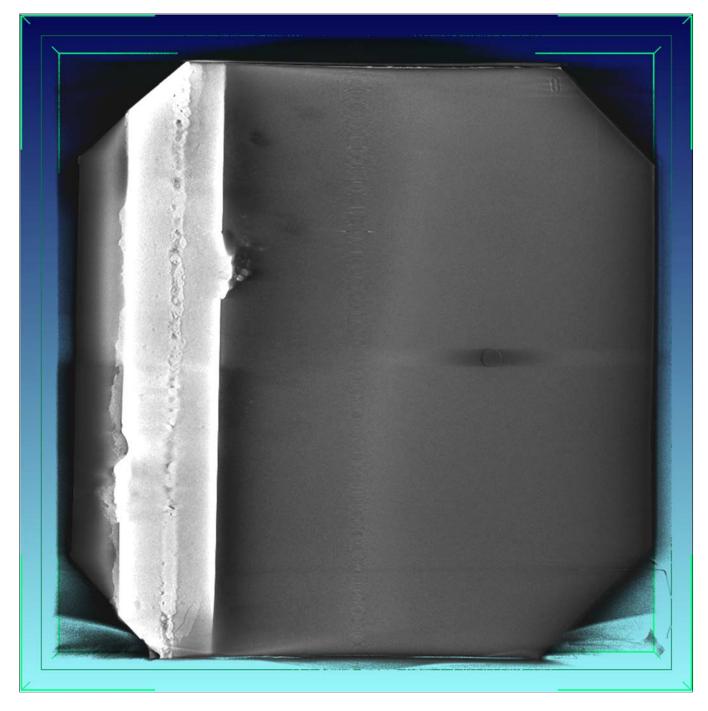
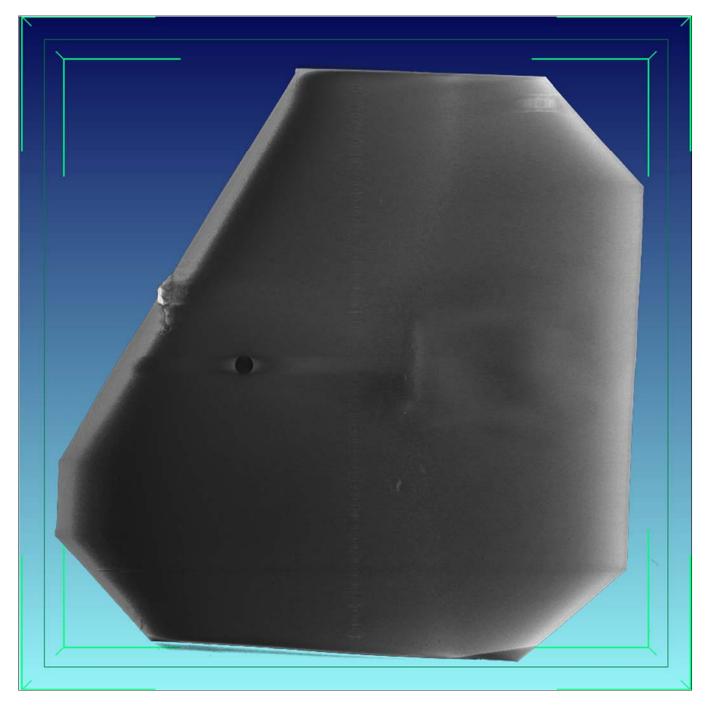


Figure 36. A representative screenshot of the CT scan performed on Coupon 7.



**Figure 37.** A representative screenshot of the CT scan performed on Coupon 8.



**Figure 38.** A representative screenshot of the CT scan performed on Coupon 10.

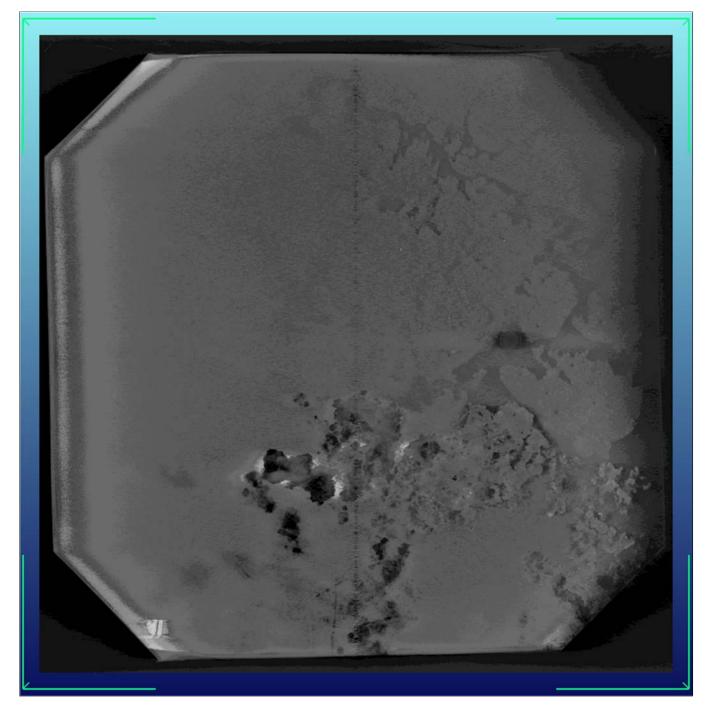
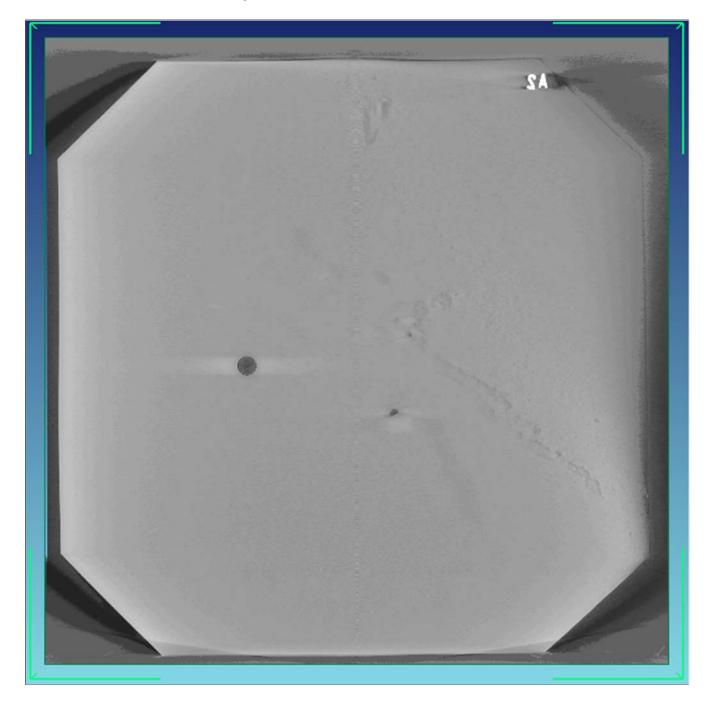
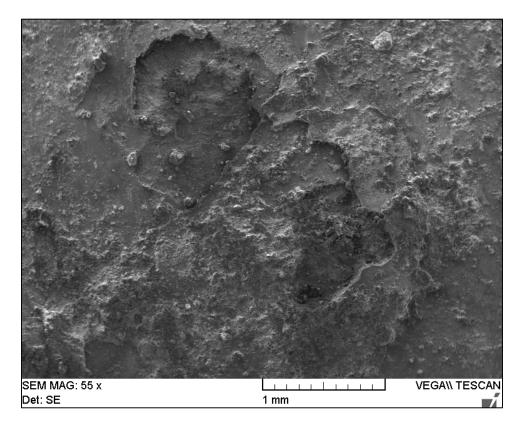


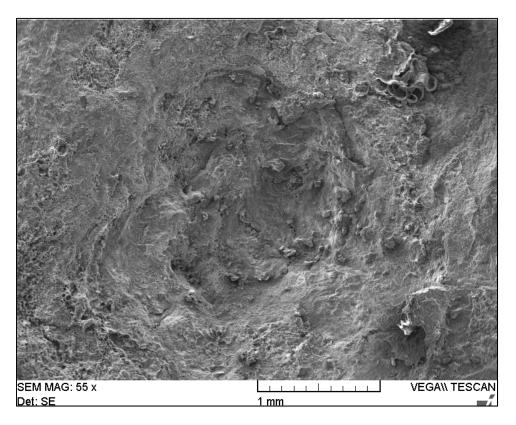
Figure 39. A representative screenshot of the CT scan performed on Coupon A1.



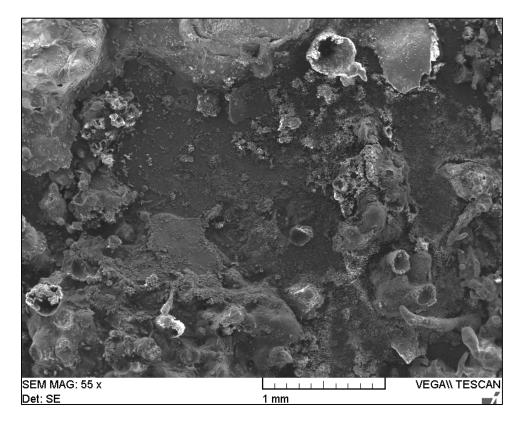
**Figure 40.** A representative screenshot of the CT scan performed on Coupon A2.



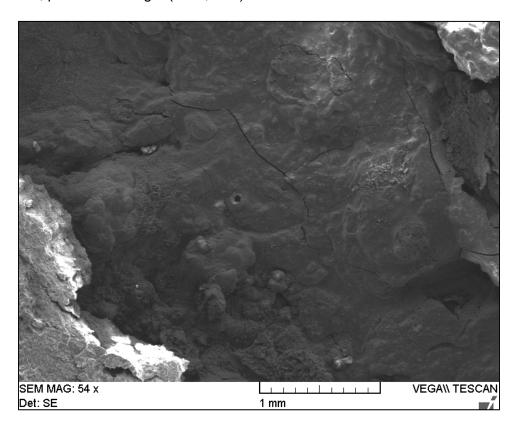
**Figure 41.** A representative SEM image of a pit on a specimen excised from Coupon 1 labeled as 1-1, prior to cleaning. (SEM, 32X)



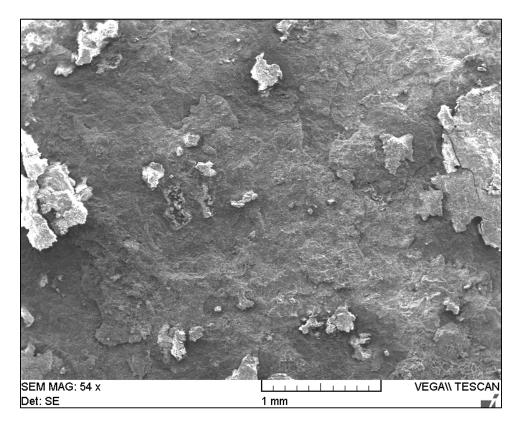
**Figure 42.** A representative SEM image of a pit on a specimen excised from Coupon 1 labeled as 1-2, prior to cleaning. (SEM, 32X)



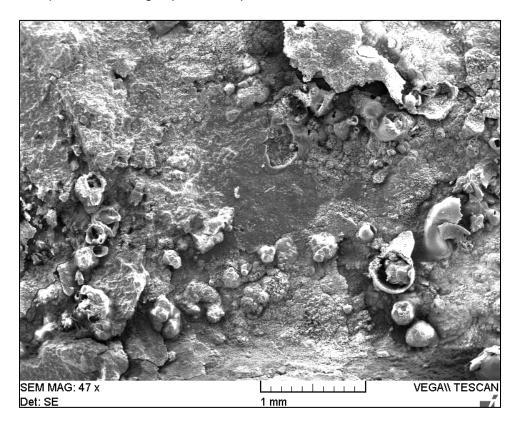
**Figure 43.** A representative SEM image of a pit on a specimen excised from Coupon 1 labeled as 1-3, prior to cleaning. (SEM, 32X)



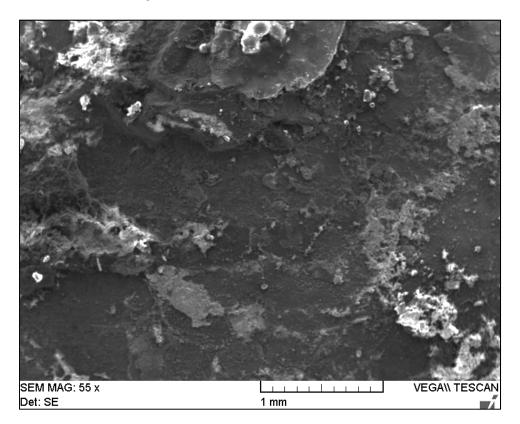
**Figure 44.** A representative SEM image of a pit on a specimen excised from Coupon 2 labeled as 2-1, prior to cleaning. (SEM, 32X)



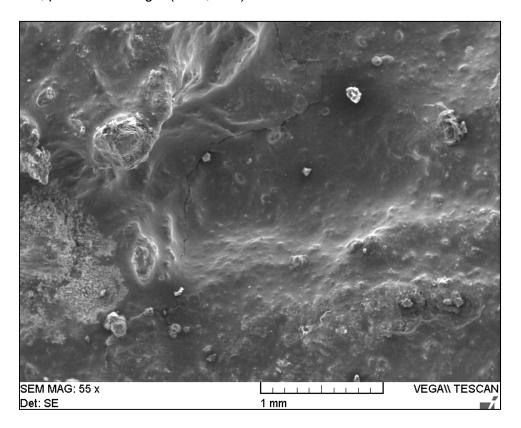
**Figure 45.** A representative SEM image of a pit on a specimen excised from Coupon 2 labeled as 2-2, prior to cleaning. (SEM, 32X)



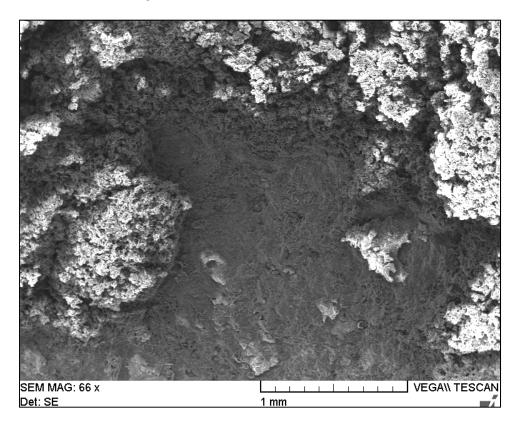
**Figure 46.** A representative SEM image of a pit on a specimen excised from Coupon 2 labeled as 2-3, prior to cleaning. (SEM, 28X)



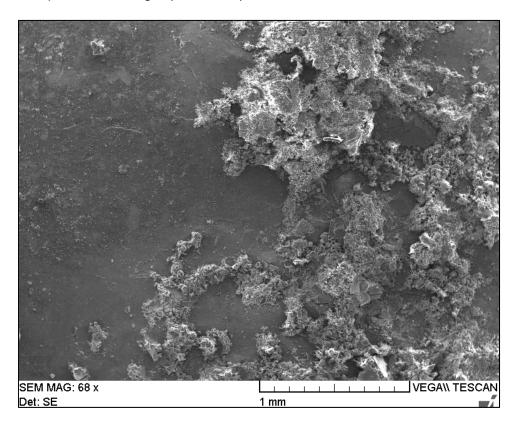
**Figure 47.** A representative SEM image of a pit on a specimen excised from Coupon 3 labeled as 3-1, prior to cleaning. (SEM, 32X)



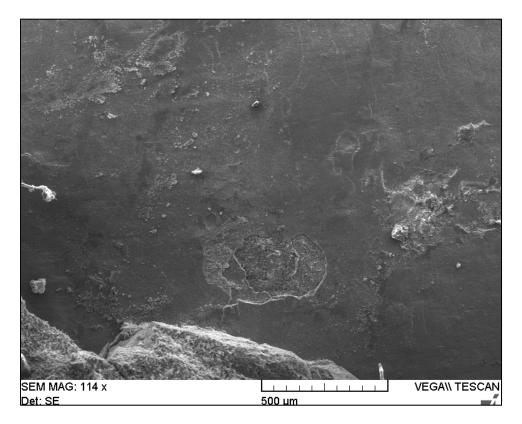
**Figure 48.** A representative SEM image of a pit on a specimen excised from Coupon 3 labeled as 3-2, prior to cleaning. (SEM, 32X)



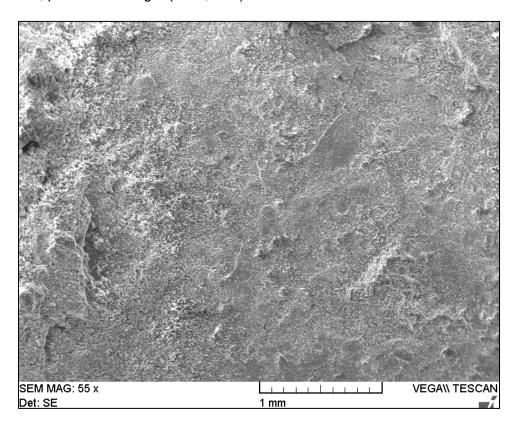
**Figure 49.** A representative SEM image of a pit on a specimen excised from Coupon 3 labeled as 3-3, prior to cleaning. (SEM, 39X)



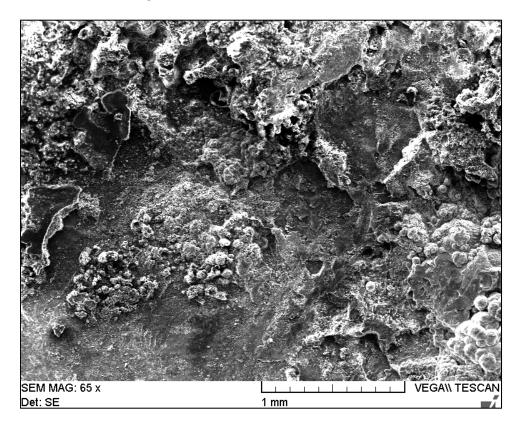
**Figure 50.** A representative SEM image of the specimen excised from Coupon 5 labeled as 5-1, prior to cleaning. No pits observed. (SEM, 40X)



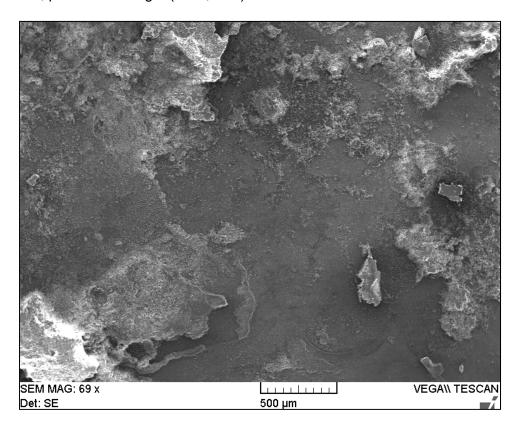
**Figure 51.** A representative SEM image of a pit on a specimen excised from Coupon 5 labeled as 5-2, prior to cleaning. (SEM, 67X)



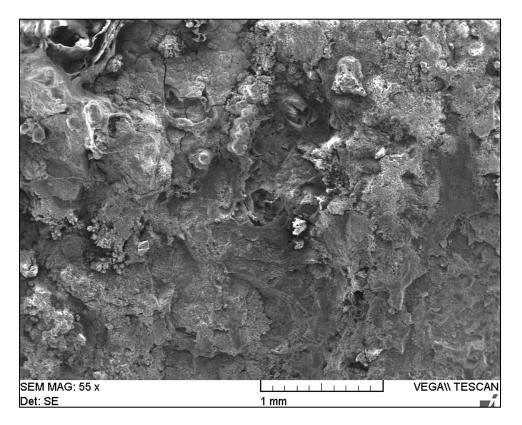
**Figure 52.** A representative SEM image of a pit on a specimen excised from Coupon 5 labeled as 5-3, prior to cleaning. (SEM, 32X)



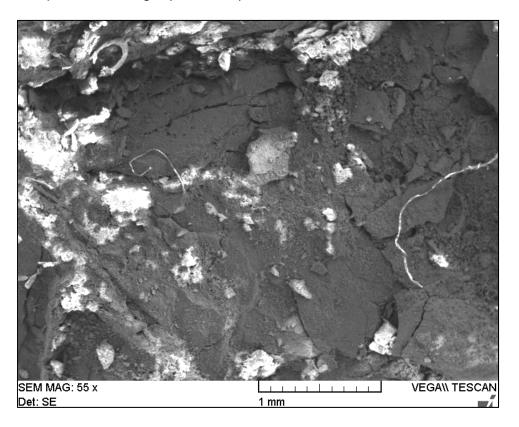
**Figure 53.** A representative SEM image of a pit on a specimen excised from Coupon 6 labeled as 6-1, prior to cleaning. (SEM, 38X)



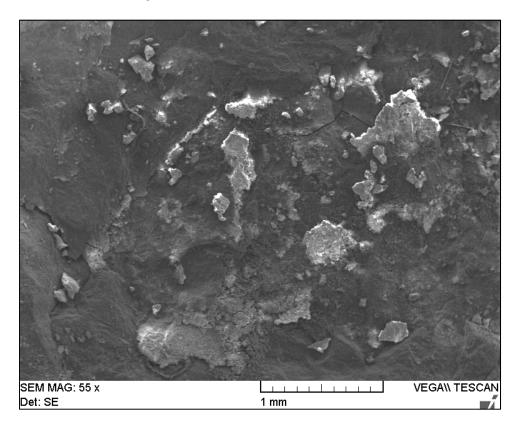
**Figure 54.** A representative SEM image the surface of a specimen excised from Coupon 6 labeled as 6-2, prior to cleaning. No pit was observed in this location. (SEM, 40X)



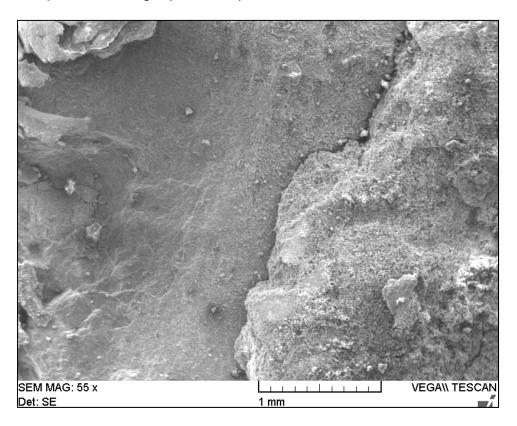
**Figure 55.** A representative SEM image of a pit on a specimen excised from Coupon 6 labeled as 6-3, prior to cleaning. (SEM, 32X)



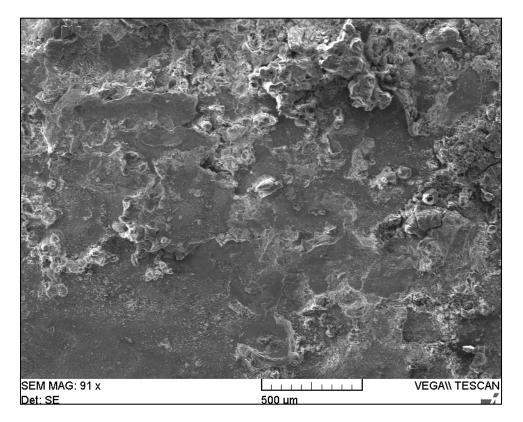
**Figure 56.** A representative SEM image of a pit on a specimen excised from Coupon 7 labeled as 7-1, prior to cleaning. (SEM, 32X)



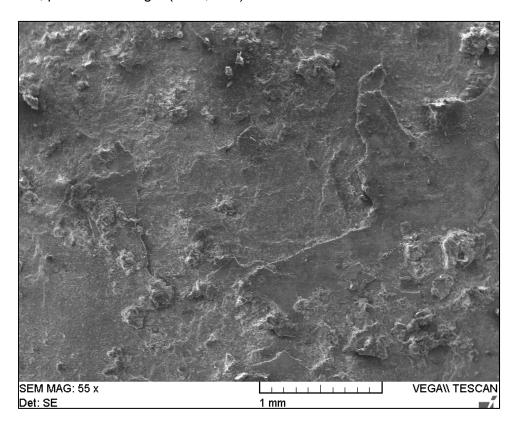
**Figure 57.** A representative SEM image of a pit on a specimen excised from Coupon 7 labeled as 7-2, prior to cleaning. (SEM, 32X)



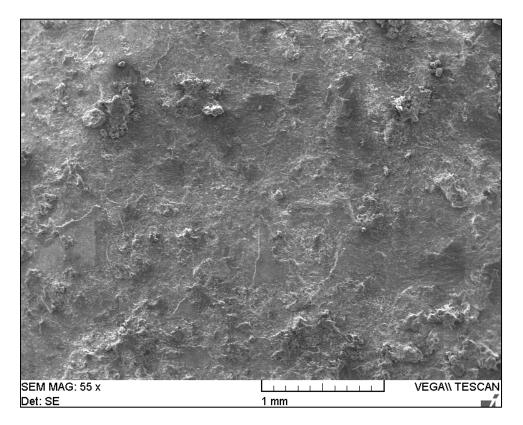
**Figure 58.** A representative SEM image of a pit on a specimen excised from Coupon 7 labeled as 7-3, prior to cleaning. (SEM, 32X)



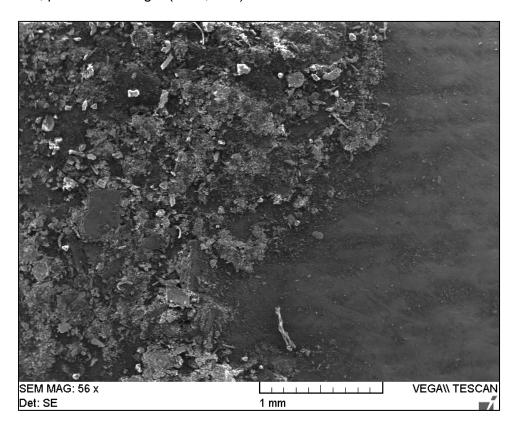
**Figure 59.** A representative SEM image of a pit on a specimen excised from Coupon 8 labeled as 8-1, prior to cleaning. (SEM, 53X)



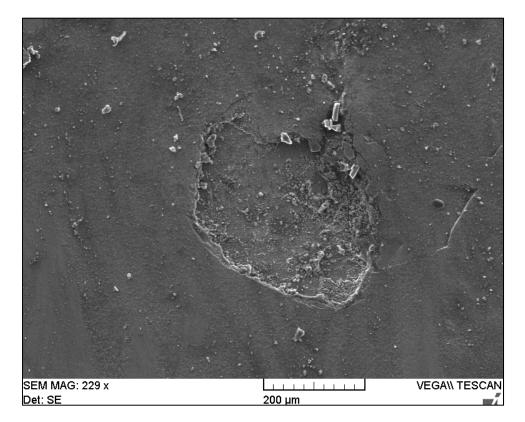
**Figure 60.** A representative SEM image of a pit on a specimen excised from Coupon 8 labeled as 8-2, prior to cleaning. (SEM, 33X)



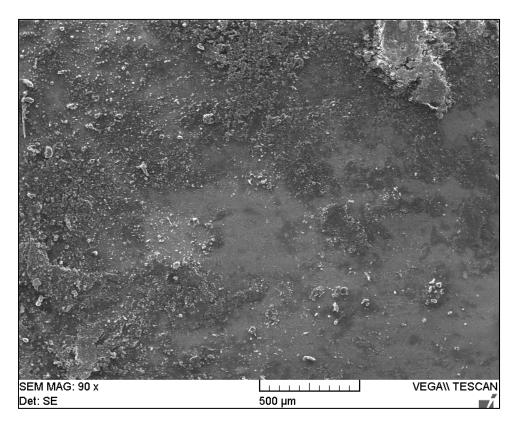
**Figure 61.** A representative SEM image of a pit on a specimen excised from Coupon 8 labeled as 8-3, prior to cleaning. (SEM, 33X)



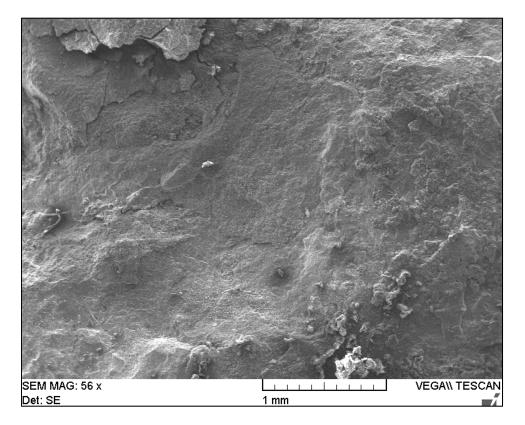
**Figure 62.** A representative SEM image of the surface of a specimen excised from Coupon 10 labeled as 10-1, prior to cleaning. No pit was observed in this location. (SEM, 33X)



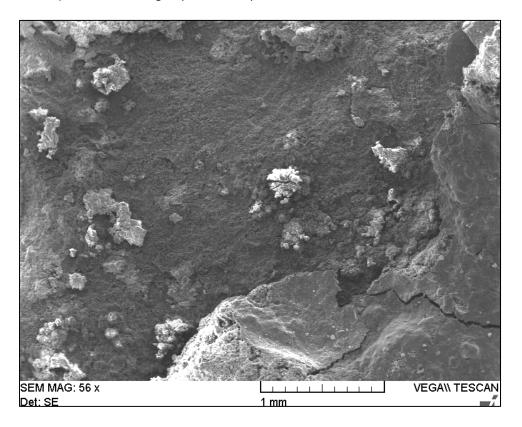
**Figure 63.** A representative SEM image of a pit on a specimen excised from Coupon 10 labeled as 10-2, prior to cleaning. (SEM, 135X)



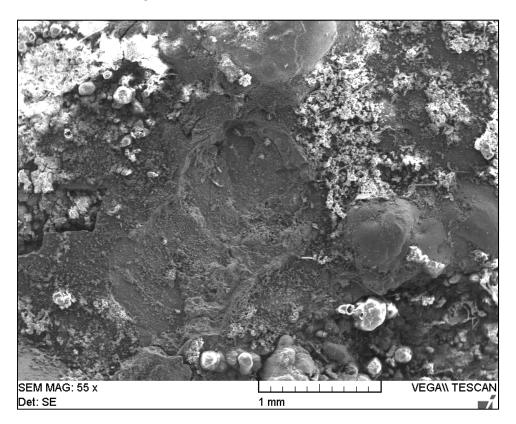
**Figure 64.** A representative SEM image of the surface on a specimen excised from Coupon 10 labeled as 10-3, prior to cleaning. No pit was observed in this location. (SEM, 53X)



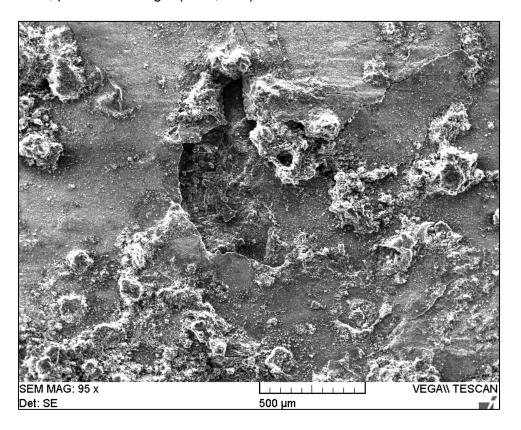
**Figure 65.** A representative SEM image of a pit on a specimen excised from Coupon A-1 labeled as A1-1, prior to cleaning. (SEM, 33X)



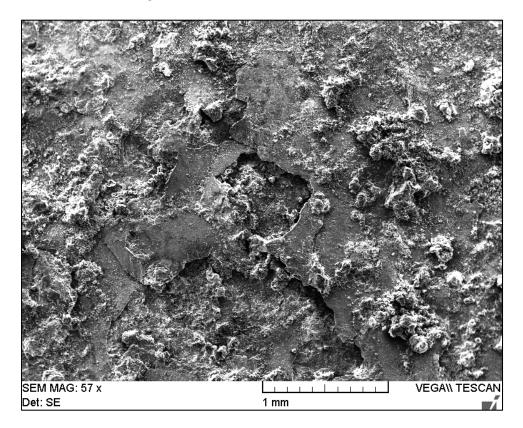
**Figure 66.** A representative SEM image of a pit on a specimen excised from Coupon A-1 labeled as A1-2, prior to cleaning. (SEM, 33X)



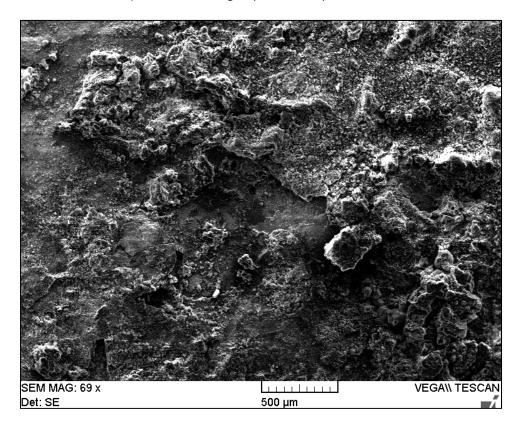
**Figure 67.** A representative SEM image of a pit on a specimen excised from Coupon A-1 labeled as A1-3, prior to cleaning. (SEM, 32X)



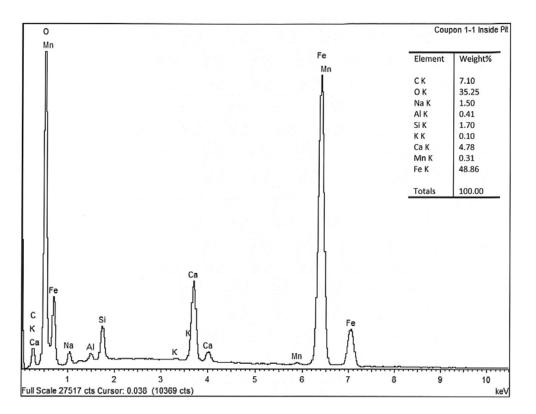
**Figure 68.** A representative SEM image of a pit on a specimen excised from Coupon A-2 labeled as A2-1, prior to cleaning. (SEM, 56X)



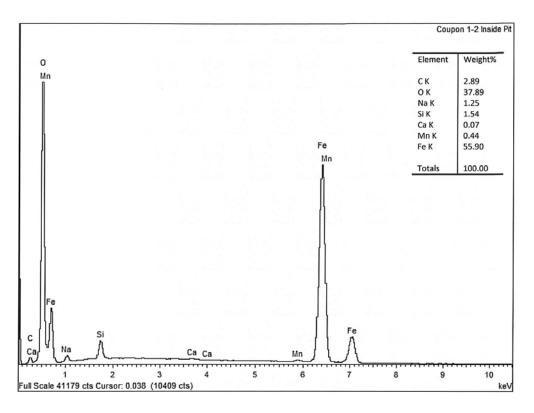
**Figure 69.** A representative SEM image of a possible pit on a specimen excised from Coupon A-2 labeled as A2-2, prior to cleaning. (SEM, 33X)



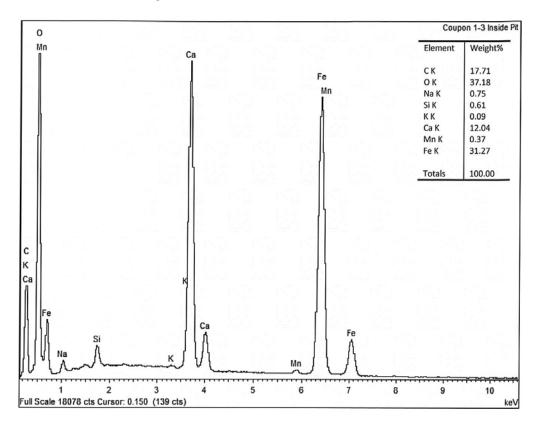
**Figure 70.** A representative SEM image of a possible pit on a specimen excised from Coupon A-2 labeled as A2-3, prior to cleaning. (SEM, 40X)



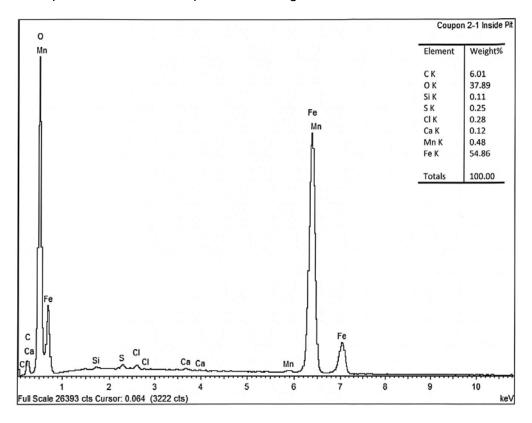
**Figure 71.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 1 labeled as 1-1, prior to cleaning.



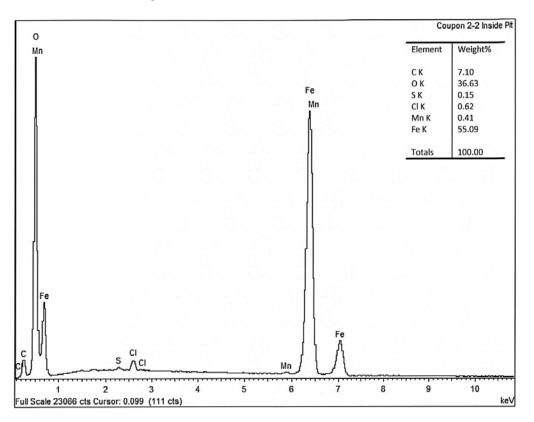
**Figure 72.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 1 labeled as 1-2, prior to cleaning.



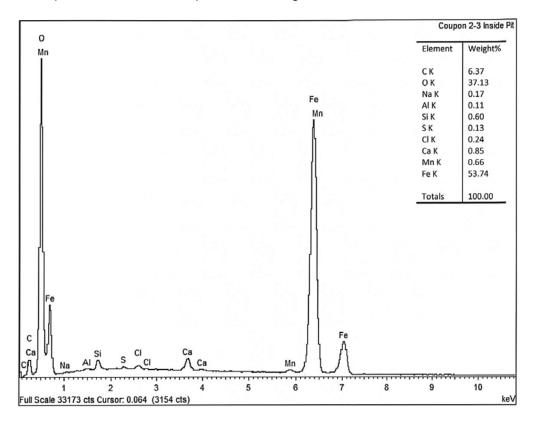
**Figure 73.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 1 labeled as 1-3, prior to cleaning.



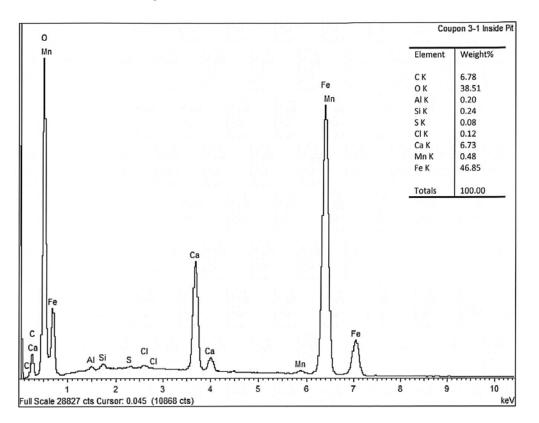
**Figure 74.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 2 labeled as 2-1, prior to cleaning.



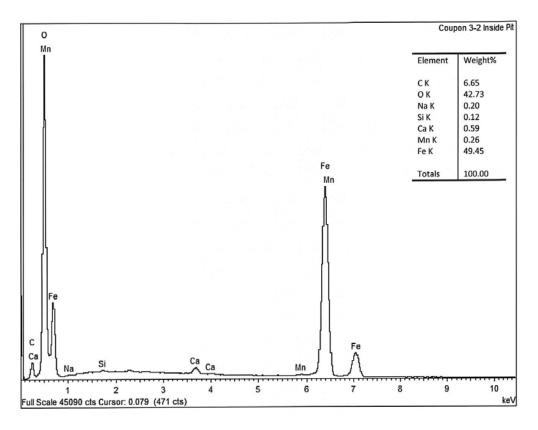
**Figure 75.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 2 labeled as 2-2, prior to cleaning.



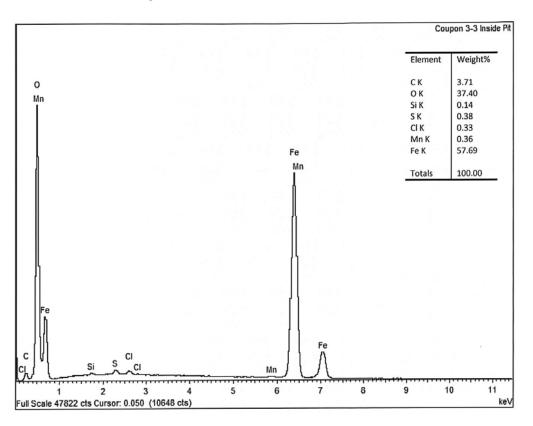
**Figure 76.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 2 labeled as 2-3, prior to cleaning.



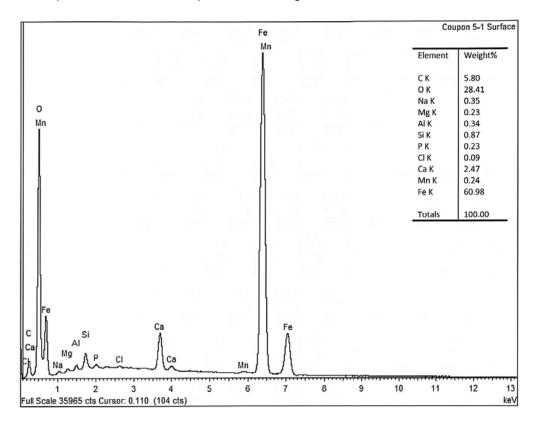
**Figure 77.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 3 labeled as 3-1, prior to cleaning.



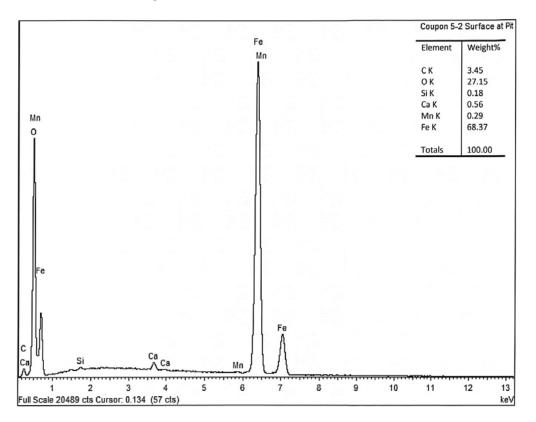
**Figure 78.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 3 labeled as 3-2, prior to cleaning.



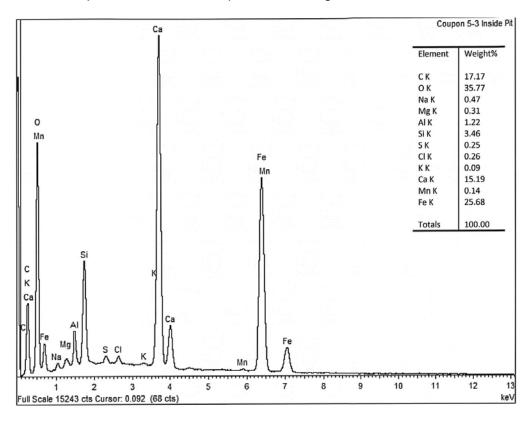
**Figure 79.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 3 labeled as 3-3, prior to cleaning.



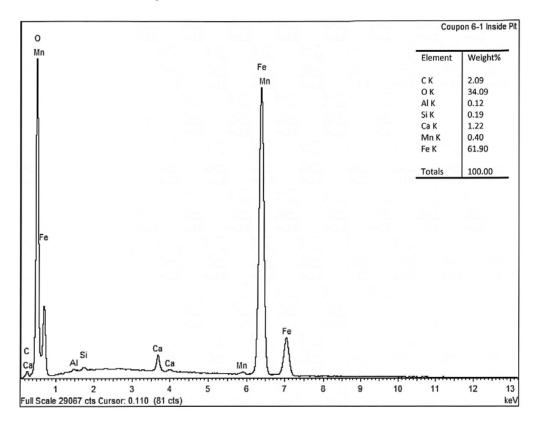
**Figure 80.** A representative EDS spectrum generated from the nominal surface of a specimen excised from Coupon 5 labeled as 5-1, prior to cleaning. No pit observed.



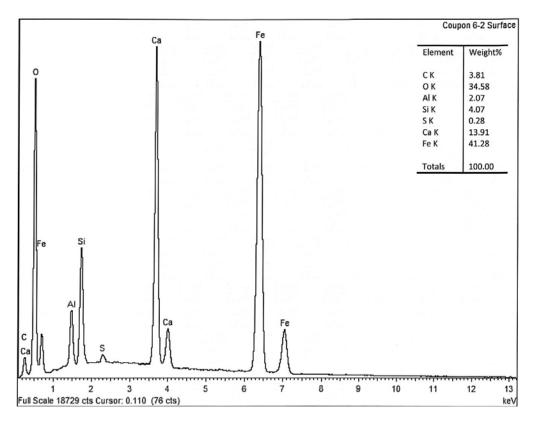
**Figure 81.** A representative EDS spectrum generated from the surface at a pit on a specimen excised from Coupon 5 labeled as 5-2, prior to cleaning.



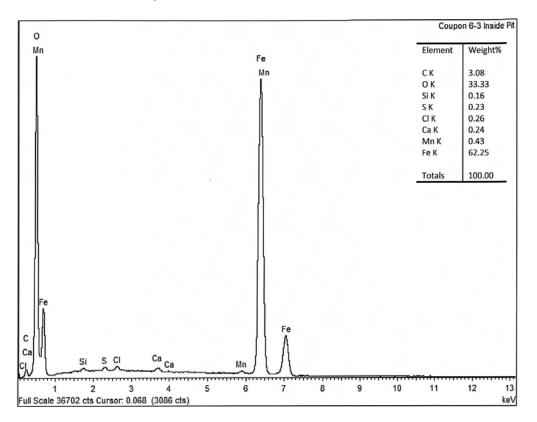
**Figure 82.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 5 labeled as 5-3, prior to cleaning.



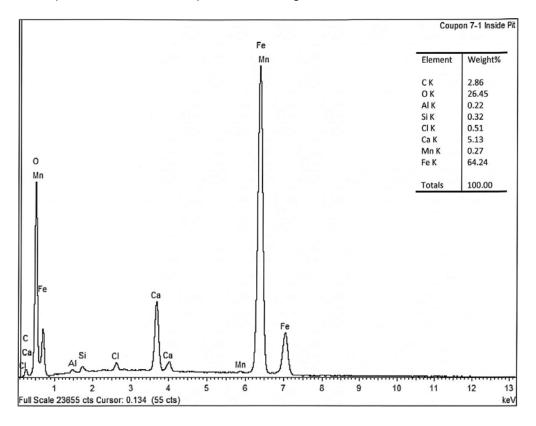
**Figure 83.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 6 labeled as 6-1, prior to cleaning.



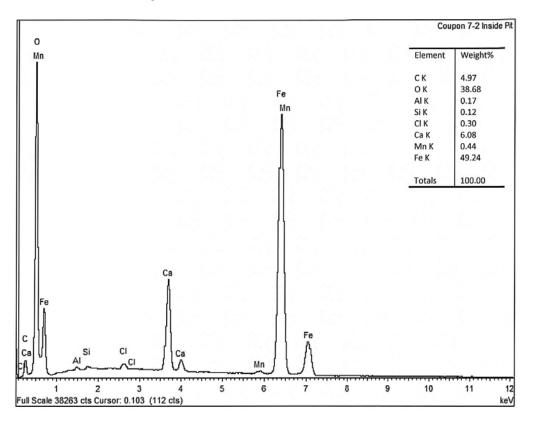
**Figure 84.** A representative EDS spectrum generated from the surface of a specimen excised from Coupon 6 labeled as 6-2, prior to cleaning.



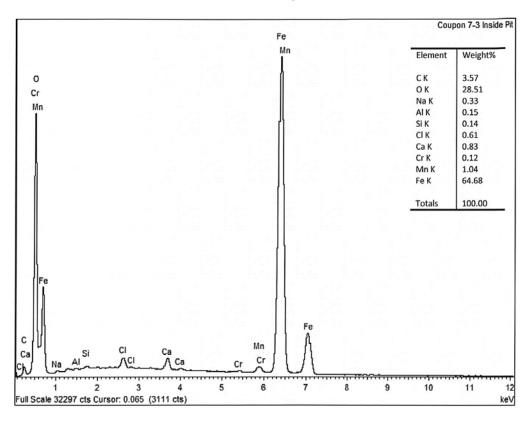
**Figure 85.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 6 labeled as 6-3, prior to cleaning.



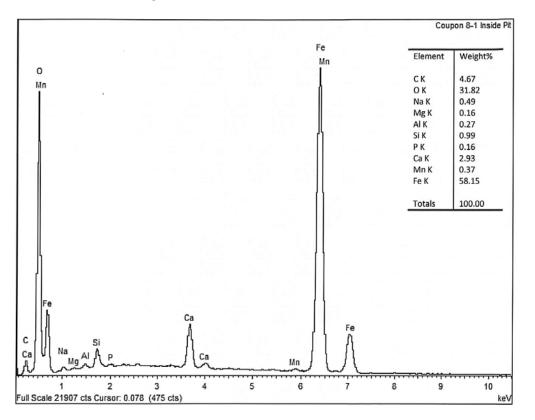
**Figure 86.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 7 labeled as 7-1, prior to cleaning.



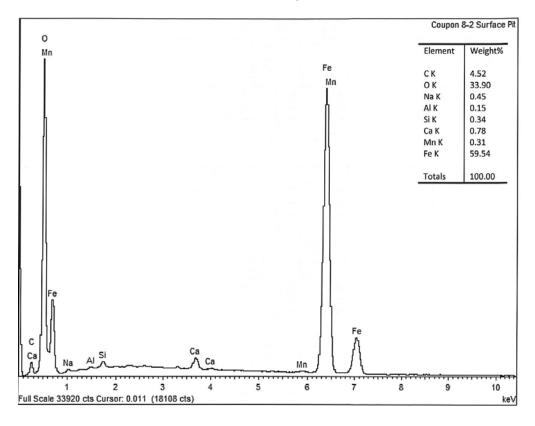
**Figure 87.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 7 labeled as 7-2, prior to cleaning.



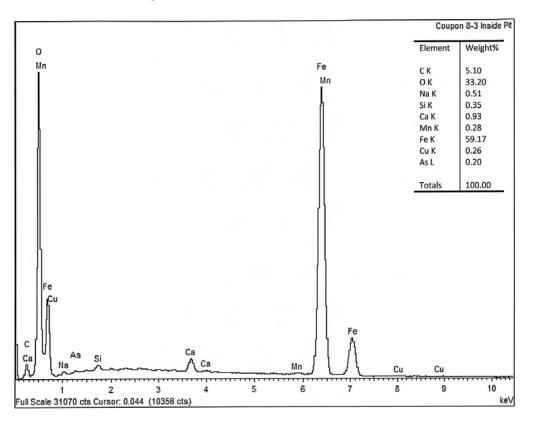
**Figure 88.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 7 labeled as 7-3, prior to cleaning.



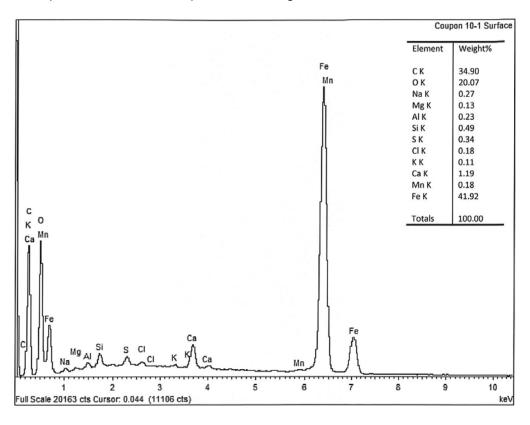
**Figure 89.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 8 labeled as 8-1, prior to cleaning.



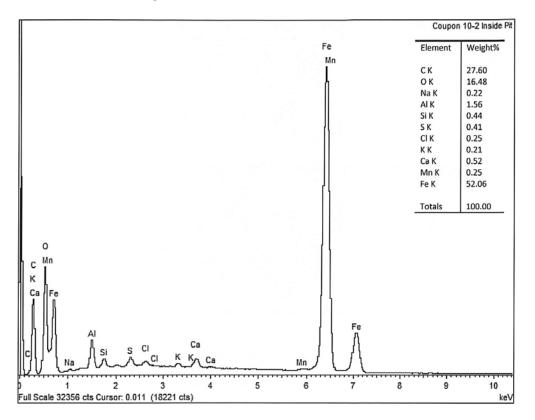
**Figure 90.** A representative EDS spectrum generated from the surface of a specimen excised from Coupon 8 labeled as 8-2, prior to cleaning.



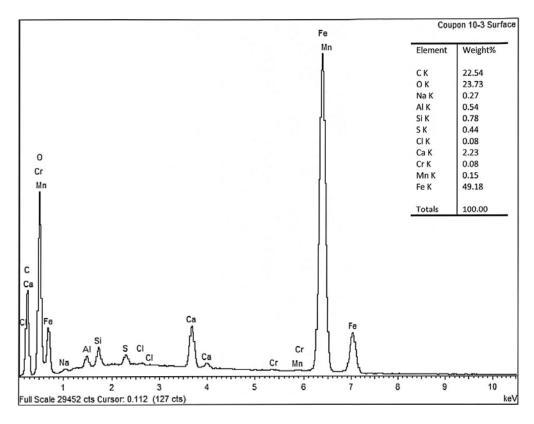
**Figure 91.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 8 labeled as 8-3, prior to cleaning.



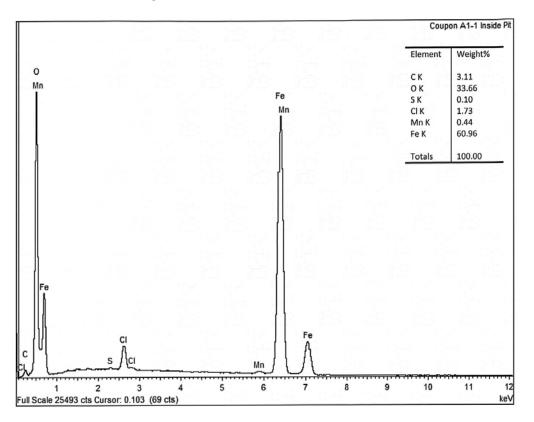
**Figure 92.** A representative EDS spectrum generated from the surface of a specimen excised from Coupon 10 labeled as 10-1, prior to cleaning.



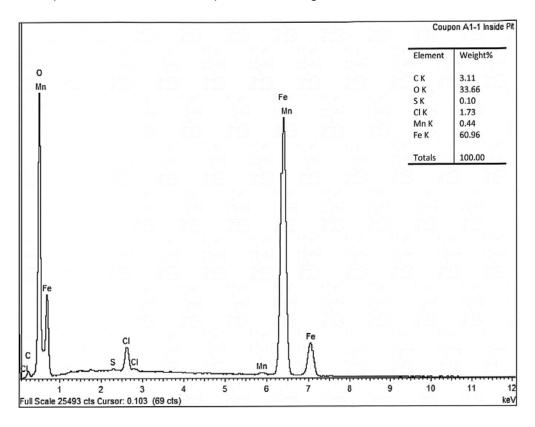
**Figure 93.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon 10 labeled as 10-2, prior to cleaning.



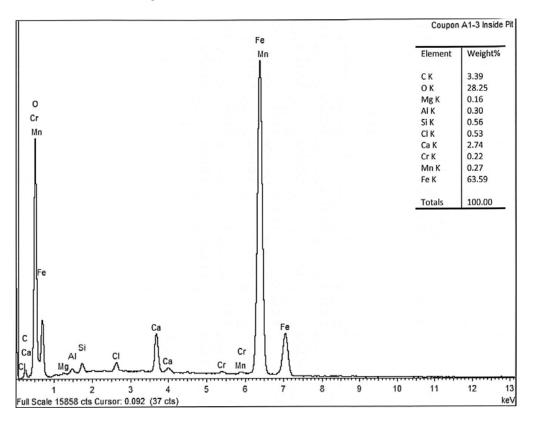
**Figure 94.** A representative EDS spectrum generated from the surface of a specimen excised from Coupon 10 labeled as 10-3, prior to cleaning.



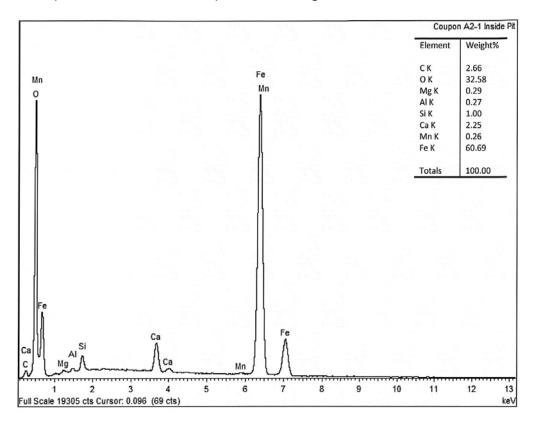
**Figure 95.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A1 labeled as A1-1, prior to cleaning.



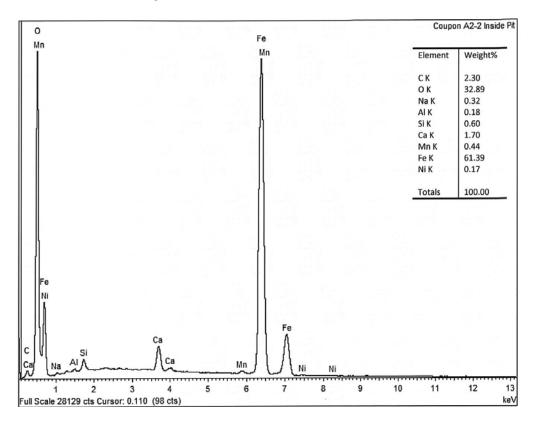
**Figure 96.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A1 labeled as A1-2, prior to cleaning.



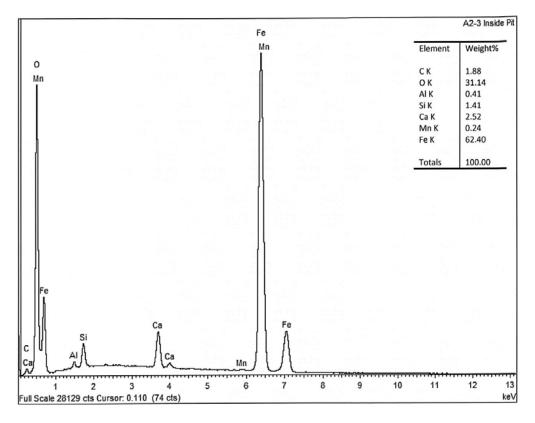
**Figure 97.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A1 labeled as A1-3, prior to cleaning.



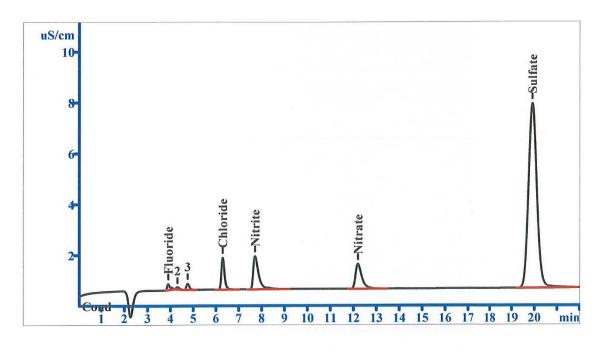
**Figure 98.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A2 labeled as A2-1, prior to cleaning.



**Figure 99.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A2 labeled as A2-2, prior to cleaning.



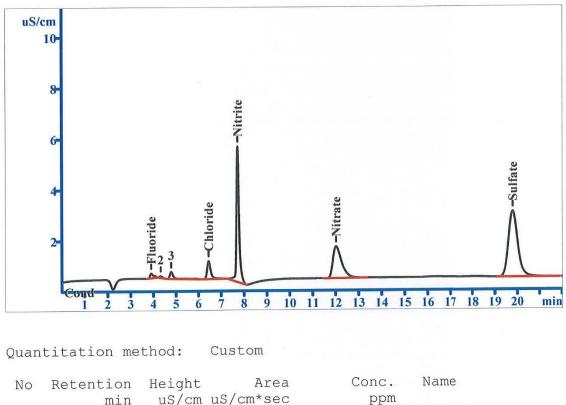
**Figure 100.** A representative EDS spectrum generated from of a pit on a specimen excised from Coupon A2 labeled as A2-3, prior to cleaning.



No	Retention	Height	Area	Conc.	Name
	min	uS/cm	uS/cm*sec	ppm	
1	3.91	0.24	2.148	10.847	Fluoride
4	6.31	1.27	12.457	82.132	Chloride
5	7.74	1.31	22.385	281.793	Nitrite
6	12.22	0.99	19.356	273.382	Nitrate
7	19.92	7.27	188.802	1518.397	Sulfate
 5	22.00	11.08	245.149	2166.551	

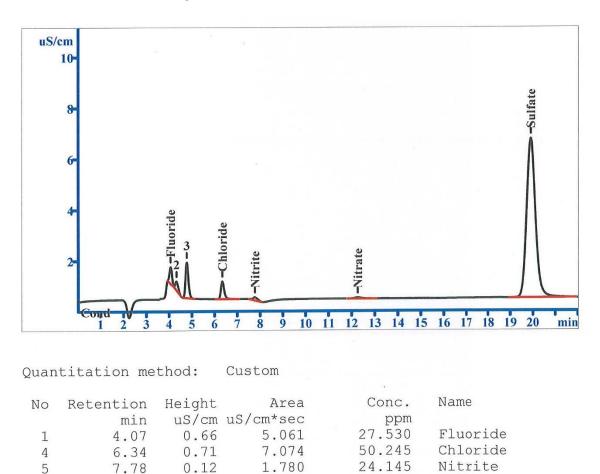
Quantitation method: Custom

Figure 101. The ion chromatogram generated from a concrete sample removed from Coupon 1.



	INO	VECENCION	ILETAILC	III CU	00110.	
		min	uS/cm	uS/cm*sec	ppm	
	1	3.91	0.18	2.024	11.408	Fluoride
	4	6.44	0.72	7.995	58.860	Chloride
	5	7.72	5.34	42.354	595.340	Nitrite
	6	12.04	1.26	33.947	535.394	Nitrate
	7	19.80	2.61	76.162	683.949	Sulfate
0	5	22.00	10.11	162.481	1884.952	

Figure 102. The ion chromatogram generated from a concrete sample removed from Coupon 2.



5	22.00	7.81	188.083	1617.158	

0.05

6.28

12.28

19.91

Figure 103. The ion chromatogram generated from a concrete sample removed from Coupon 3.

0.957

173.210

14.555

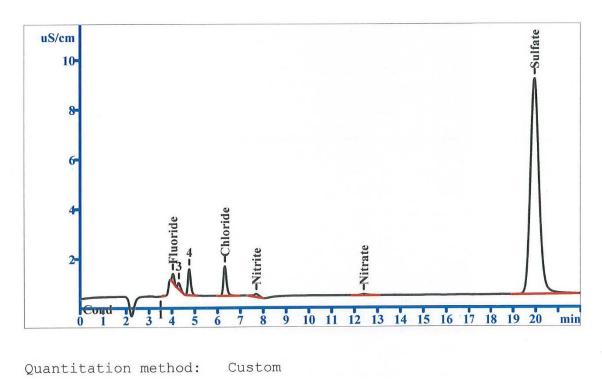
1500.682

Nitrate

Sulfate

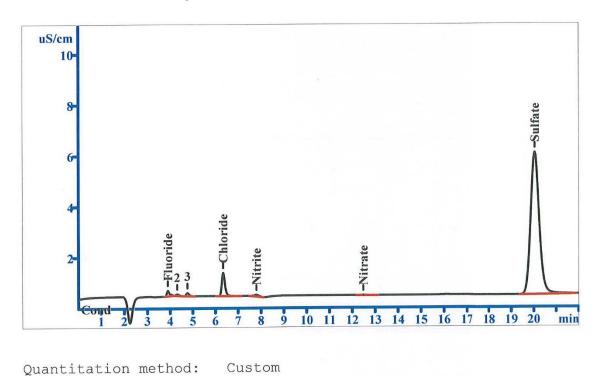
6

7



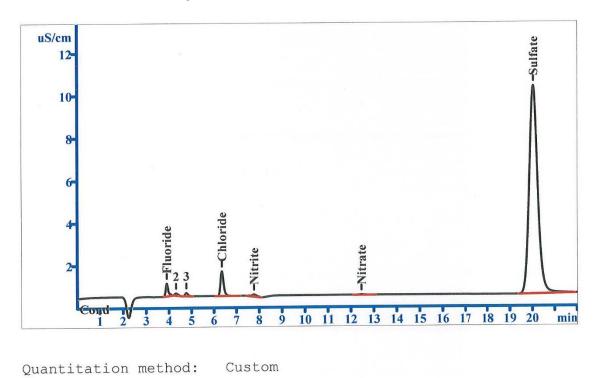
No	Retention	Height	Area		Conc.	Name
	min	uS/cm	uS/cm*sec		ppm	
2	4.06	0.35	2.415		12.764	Fluoride
5	6.35	1.20	11.646		80.382	Chloride
6	7.72	0.12	1.989		26.215	Nitrite
7	12.41	0.04	0.804		11.895	Nitrate
8	19.97	8.69	235.110	19	79.422	Sulfate
5	22.00	10.41	251.964	21	10.679	

Figure 104. The ion chromatogram generated from a concrete sample removed from Coupon 5.



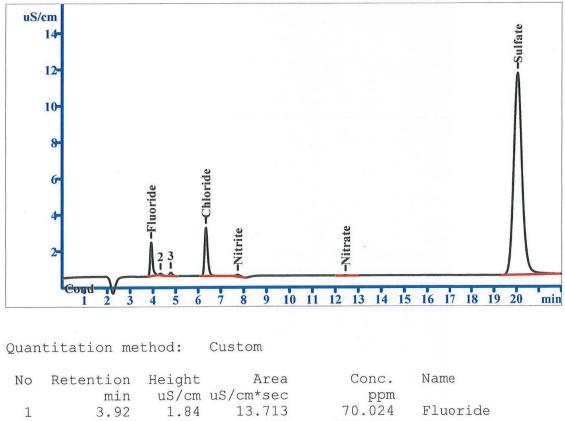
No	Retention	Height	Area	Conc.	Name
	min	uS/cm	uS/cm*sec	ppm	
1	3.92	0.21	1.820	12.711	Fluoride
4	6.35	0.91	8.882	81.024	Chloride
5	7.80	0.07	0.999	17.394	Nitrite
6	12.48	0.01	0.154	3.004	Nitrate
7	20.07	5.59	149.208	1660.246	Sulfate
5	22.00	6.79	161.062	1774.379	

Figure 105. The ion chromatogram generated from a concrete sample removed from Coupon 6.



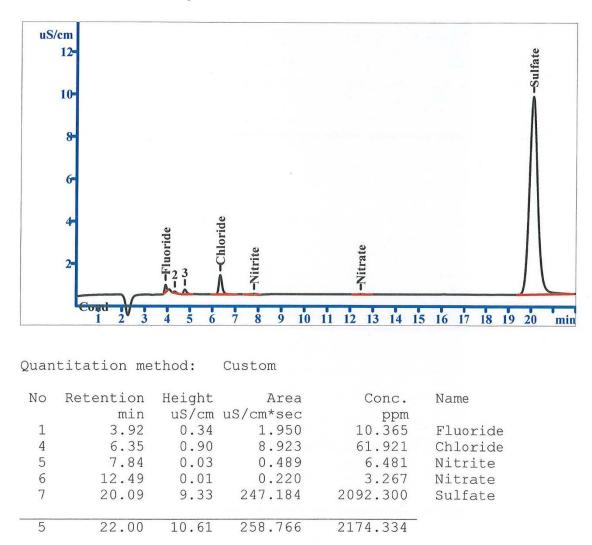
No	Retention	Height	Area	Conc.	Name
	min	uS/cm	uS/cm*sec	ppm	
1	3.92	0.60	4.635	24.567	Fluoride
4	6.35	1.16	11.342	78.508	Chloride
5	7.78	0.10	1.492	19.715	Nitrite
6	12.47	0.02	0.348	5.162	Nitrate
7	20.06	9.81	259.442	2190.390	Sulfate
5	22.00	11.69	277.260	2318.341	

Figure 106. The ion chromatogram generated from a concrete sample removed from Coupon 7.

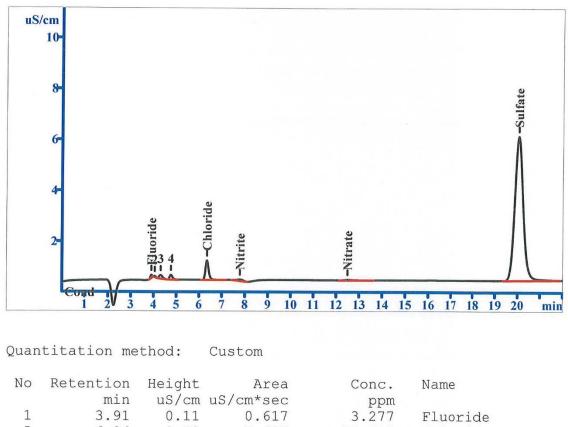


		up/ cm	ub/ on bee	[ [ ] Lorre	
1	3.92	1.84	13.713	70.024	Fluoride
4	6.34	2.65	25.697	171.359	Chloride
5	7.75	0.10	1.421	18.089	Nitrite
6	12.47	0.02	0.309	4.417	Nitrate
7	20.05	11.12	294.084	2392.060	Sulfate
5	22.00	15.73	335.223	2655.950	

Figure 107. The ion chromatogram generated from a concrete sample removed from Coupon 8.

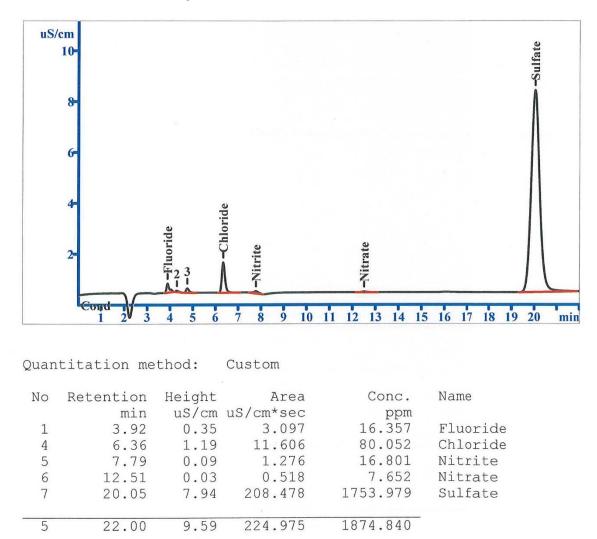


**Figure 108.** The ion chromatogram generated from a concrete sample removed from Coupon 10.

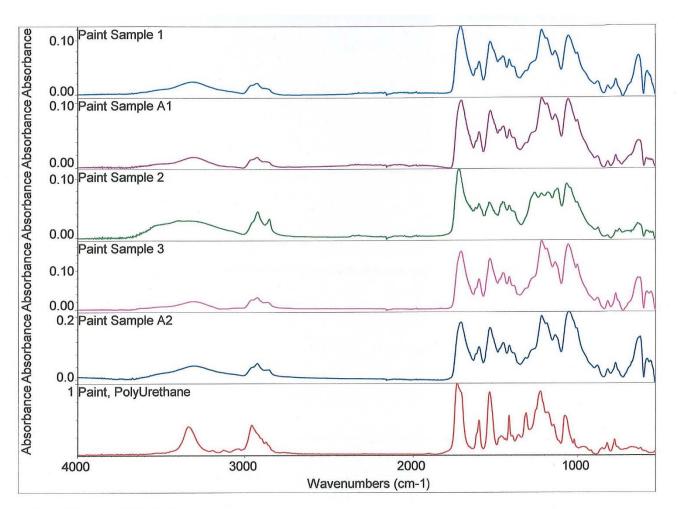


	11111	us/cm t	15/CIII^SeC	ppm	
1	3.91	0.11	0.617	3.277	Fluoride
5	6.36	0.78	7.603	52.710	Chloride
6	7.82	0.06	1.132	14.978	Nitrite
7	12.50	0.02	0.412	6.112	Nitrate
8	20.06	5.68	149.697	1265.856	Sulfate
 5	22.00	6.64	159.460	1342.933	

Figure 109. The ion chromatogram generated from a concrete sample removed from Coupon A1.



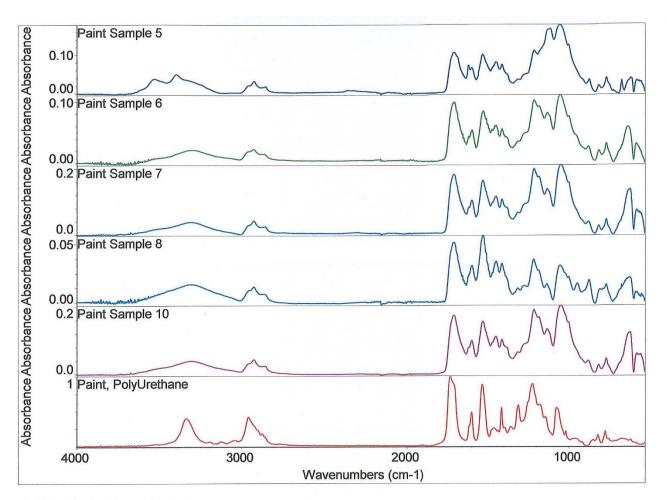
**Figure 110.** The ion chromatogram generated from a concrete sample removed from Coupon A2.



Wed Oct 03 15:47:52 2018 (GMT-04:00)

ASTM E1252-98(13)e1 - IMR Test Labs - FTIR - CSDR#E143 - User: H Marini

**Figure 111.** The FTIR spectra generated from a paint samples removed from the exterior surfaces of Coupons 1, A1, 2, A2 and 3 the best matches from the spectral FTIR library of standards for polyurethane enamel.



Wed Oct 03 15:46:55 2018 (GMT-04:00)

ASTM E1252-98(13)e1 - IMR Test Labs - FTIR - CSDR#E143 - User: H Marini

**Figure 112.** The FTIR spectra generated from a paint samples removed from the exterior surfaces of Coupons 5, 6, 7, 8 and 10 the best matches from the spectral FTIR library of standards for polyurethane enamel.

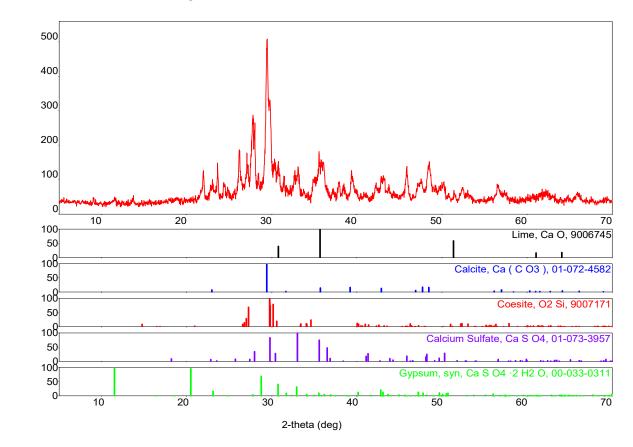
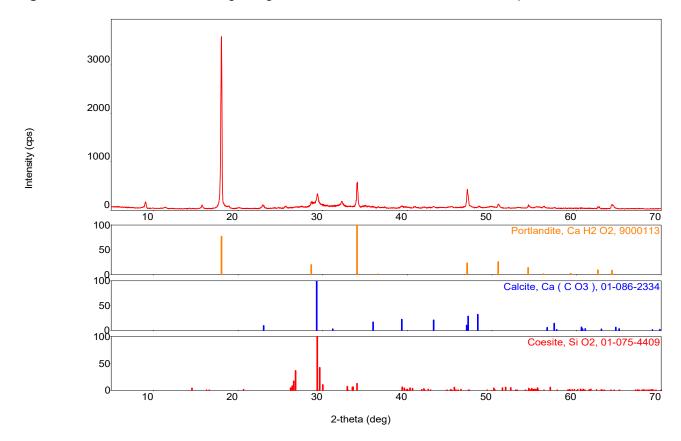
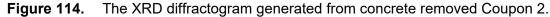
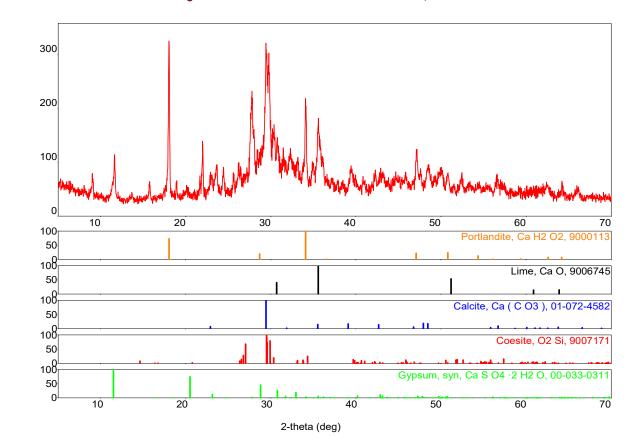


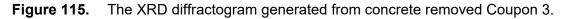
Figure 113. The XRD diffractogram generated from concrete removed Coupon 1.

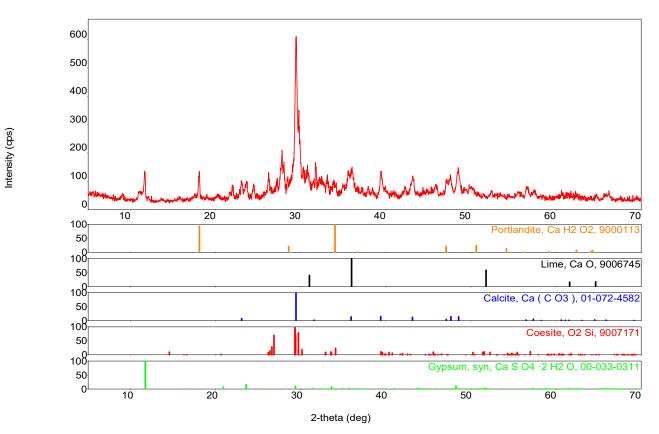


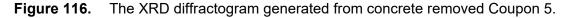




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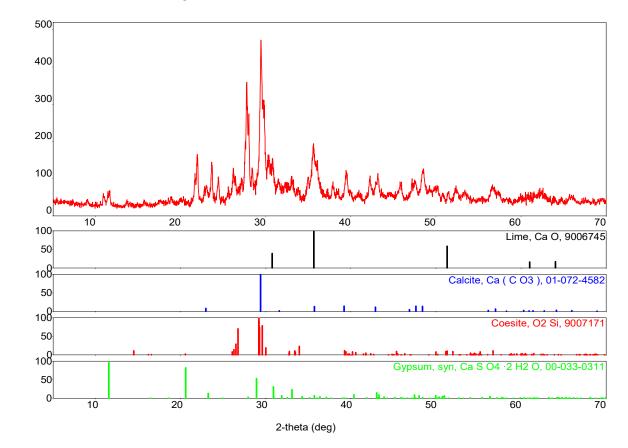


Figure 117. The XRD diffractogram generated from concrete removed Coupon 6.

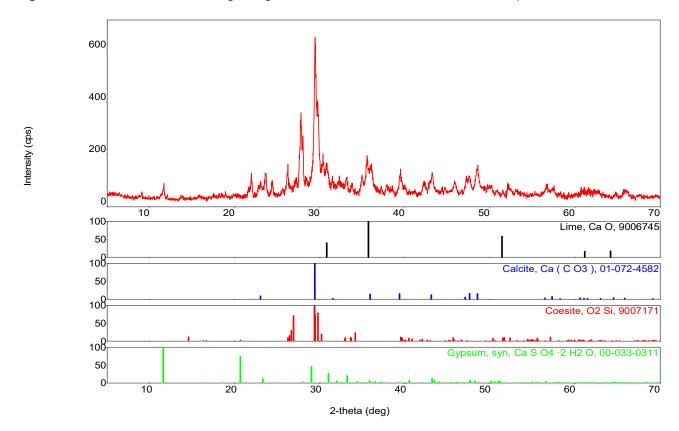


Figure 118. The XRD diffractogram generated from concrete removed Coupon 7.

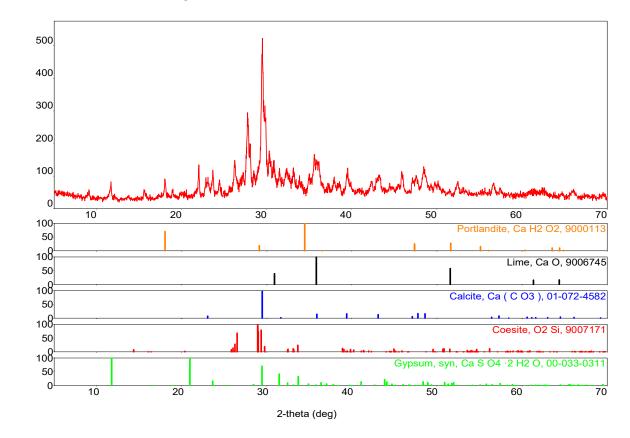


Figure 119. The XRD diffractogram generated from concrete removed Coupon 8.

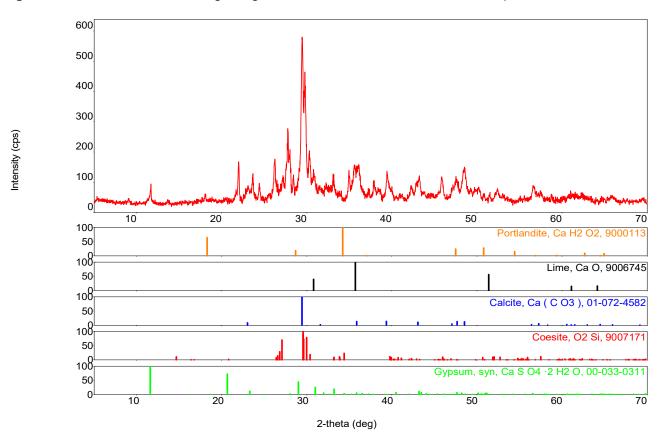


Figure 120. The XRD diffractogram generated from concrete removed Coupon 10.

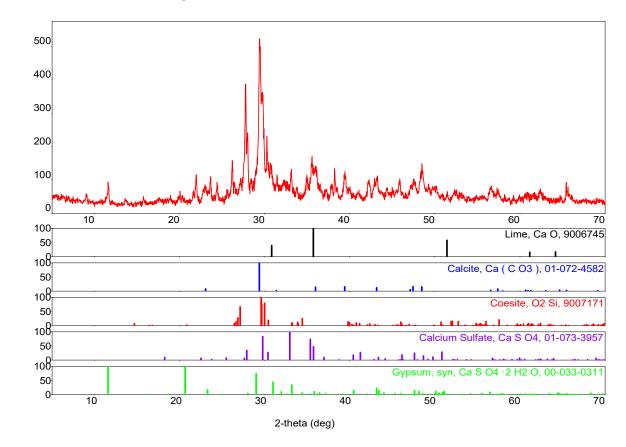
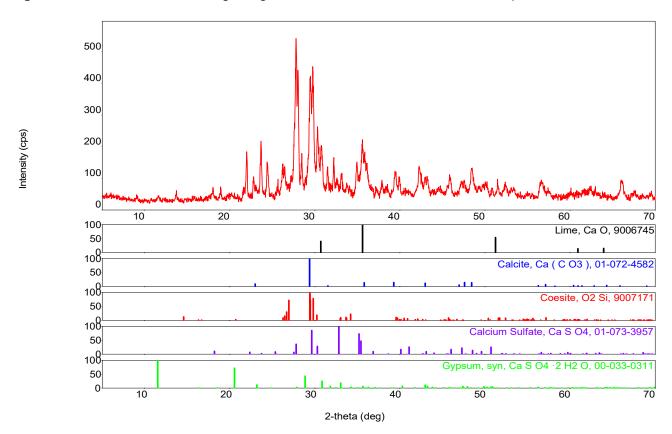
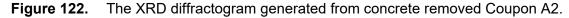


Figure 121. The XRD diffractogram generated from concrete removed Coupon A1.





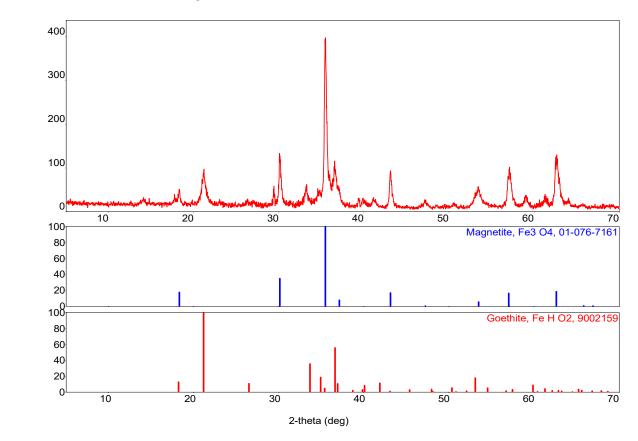


Figure 123. The XRD diffractogram generated from corrosion product removed Coupon 1.

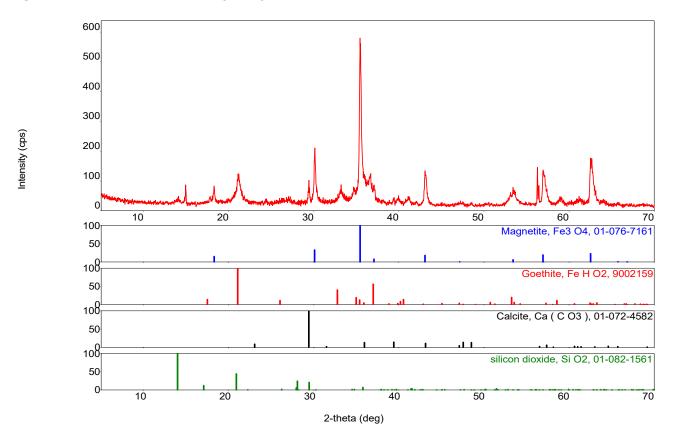


Figure 124. The XRD diffractogram generated from corrosion product removed Coupon 2.

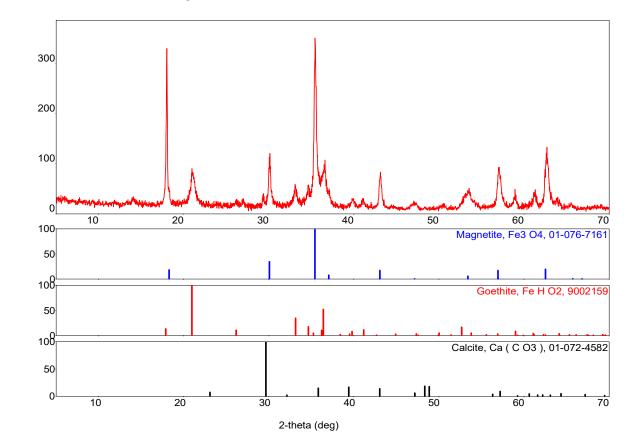
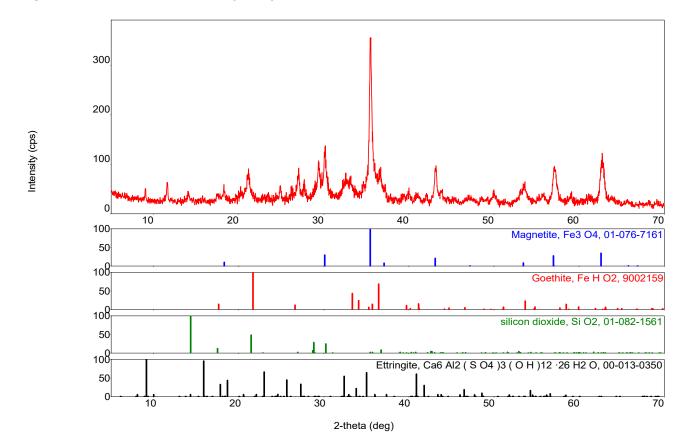


Figure 125. The XRD diffractogram generated from corrosion product removed Coupon 3.





Solomon Resources, LLC

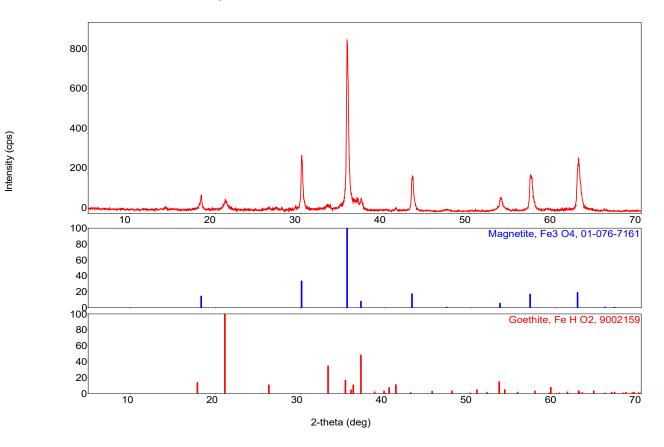


Figure 127. The XRD diffractogram generated from corrosion product removed Coupon 7.

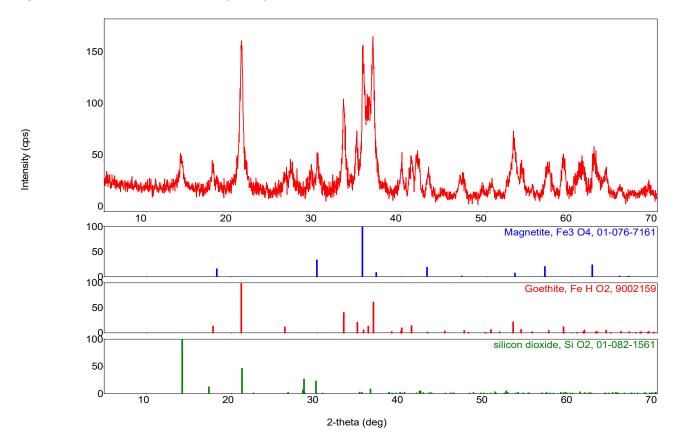


Figure 128. The XRD diffractogram generated from corrosion product removed Coupon 8.



Figure 129. An image of the interior surface of Coupon 1 after CO<sub>2</sub> blast cleaning.



Figure 130. An image of the exterior surface of Coupon 1 after CO<sub>2</sub> blast cleaning.



Figure 131. An image of the interior surface of Coupon 2 after CO<sub>2</sub> blast cleaning.



Figure 132. An image of the exterior surface of Coupon 2 after CO<sub>2</sub> blast cleaning.



**Figure 133.** An image of the interior surface of Coupon 3 after CO<sub>2</sub> blast cleaning.







Figure 135. An image of the interior surface of Coupon 5 after CO<sub>2</sub> blast cleaning.



Figure 136. An image of the exterior surface of Coupon 5 after CO<sub>2</sub> blast cleaning.



Figure 137. An image of the interior surface of Coupon 6 after CO<sub>2</sub> blast cleaning.



Figure 138. An image of the exterior surface of Coupon 6 after CO<sub>2</sub> blast cleaning.



Figure 139. An image of the interior surface of Coupon 7 after CO<sub>2</sub> blast cleaning.



Figure 140. An image of the exterior surface of Coupon 7 after CO<sub>2</sub> blast cleaning.



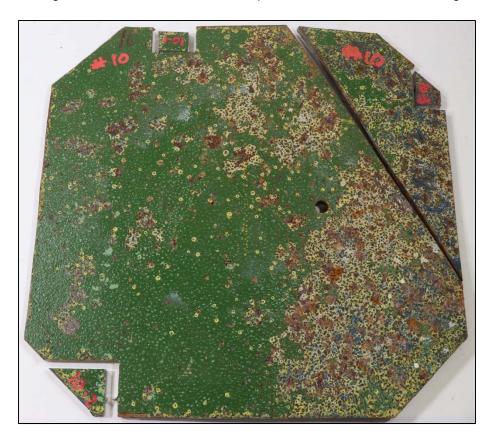
Figure 141. An image of the interior surface of Coupon 8 after CO<sub>2</sub> blast cleaning.



Figure 142. An image of the exterior surface of Coupon 8 after CO<sub>2</sub> blast cleaning.



**Figure 143.** An image of the interior surface of Coupon 10 after CO<sub>2</sub> blast cleaning.







**Figure 145.** An image of the interior surface of Coupon A1 after CO<sub>2</sub> blast cleaning.



Figure 146. An image of the exterior surface of Coupon A1 after CO<sub>2</sub> blast cleaning.



Figure 147. An image of the interior surface of Coupon A2 after CO<sub>2</sub> blast cleaning.







**Figure 149.** A screenshot of the 3D digital image of a specimen excised from Coupon 1 labeled as 1-1, after CO<sub>2</sub> blast cleaning



**Figure 150.** A screenshot of the 3D digital image of a specimen excised from Coupon 1 labeled as 1-2, after CO<sub>2</sub> blast cleaning



**Figure 151.** A screenshot of the 3D digital image of a specimen excised from Coupon 1 labeled as 1-3, after CO<sub>2</sub> blast cleaning



**Figure 152.** A screenshot of the 3D digital image of a specimen excised from Coupon 2 labeled as 2-1, after CO<sub>2</sub> blast cleaning



Figure 153. A screenshot of the 3D digital image of a specimen excised from Coupon 2 labeled as 2-2, after  $CO_2$  blast cleaning



**Figure 154.** A screenshot of the 3D digital image of a specimen excised from Coupon 2 labeled as 2-3, after CO<sub>2</sub> blast cleaning



Figure 155. A screenshot of the 3D digital image of a specimen excised from Coupon 3 labeled as 3-1, after CO<sub>2</sub> blast cleaning



**Figure 156.** A screenshot of the 3D digital image of a specimen excised from Coupon 3 labeled as 3-2, after CO<sub>2</sub> blast cleaning



**Figure 157.** A screenshot of the 3D digital image of a specimen excised from Coupon 3 labeled as 3-3, after CO<sub>2</sub> blast cleaning



**Figure 158.** A screenshot of the 3D digital image of a specimen excised from Coupon 5 labeled as 5-1, after CO<sub>2</sub> blast cleaning



**Figure 159.** A screenshot of the 3D digital image of a specimen excised from Coupon 5 labeled as 5-2, after CO<sub>2</sub> blast cleaning



**Figure 160.** A screenshot of the 3D digital image of a specimen excised from Coupon 5 labeled as 5-3, after CO<sub>2</sub> blast cleaning



**Figure 161.** A screenshot of the 3D digital image of a specimen excised from Coupon 6 labeled as 6-1, after CO<sub>2</sub> blast cleaning



**Figure 162.** A screenshot of the 3D digital image of a specimen excised from Coupon 6 labeled as 6-2, after CO<sub>2</sub> blast cleaning



**Figure 163.** A screenshot of the 3D digital image of a specimen excised from Coupon 6 labeled as 6-3, after CO<sub>2</sub> blast cleaning



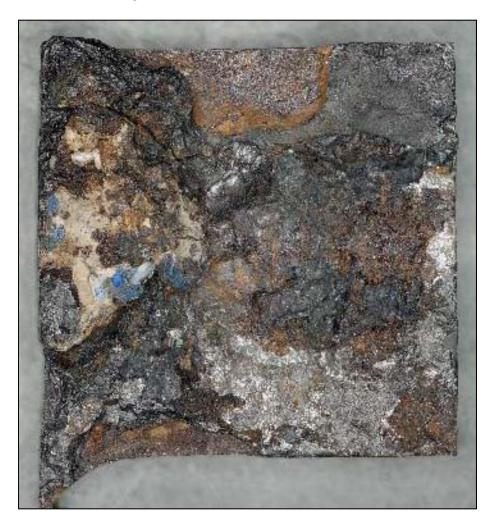
**Figure 164.** A screenshot of the 3D digital image of a specimen excised from Coupon 7 labeled as 7-1, after  $CO_2$  blast cleaning



Figure 165. A screenshot of the 3D digital image of a specimen excised from Coupon 7 labeled as 7-2, after  $CO_2$  blast cleaning.



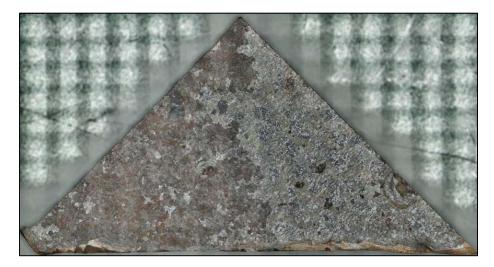
**Figure 166.** A screenshot of the 3D digital image of a specimen excised from Coupon 7 labeled as 7-3, after  $CO_2$  blast cleaning



**Figure 167.** A screenshot of the 3D digital image of a specimen excised from Coupon 7 labeled as 7-4, after CO<sub>2</sub> blast cleaning



**Figure 168.** A screenshot of the 3D digital image of a specimen excised from Coupon 8 labeled as 8-1, after CO<sub>2</sub> blast cleaning



**Figure 169.** A screenshot of the 3D digital image of a specimen excised from Coupon 8 labeled as 8-2, after CO<sub>2</sub> blast cleaning



**Figure 170.** A screenshot of the 3D digital image of a specimen excised from Coupon 8 labeled as 8-3, after CO<sub>2</sub> blast cleaning



**Figure 171.** A screenshot of the 3D digital image of a specimen excised from Coupon 10 labeled as 10-1, after CO<sub>2</sub> blast cleaning



**Figure 172.** A screenshot of the 3D digital image of a specimen excised from Coupon 10 labeled as 10-2, after CO<sub>2</sub> blast cleaning



**Figure 173.** A screenshot of the 3D digital image of a specimen excised from Coupon 10 labeled as 10-3, after CO<sub>2</sub> blast cleaning



**Figure 174.** A screenshot of the 3D digital image of a specimen excised from Coupon A-1 labeled as A1-1, after CO<sub>2</sub> blast cleaning



**Figure 175.** A screenshot of the 3D digital image of a specimen excised from Coupon A-1 labeled as A1-2, after CO<sub>2</sub> blast cleaning



**Figure 176.** A screenshot of the 3D digital image of a specimen excised from Coupon A-1 labeled as A1-3, after CO<sub>2</sub> blast cleaning



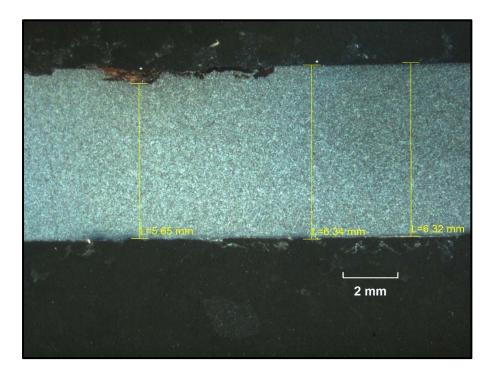
**Figure 177.** A screenshot of the 3D digital image of a specimen excised from Coupon A-2 labeled as A2-1, after CO<sub>2</sub> blast cleaning



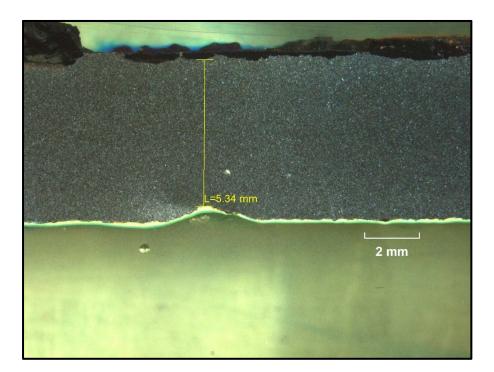
**Figure 178.** A screenshot of the 3D digital image of a specimen excised from Coupon A-2 labeled as A2-2, after CO<sub>2</sub> blast cleaning



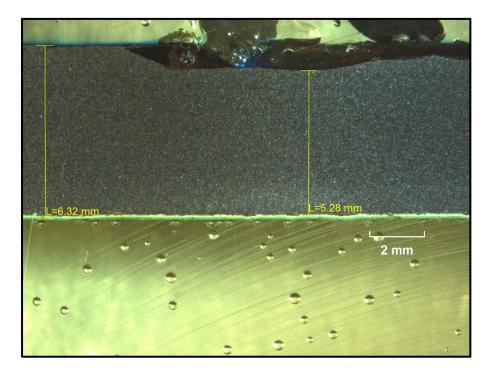
**Figure 179.** A screenshot of the 3D digital image of a specimen excised from Coupon A-2 labeled as A2-3, after CO<sub>2</sub> blast cleaning



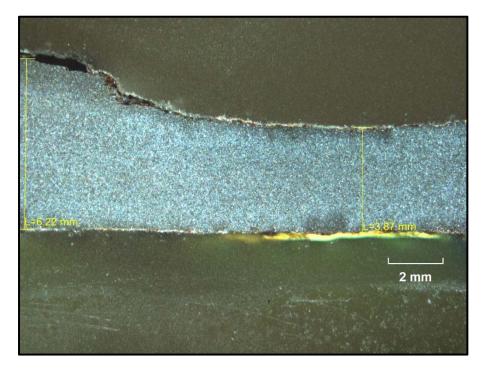
**Figure 180.** The prepared metallographic cross-section from Sample 1-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



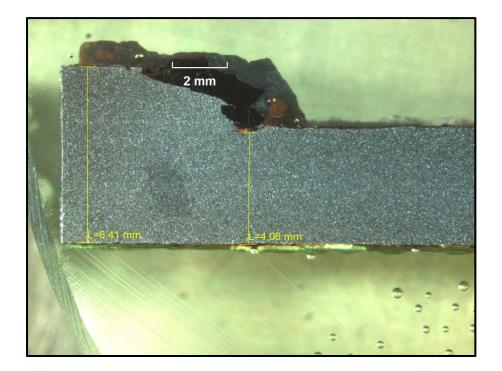
**Figure 181.** The prepared metallographic cross-section from Sample 1-3 is shown during examination with a stereomicroscope. The thickest remaining wall is shown. Note that the reduction in wall thickness was due to a depression on the painted face of the specimen. (7X)



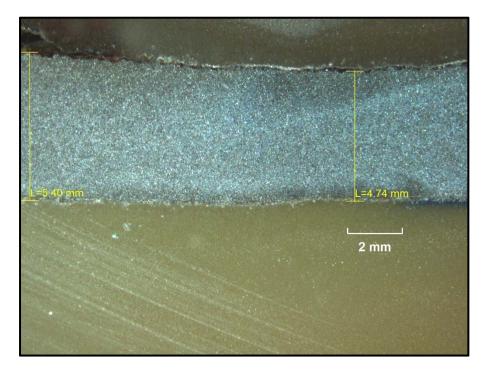
**Figure 182.** The prepared metallographic cross-section from Sample 1-4 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



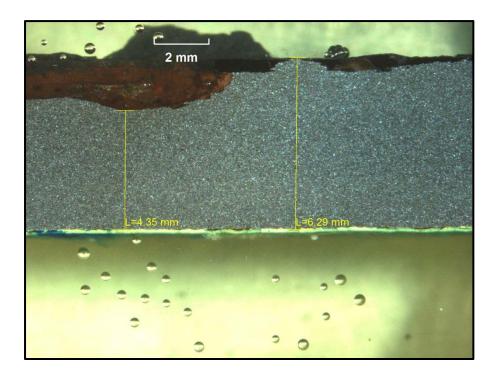
**Figure 183.** The prepared metallographic cross-section from Sample 2-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



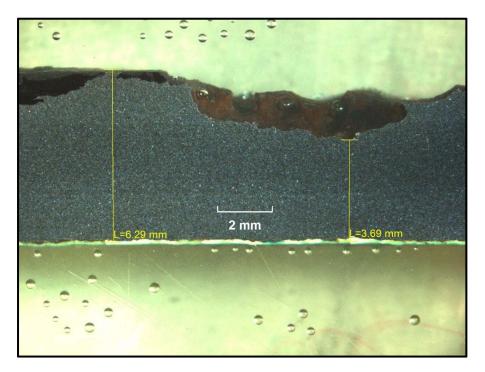
**Figure 184.** The prepared metallographic cross-section from Sample 2-2 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



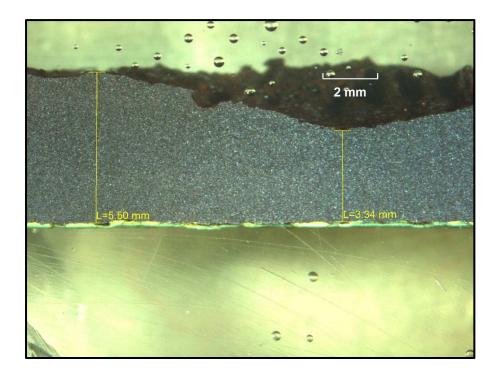
**Figure 185.** The prepared metallographic cross-section from Sample 2-3 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



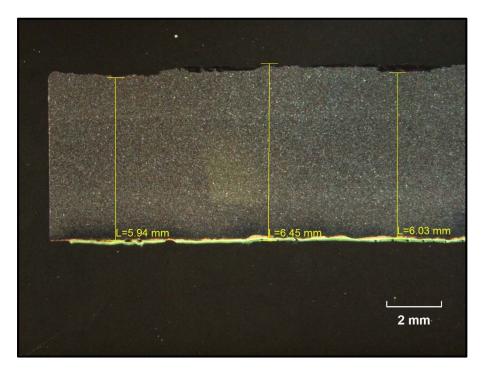
**Figure 186.** The prepared metallographic cross-section from Sample 3-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



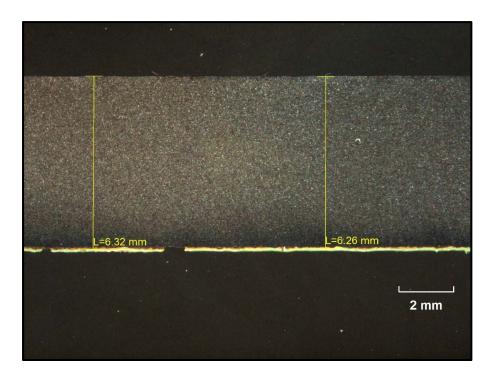
**Figure 187.** The prepared metallographic cross-section from Sample 3-2 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



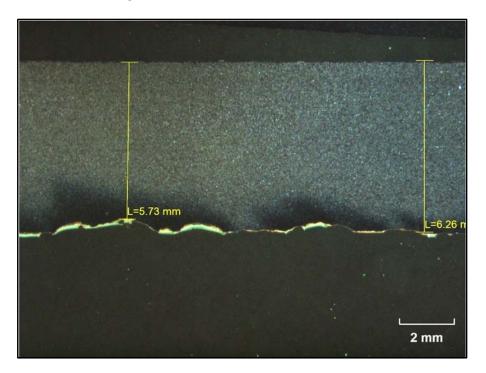
**Figure 188.** The prepared metallographic cross-section from Sample 3-3 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



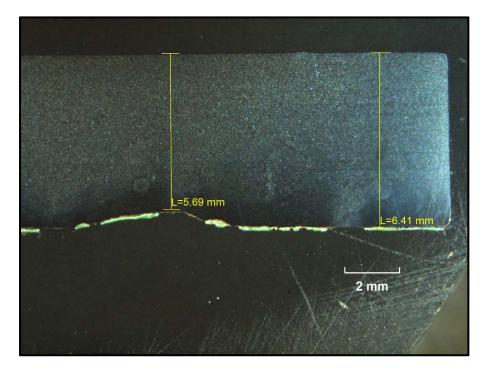
**Figure 189.** The prepared metallographic cross-section from Sample 3-4 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



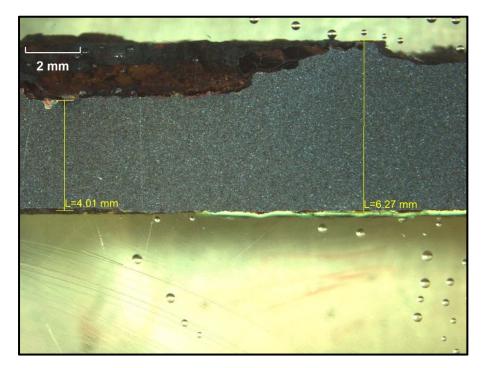
**Figure 190.** The prepared metallographic cross-section from Sample 5-1 is shown during examination with a stereomicroscope. No significant corrosion was observed. (7X)



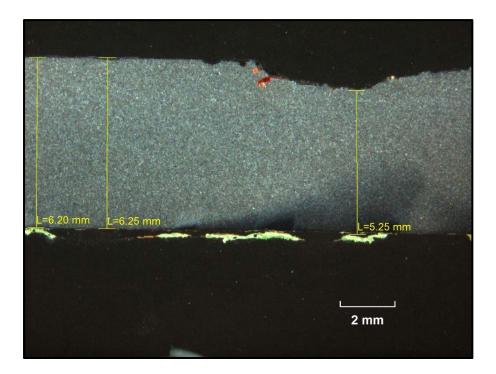
**Figure 191.** The prepared metallographic cross-section from Sample 5-4 is shown during examination with a stereomicroscope. The thickest remaining wall is shown. Note that the reduction in wall thickness was due to a depression on the painted face of the specimen. (7X)



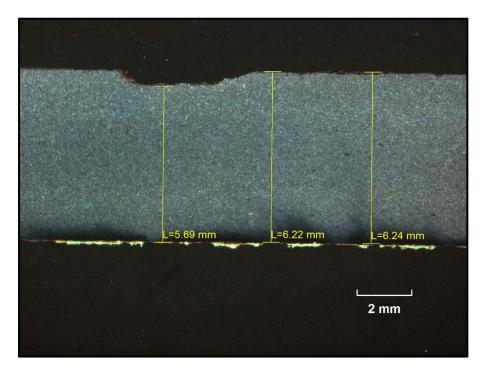
**Figure 192.** The prepared metallographic cross-section from Sample 5-5 is shown during examination with a stereomicroscope. The thickest remaining wall is shown. Note that the reduction in wall thickness was due to a depression on the painted face of the specimen. (7X)



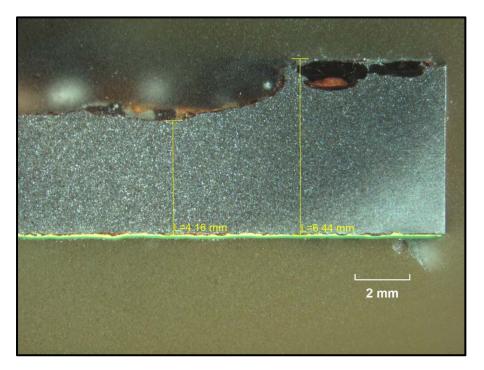
**Figure 193.** The prepared metallographic cross-section from Sample 6-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



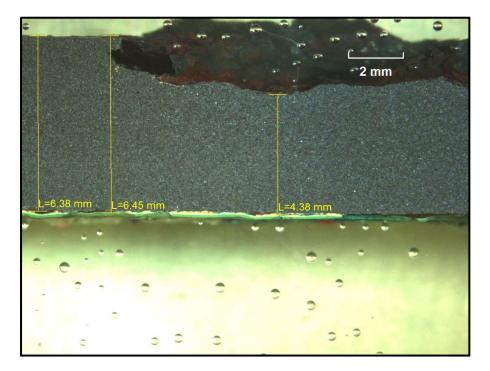
**Figure 194.** The prepared metallographic cross-section from Sample 6-2 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



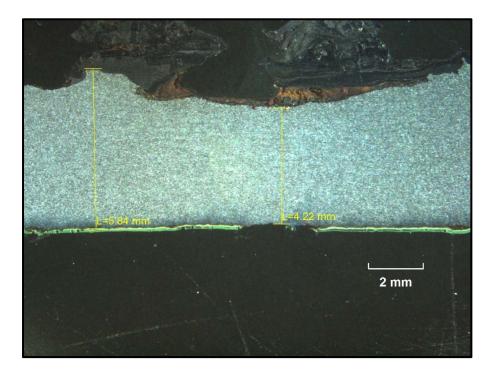
**Figure 195.** The prepared metallographic cross-section from Sample 6-3 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



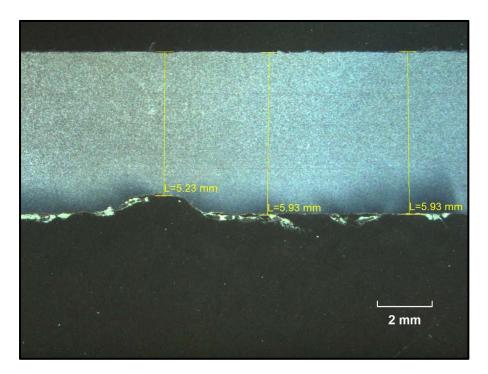
**Figure 196.** The prepared metallographic cross-section from Sample 7-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



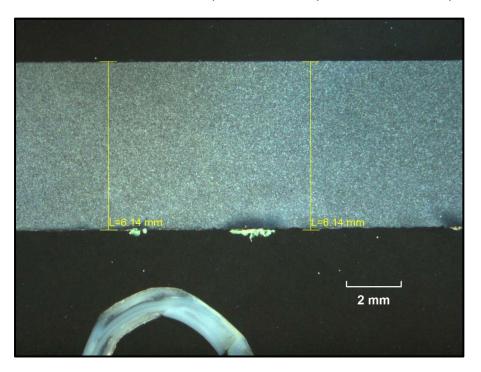
**Figure 197.** The prepared metallographic cross-section from Sample 7-3 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



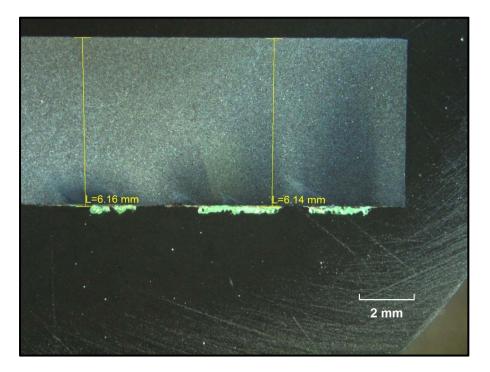
**Figure 198.** The prepared metallographic cross-section from Sample 7-4 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



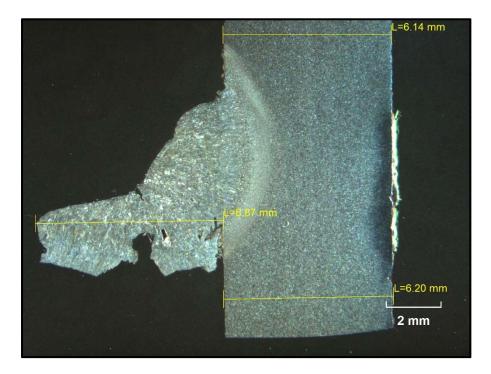
**Figure 199.** The prepared metallographic cross-section from Sample 8-3 is shown during examination with a stereomicroscope. The thickest remaining wall is shown. Note that the reduction in wall thickness was due to a depression on the painted face of the specimen. (7X)



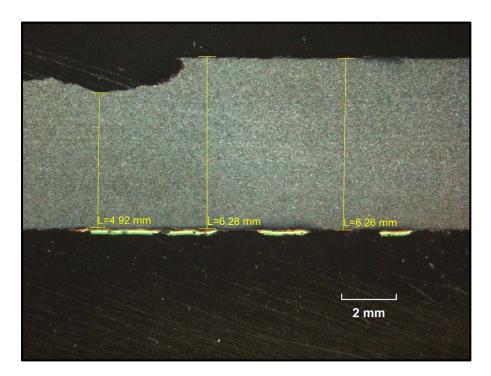
**Figure 200.** The prepared metallographic cross-section from Sample 10-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown. No pitting was observed. (7X)



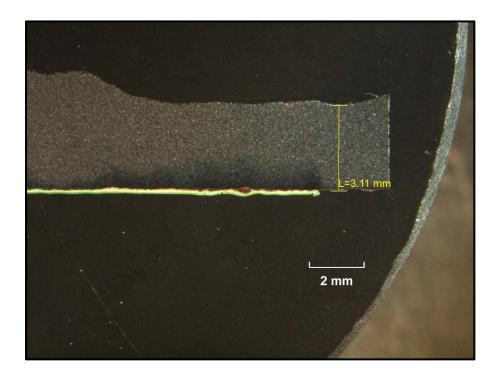
**Figure 201.** The prepared metallographic cross-section from Sample 10-2 is shown during examination with a stereomicroscope. The thickest location(s) are shown. No pitting was observed. (7X)



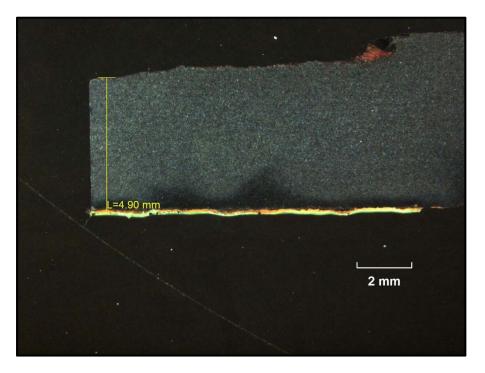
**Figure 202.** The prepared metallographic cross-section from Sample 10-4 is shown during examination with a stereomicroscope. Image is rotated 90° from other images. The anomaly detected during NDE was likely the weld nugget on the interior surface of the coupon (left). No pitting or significant wall loss was observed. (7X)



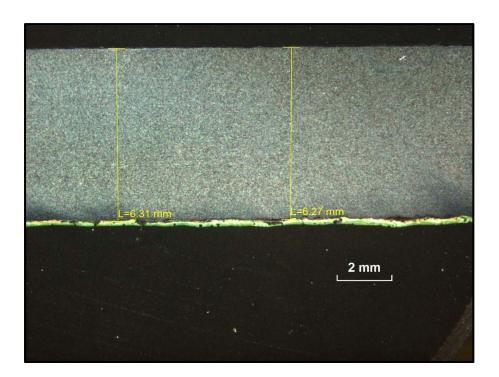
**Figure 203.** The prepared metallographic cross-section from Sample A1-1 is shown during examination with a stereomicroscope. The thickest location(s) are shown, as well as the thickness remaining underneath the deepest observed pit. (7X)



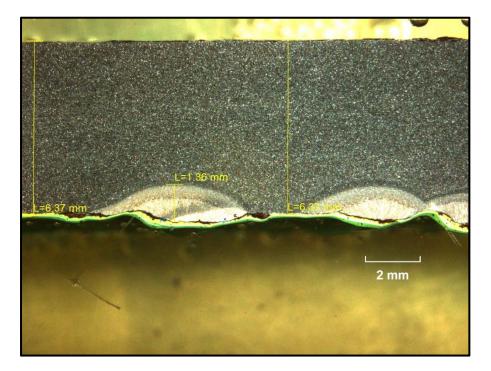
**Figure 204.** The prepared metallographic cross-section from Sample A1-4 is shown during examination with a stereomicroscope. The thickness remaining underneath the deepest observed pit is shown. (7X)



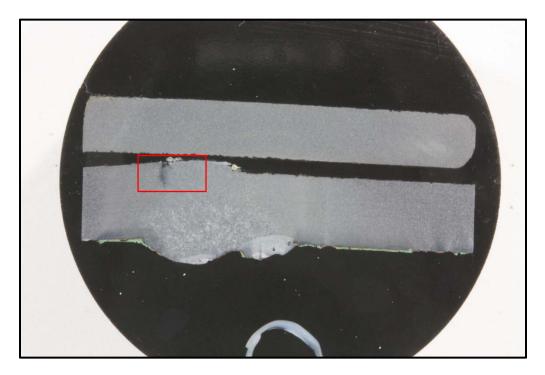
**Figure 205.** The prepared metallographic cross-section from Sample A1-5 is shown during examination with a stereomicroscope. The thickness remaining underneath the deepest observed pit is shown. (7X)



**Figure 206.** The prepared metallographic cross-section from Sample A2-3 is shown during examination with a stereomicroscope. No pitting was observed. (7X)



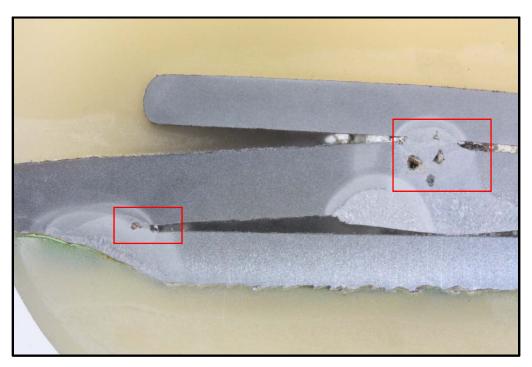
**Figure 207.** The prepared metallographic cross-section from Sample A2-4 is shown during examination with a stereomicroscope. The NDE indication was most likely locations of melting/re-solidification underneath the painted surface. No pitting was observed. (7X)



**Figure 208.** The prepared metallographic cross-section through welded sample 8-1 is shown in an overall view. The painted exterior surface is along the bottom section. The area identified by the red box is shown in the next figure at higher magnification.



**Figure 209.** The location identified in the previous figure is shown at higher magnification. The weld indication (arrow) appeared to be an area of incomplete fusion.



**Figure 210.** The prepared metallographic cross-section through welded sample 8-4 is shown in an overall view. The painted exterior surface is along the bottom section. The areas identified by the red boxes are shown in the next two figures at higher magnification.



**Figure 211.** The left-hand feature in Figure 210 is shown at higher magnification. The weld indication (arrow) appeared to be an area of porosity.



**Figure 212.** The right-hand feature in Figure 210 is shown at higher magnification. The weld indications appeared to be porosity.

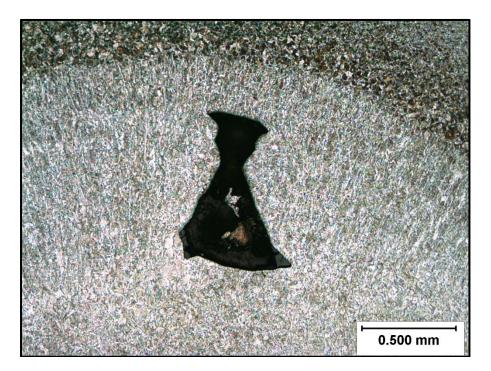
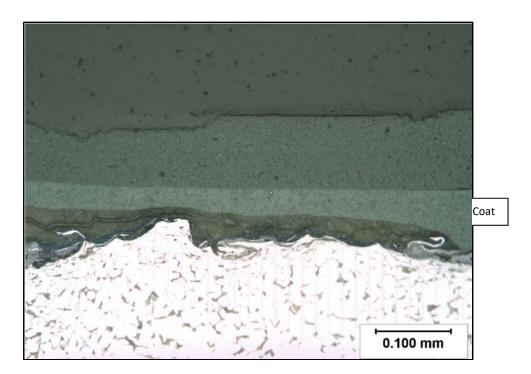


Figure 213. The one of the voids is shown at higher magnification. (2% Nital Etch, 50X)



**Figure 214.** A typical paint layer is shown in cross-section, with key characteristics identified. (2% Nital Etch, 200X)