Highland International

Coatings For Peak Performance

74-HF Series Chem-Temp Hybrid Novolac Epoxy Performance Criteria

Adhesion

Method: ASTM D4541

System: Two coats 74 Series @ 8 mils DFT per coat applied to:
1) SSPC-SP5 White Metal prepared steel
2) SSPC-SP6 Commercial Blast prepared steel
3) No surface preparation
Cured 14 days at 21°C (70°F)

Result: 1) No less than 1200 psi with SSPC-SP5 White Metal blast
2) No less than 1000 psi with SSPC-SP6 Commercial Blast
3) No less than 900 psi with no surface preparation

Chemical Immersion

Method: Continuous Immersion at 93°C (200°F)

System: Two coats 74 Series @ 8 mils DFT per coat applied to SSPC-SP5 White Metal prepared steel. Cured 14 days at 21°C (70°F).

Result: No cracking, lifting or delamination after 60 days of continuous exposure.

Reagents: 10% Methanol, 50% Methanol, 10% Sulfuric Acid, 25% Sulfuric Acid, 10% Sodium Hydroxide, 50% Sodium Hydroxide.

Heat Resistance

Method: Continuous Heat Exposure at 260°C (500°F)

System: A Single coat as well as two coats 74 Series @ 8 mils DFT per coat applied to:
1) SSPC-SP6 Commercial Blast prepared steel
2) No surface preparation
3) No surface preparation with tight rust
Cured 14 days at 21°C (70°F)

Result: No cracking or delamination of the film after 3000 hours of continuous exposure.

Sulfuric Acid Spot Testing

Method: Continuous heat at 177°C (350°F) for 1500 hours. After 1500 hours, spot testing was performed with 98% sulfuric acid for 72 hours.

System: Two coats 74 Series @ 8 mils DFT per coat applied to SSPC-SP6 Commercial Blast pre- pared steel. Cured 24 hours at 21°C (70°F).

Result: No softening or cracking of the film (some discoloration was observed).

Acid Condensation Bath

Method: Coated panels exposed to a condensation bath with 50% sulfuric acid and water. The test duration was 1000 hours total at 177°C (350°F) and the panels were scribed with an "X" to evaluate corrosion. The acid bath was performed in an enclosed apparatus that retained the sulfuric acid concentration, and the panels were suspended in the headspace.

System: Single coat as well as two coats 74 Series @ 8 mils DFT per coat applied to SSPC-SP6 Commercial Blast prepared steel. Cured 24 hours at 21°C (70°F)

Result: No rust creepage, softening, cracking or delamination of the film after 1000 hours of continuous exposure.

Elongation

Method: ASTM D 522.

System: A single coat as well as two coats 74 Series @ 8 mils DFT per coat applied to steel Q Panel.

Result: Pass 1" Mandrel
Elongation at 8 mils: 4.98%
Elongation at 16 mils: 6.70%

Abrasion Resistance

Method: ASTM D 4060 (CS-17 Wheel, 1000 gram load).

System: A single coat 74 Series @ 8 mils DFT.

Result: Average 83 mg loss after 1000 cycles.

Independent Testing - Autoclave

Document Reference: 1516-LS-LGSG-00-007-750 Autoclave Test Report with Profile Provided by RAE Engineering and Inspection Ltd.

Report Date: May 2, 2011

Test Panel Key (as ranked by test report):

#1	Panel 403	Highland 74 Series Hybrid Epoxy Novolac over shallow steel profile (less than 0.5 mil smooth steel profile)
#2	Panel 401	Highland 47 Series Hybrid Epoxy Novolac over shallow steel profile (less than 0.5 mil smooth steel profile)
#3	Panel 402	Highland 74 Series "Experimental" Epoxy Novolac over shallow steel profile (less than 0.5 mil smooth steel profile)
#4	Panel 320	Competitor 100% Solids Epoxy over shallow steel profile (less than 0.5 mil smooth steel profile)
#5	Panel 405	Highland 74 Series "Experimental" low temperature cure Epoxy Novolac over shallow steel profile (less than 0.5 mil smooth steel profile)
#6	Panel 57	Leading Competitor alternative Epoxy Novolac over 2.5-3.5 mil jagged steel profile
#7	Panel 405	Leading Competitor alternative Epoxy Novolac over shallow steel profile (less than 0.5 mil smooth steel profile)

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Autoclave Testing Report

Subject: Evaluation of Organic Coatings by Autoclave Testing

Client: Highland International

Report Date: May 2, 2011

Document Number: 1516-LS-LGSG-00-007-750 Autoclave Test Report With Profile

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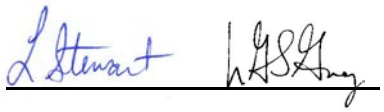
Signature: 

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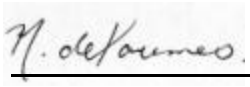
Signature: 

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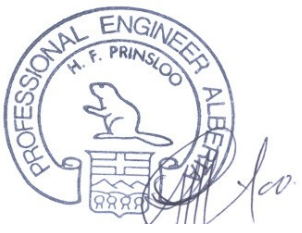
Signature: 

APEGGA Permit to Practice P5158

EXECUTIVE SUMMARY

- Seven coated panels were subjected to an autoclave test under the following conditions:
  - Temperature: 177°C (350°F)
  - Pressure: 240 psig
  - Duration: 4 days
  - Aqueous phase: 5% NaCl solution
  - Organic phase: 1:1 ratio of kerosene to toluene
  - Gaseous phase: 5% H_2S, 5%CO_2 and 90% methane (CH_4).
- The samples tested, supplied by Highland International, were labelled 57, 320, 401, 402, 403, 404 and 405.
- Coatings 403 and 401 did not develop blisters after exposure to the autoclave conditions. Coating 402 developed blisters in the aqueous phase only and coatings 405 and 57 developed blisters in the hydrocarbon phase only. Coating 405 also showed severe cracking in the gas phase. Coating 320 showed blistering in all three phases. Blistering could not be assessed on coating 404 due to severe disbondment of the coating in all three phases.
- Coatings 403, 402 and 320 showed excellent adhesion retention by attaining a top rating (A) in all three phases after exposure to autoclave test conditions. Coating 401 showed ratings of B in all three phases after exposure. 405 also showed B ratings, with the exception of a C rating in the gas phase. Coating 57 showed ratings of D in each phase after exposure and coating 404 showed very poor adhesion, with ratings of E in all three phases.
- Coatings 403 and 320 did not develop undercreep after exposure to the autoclave test conditions. Coating 401 showed tiny amounts of undercreep in the aqueous phase only and coating 405 showed 3 mm undercreep in the gas phase only. Coatings 402 and 57 showed undercreep in all three phases, ranging from 0.5 mm to 2 mm. Undercreep testing could not be performed on coating 404 due to severe disbondment of the coating.
- Coatings 320 and 57 showed excellent impedance before and after test with the lowest Log Z value being 10.0. Coatings 403 and 401 showed very good impedance before and after test with Log Z = 8.4 as the lowest value. Coating 405 also showed very good impedance on the control sample as well as in the hydrocarbon and aqueous phases of the test panel (low of Log Z = 8.1). However, due to severe cracking, no impedance measurement could be performed in the gas phase on coating 405. Coating 402 showed varied barrier properties with Log Z ranging from 5.1 to 8.0 on the control and test panels. Due to disbondment, impedance measurements could not be performed in the gas or hydrocarbon phase on coating 404. Coating 404 showed excellent impedance on the control panel and very good impedance in the aqueous phase (Log Z = 8.3).
- Coatings 403, 401, 402, 405, 57 and 404 all received foam ratings of S/F on the control panels as well as in all three phases of the test panels. Coating 320 received foam ratings of S/M on the control and test panels.

- The performance of Coating 404 was unexpectedly poor. Further investigation indicated the cause was the low surface profile (0.5 mils) of the steel used in preparing the test panel. Subsequent investigation showed that the steel profile under Coatings 402, 403, and 405 was also 0.5 mils or lower.

- The performance of Coatings 402, 403, and 405 can therefore be considered outstanding given the low profile (0.5 mils) of the steel surface used to prepare these panels. The performance would be expected to be even better with the specified profile of 1.5 to 3 mils.

- Coatings 402, 403, and 405 also produced very tough residues that tightly adhered to the steel panel after the 850°F burn-off. In comparison, Coating 57 was charred and ashed and could easily be brushed off the steel surface after burn-off.

- Overall, Coating 403 performed the best of the seven panels tested. This coating showed no blistering, excellent adhesion in all three of the phases, no undercreep and excellent barrier properties.

The overall performance of the coatings is ranked below based on blistering, adhesion, undercreep and impedance results;

403 > 401, 402 > 320 > 405, 57 >> 404

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1 INTRODUCTION

RAE Engineering and Inspection Ltd. (RAE Engineering) was requested by Highland Protective Coatings to perform an autoclave test with Electrochemical Impedance Spectroscopy (EIS) analysis to evaluate seven coated steel samples as provided. The panels were labeled by Highland as follows: 57, 320, 401, 402, 403, 404 and 405.

This report conveys the results of the autoclave test and subsequent evaluation tests of the coatings.

2 SAMPLES

Seven test panels and seven control panels were received from Highland International. The test panels provided were approximately 1.5 inches x 4.5 inches in size and were approximately 1/4 inch in thickness. The control panels were approximately 1.5 inches x 1.5 inches and 1/4 inch in thickness.

The coating identification summary is shown below in Table 1.

Table 1: Sample Identification

HIGHLAND Sample #	RAE Sample #	Panel	Description
57	001	Control	Light Blue
	101	Test	
320	003	Control	Beige
	103	Test	
401	004	Control	Green
	104	Test	
402	005	Control	Gold
	105	Test	
403	006	Control	Dark Red
	106	Test	
404	007	Control	White
	107	Test	
405	008	Control	Dark Red
	108	Test	

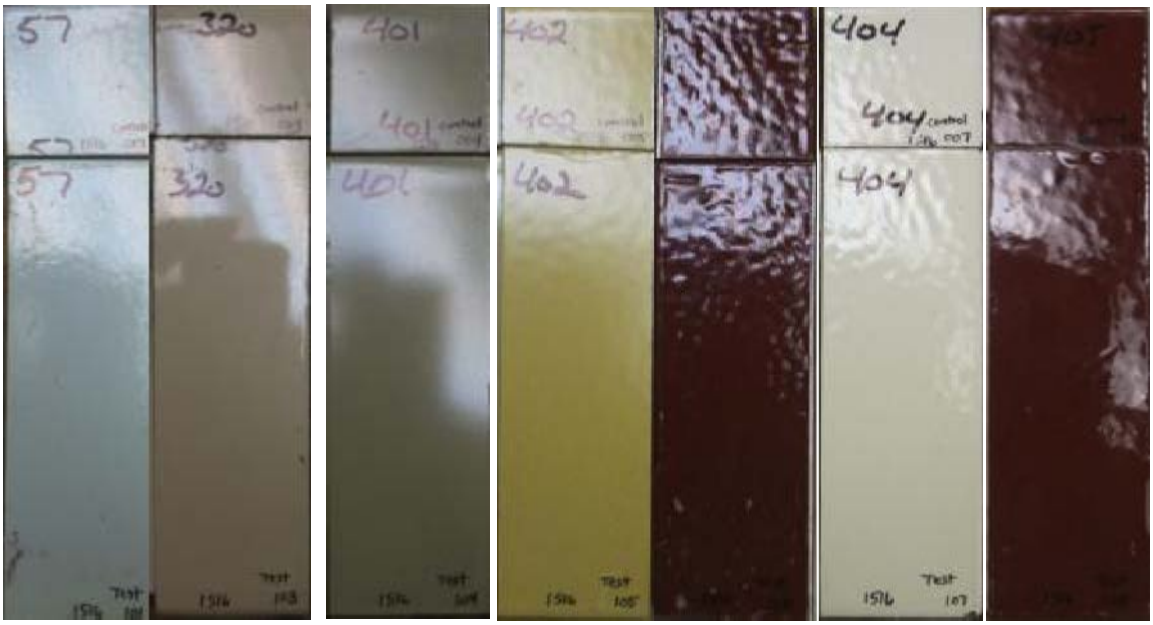


Figure 1: Autoclave Samples – As Received
(Left to Right: 57, 320, 401, 402, 403, 404 and 405)

3 EVALUATION PROCEDURES

3.1 Test Method

NACE TM0185 - Evaluation of Internal Plastic Coatings for Corrosion Control of Tubular Goods by Autoclave Testing.

3.2 Equipment

Teflon-lined 1-litre autoclave, heated with a silicone oil bath, equipped with a pressure transducer, pressure intensifier and thermocouple.

3.3 Test Conditions

Temperature:	177°C (350°F)
Pressure:	240 psig
Duration:	4 days
Gas Phase:	5% hydrogen sulphide (H_2S)
	5% carbon dioxide (CO_2)
	90% methane (CH_4)

Organic Phase: 1:1 ratio of kerosene to toluene

Aqueous Phase: 5% NaCl solution

3.4 Release Procedure:

During cooling of the autoclave from 177°C to 42°C over approximate y 2.5 hrs, the pressure dropped from 260 psig to 48 psig. The remaining pressure was then released uniformly to ambient at no more than 13 psig/minute (actual rate was an average of 12 psig/minute).

3.5 Pre-Test Analysis

Film thickness measurements, adhesion testing, color and EIS impedance at 0.1 Hz after a 48 hr soak in 5% NaCl were performed on the coated panels.

3.6 Post-Test Analysis

Film thickness, blistering, parallel scribe adhesion, undercreep, color change, foam and impedance at 0.1 Hz in gas, hydrocarbon and aqueous phases after 48 hr soak in 5% NaCl at 23°C (73°F).

3.7 Blistering

Blistering was rated using ASTM D714, "Evaluating Degree of Blistering of Paints". This is a pictorial standard, based on blister size and density.

3.8 Adhesion Analysis

A parallel scribe method was used on the coated test and control panels. Two cuts, 1/8 inch apart, are cut through the coating to base metal with a CSA blade. The adhesion of the coating between the scribe marks is evaluated by prying with a utility knife. Adhesion is evaluated within one hour after the panels are removed from the autoclave. The following scale was used to rate the adhesion:

Rating	Description
A	No change/no disbondment
B	Slight Change of Adhesion (>50% still attached)
C	Moderate Loss of Adhesion (<50% still attached)
D	Severe loss of adhesion
E	Disbondment

3.9 Undercreep

Undercreep from the bare edge of the panel is reported in mm.

3.10 Color Change

Color change was assessed based on NACE TM0185. Change in color is typical for tank lining products in autoclave testing. Staining was not considered a color change.

N	No change	M	Moderate change
S	Slight change	SE	Severe change

3.11 Microscopy/Visual Observations

Tested panels were examined under a stereomicroscope to gain additional information.

3.12 Foam

Foam size and density was determined by cutting through the coating film thickness at an angle between 30° and 45°, when disbonded coating chips could not be used. The foam size and density is assessed in the context of film thickness according to the following key:

F	Few	S	Small (< 10% of total film thickness)
M	Medium	M	Medium (10% - 40% of total film thickness)
MD	Medium-dense	L	Large (> 40% of total film thickness)
D	Dense	N	None

3.13 Determining of surface profile

Surface profile of the steel panels to which the coating had been applied was determined by the following method. A small sample of the coated panel (either tested or control portion) was placed in a muffle furnace at 850°F for 2 to 4 hours, after which the furnace was turned off and the sample cooled. The ash was brushed from the surface with a stiff steel brush. The sample was then placed in Dynosolve, a paint stripper. The sample was rinsed, dried, and brushed once more. If residue remained, it was scraped and broken away from the surface using a utility knife while the sample was under a stereomicroscope. The exposed surface profile was measured based on comparison with a Keane Tator Profile Comparator, under the stereomicroscope.

3.14 Electrochemical Impedance Spectroscopy (EIS)

EIS is a laboratory method for evaluating the protectiveness of organic coatings. EIS provides a quantitative measurement of the barrier properties of a coating and is related to the permeability of the coating to aqueous and electrolyte. The higher the impedance of a coating, the lower its permeability to corrosive species, and hence the more protective the coating is. Impedance, but not Log Z impedance, theoretically increases as linear function of film thickness. EIS does not evaluate the adhesion of a coating; that property must be evaluated by a different test method.

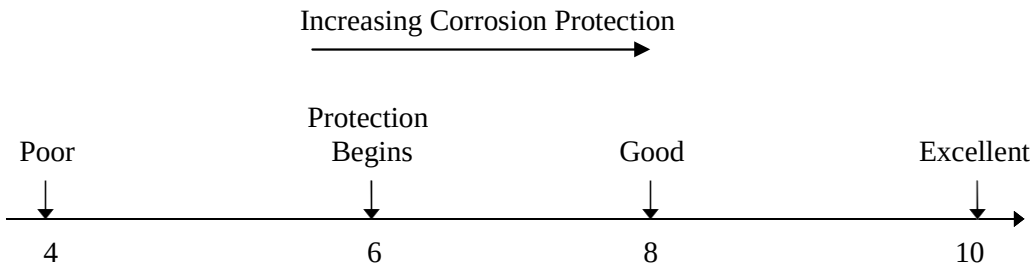
Experimentally, impedance of a coating is determined as a function of the frequency of an applied AC voltage. The data consist of a Bode plot of Log Z versus Log f, where Z is impedance in ohms*cm^2 and f is frequency in Hertz (0.05 Hz to 100 kHz). From the Bode plot, Log Z at 0.1 Hz is determined by interpolation.

The Log Z value at 0.1 Hz is tabulated and used as the basis of comparison between coatings, or for monitoring the change of a coating as a function of exposure time to a test environment. Selection of Log Z at 0.1 Hz is somewhat arbitrary, but represents a compromise between speed of analysis and selection of a frequency at which differences in coating performance can be reliably determined.

Anticipated performance of a coating based on Log Z is shown below in the Figure, which is derived from a large literature of laboratory and fieldwork.

In the autoclave test, the impedance of the coated samples was measured before and after exposure to autoclave conditions. Pre-test is a baseline, against which post-run values are compared to assess deterioration. Post-run measurements were made in the aqueous, organic and gas phase areas of the coated test panels.

Corrosion Protection of Organic Coatings



Coating Impedance, Log Z (Z in ohms cm^2 @ 0.1 Hz) at 73°F (23°C)

4 RESULTS

4.1 Coating 403

Results are shown in Table 2b and Table 3.

4.1.1. Blistering

No blistering was observed in the gas, hydrocarbon or aqueous phases of the test panel.

4.1.2. Adhesion

Results are shown in Figure 4b.

Coating 403 showed excellent resistance to removal, having parallel scribe adhesion ratings of A both before and after exposure to the autoclave test conditions.

4.1.3. Undercreep

Results are shown in Figure 5.

Coating 403 showed no evidence of undercreep in all three phases.

4.1.4. Color Change

Results are shown Figure 2.

Coating 403 appeared slightly darker in the gas and hydrocarbon phases. Slight bleaching of the coating occurred in the aqueous phase.

4.1.5. Visual Observations

Coating 403 showed no cracking or other signs of degradation.

4.1.6. Foam

Results are shown in Table 2b and Figure 6f.

Coating 403 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.1.7. EIS

Coating 403 showed high impedance before exposure to the autoclave test conditions, with a Log Z value of 9.9 on the control panel. The exposed test panel also showed high impedance, with Log Z values of 10.7 in the gas phase, 9.9 in the hydrocarbon phase and 8.4 in the aqueous phase.

4.2 Coating 401

Results are shown in Table 2b and Table 3.

4.2.1. Blistering

No blistering was observed in the gas, hydrocarbon or aqueous phases of the test panel.

4.2.2. Adhesion

Results are shown in Figure 4a.

Coating 401 showed good resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of B on the control panel. After exposure to the autoclave test conditions, the coating retained adhesion ratings of B in all three phases, although the coating could be removed adhesively in 2 mm pieces along the scribed lines.

4.2.3. Undercreep

Results are shown in Figure 5.

Coating 401 showed a very small amount (0.5 mm) of undercreep in the aqueous phase. The substrate appeared slightly tarnished but no corrosion products were observed. The coating showed no undercreep in the gas and hydrocarbon phases, although pieces up to 1 mm could be removed cohesively.

4.2.4. Color Change

Coating 401 appeared slightly bleached in the gas phase and moderate yellowing occurred in the hydrocarbon and aqueous phases.

4.2.5. Visual Observations

Coating 401 showed no cracking or other signs of degradation.

4.2.6. Foam

Results are shown in Table 2b and Figure 6b.

Coating 401 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.2.7. EIS

Coating 401 showed good impedance before exposure to the autoclave test conditions, with a Log Z value of 8.9 on the control panel. The coating showed higher impedance on the exposed test panel, with Log Z values of 10.7 in the gas phase, 10.6 in the hydrocarbon phase and 9.6 in the aqueous phase. The increase in impedance after exposure may be attributed to chemical or physical changes in the film introduced by the autoclave test conditions, mainly the high temperature.

4.3 Coating 402

Results are shown in Table 2b and Table 3.

4.3.1. Blistering

Results are shown in Figure 3c.

Coating 402 developed blisters of ASTM D714 size #8MD in the aqueous phase only. The blisters were <1 mm in diameter, were dry inside and did not penetrate to the substrate. No blistering was observed in the gas and hydrocarbon phases of the test panel.

4.3.2. Adhesion

Results are shown in Figure 4a.

Coating 402 showed excellent resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of A on the control panel. After exposure to the autoclave test conditions, the coating retained adhesion ratings of A in all three phases although the coating could be removed, only cohesively, in 1 mm pieces along the scribe lines.

4.3.3. Undercreep

Results are shown in Figure 5.

Coating 402 showed very small amounts (0.5 mm) of undercreep in the hydrocarbon and aqueous phases. Slightly more undercreep was observed in the gas phase (1 mm). The substrate appeared tarnished.

4.3.4. Color Change

No color change occurred in the gas phase, however some circular staining was observed. Coating 402 appeared slightly darker in the hydrocarbon phase and moderately darker in the aqueous phase.

4.3.5. Visual Observations

Coating 402 showed no cracking or other signs of degradation.

4.3.6. Foam

Results are shown in Table 2b and Figure 6e.

Coating 402 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.3.7. EIS

Coating 402 showed good impedance before exposure to the autoclave test conditions, with a Log Z value of 7.6 on the control panel. The coating showed

comparable impedance in the gas phase of the exposed test panel, with Log Z values of 8.0. Lower impedance was recorded in the hydrocarbon and aqueous phases, with Log Z values of 5.6 and 5.1, respectively.

4.4 Coating 320

Results are shown in Table 2a and Table 3.

4.4.1. Blistering

Results are shown in Figure 3b.

Coating 320 developed blisters of ASTM D714 size #4M in the gas and hydrocarbon phases. The blisters were 2 mm to 3 mm in diameter. Blisters of size #8F (<1 mm diameter) were observed in the aqueous phase of the test panel. All blisters appeared to be dry inside and did not penetrate to the substrate.

4.4.2. Adhesion

Results are shown in Figure 4a.

Coating 320 showed excellent resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of A on the control panel. After exposure to the autoclave test conditions, the coating retained adhesion ratings of A in all three phases, although the entire test area could be removed cohesively in 2 mm to 6 mm pieces. An even layer of base coat remained adhered, preventing exposure of the substrate. Adhesive disbondment was observed at one corner of the test panel in the gas phase upon removal from the autoclave. This disbondment appeared to originate at the site of the drilled hole and was not considered in the adhesion analysis.

4.4.3. Undercreep

Results are shown in Figure 5.

Coating 320 showed no evidence of undercreep in all three phases.

4.4.4. Color Change

Coating 320 appeared slightly bleached in the gas phase and had yellowed slightly in the hydrocarbon and aqueous phases.

4.4.5. Visual Observations

Tiny indents were observed on the surface of the coating in the aqueous phase. These indents were <1 mm in diameter and resembled pinholes that were not yet open to the atmosphere.

4.4.6. Foam

Results are shown in Table 2a and Figure 6c.

Coating 320 received foam ratings of S/M on the control panel as well as in all three phases of the test panel.

4.4.7. EIS

Coating 320 showed excellent impedance before exposure to the autoclave test conditions, with a Log Z value of 11.1 on the control panel. The coating also showed excellent impedance on the exposed test panel, with Log Z values of 10.8 in the gas and hydrocarbon phases and 10.7 in the aqueous phase.

4.5 Coating 405

Results are shown in Table 2c and Table 3.

4.5.1. Blistering

Results are shown in Figure 3d.

Coating 405 developed blisters of ASTM D714 size #3F in the hydrocarbon phase only. The largest blister was 3 mm in diameter and was cracked across the top. A few smaller blisters were observed in the surrounding area. All blisters appeared damp inside and penetrated to the substrate.

4.5.2. Adhesion

Results are shown in Figure 4b.

Coating 405 showed good resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of B on the control panel. After exposure to the autoclave test conditions, the coating retained adhesion ratings of B in the hydrocarbon and gas phases, although coating pieces 2 mm to 3 mm in size could be removed adhesively. The coating in the gas phase could be removed adhesively in 2 mm to 4 mm pieces until most of the area had disbonded, resulting in a C rating.

4.5.3. Undercreep

Results are shown in Figure 5.

Coating 405 showed undercreep (3 mm) in the gas phase and the substrate appeared tarnished.

4.5.4. Color Change

Coating 405 appeared slightly bleached in the gas and hydrocarbon phases and moderately bleached in the aqueous phase.

4.5.5. Visual Observations

Results are shown in Figure 3e.

Severe cracking occurred across the entire coating surface in the gas phase. A single crack was observed diagonally across the surface of the hydrocarbon phase. The cracks appeared to penetrate to the substrate, although the surrounding coating remained adhered to the substrate.

4.5.6. Foam

Results are shown in Table 2c and Figure 6h.

Coating 405 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.5.7. EIS

EIS measurements could not be performed in the gas phase due to severe cracking. Coating 405 showed excellent impedance before exposure to the autoclave test conditions, with a Log Z value of 10.1 on the control panel. The coating showed very good impedance on the exposed test panel, with Log Z values of 8.1 in the hydrocarbon phase and 9.4 in the aqueous phase.

4.6 Coating 57

Results are shown in Table 2a and Table 3.

4.6.1. Blistering

Results are shown in Figure 3a.

Coating 57 developed blisters of ASTM D714 size #4F in the hydrocarbon phase only. The blisters were 1.5 mm to 3 mm in diameter and were dry inside and did not appear to penetrate to the substrate.

4.6.2. Adhesion

Results are shown in Figure 4a.

Coating 57 showed excellent resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of A on the control panel. After exposure to the autoclave test conditions the coating showed a severe loss of adhesion, with ratings of D in all three phases. Coating pieces 2 mm to 4 mm in size could be removed adhesively until the entire area had disbonded.

4.6.3. Undercreep

Results are shown in Figure 5.

Coating 57 showed small amounts (1 mm) of undercreep in the hydrocarbon phase. Slightly more undercreep was observed in the gas and aqueous phases (2 mm). The substrate appeared tarnished.

4.6.4. Color Change

Coating 57 appeared slightly bleached in the gas and aqueo s phases and severely bleached in the hydrocarbon phase.

4.6.5. Visual Observations

Coating 57 showed no cracking or other signs of degradation.

4.6.6. Foam

Results are shown in Table 2a and Figure 6a.

Coating 57 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.6.7. EIS

Coating 57 showed excellent impedance before exposure to the autoclave test conditions, with a Log Z value of 10.6 on the control panel. The coating also showed excellent impedance on the exposed test panel, with Log Z values of 10.7 in the gas phase, 10.6 in the hydrocarbon phase and 10.0 in the aqueous phase.

4.7 Coating 404

Results are shown in Table 2c and Table 3.

4.7.1. Blistering

No blistering could be assessed due to severe disbondment of the coating in all three phases.

4.7.2. Adhesion

Results are shown in Figure 4b.

Coating 404 showed good resistance to removal prior to autoclave exposure, having a parallel scribe adhesion rating of B on the control panel. After exposure to the autoclave test conditions, the coating showed a severe loss in adhesion, having ratings of E in all three phases. The coating was removed adhesively in a single strip across all three phases.

4.7.3. Undercreep

Undercreep testing could not be performed due to severe disbondment of the coating in all three phases.

4.7.4. Color Change

Coating 404 showed slight yellowing in the hydrocarbon and aqueous phases. No change was observed in the gas phase.

4.7.5. Foam

Results are shown in Table 2c and Figure 6g.

Coating 404 received foam ratings of S/F on the control panel as well as in all three phases of the test panel.

4.7.6. EIS

EIS measurements could not be performed in the gas and hydrocarbon phases due to severe disbondment. Coating 404 showed excellent impedance before exposure to the autoclave test conditions, with a Log Z value of 10.2 on the control panel. The coating showed good impedance on the exposed test panel, with a Log Z value of 8.3 in the aqueous phase.

4.8 Analysis of surface profile of test panels

The performance of Coating 404 was unexpectedly poor. Its performance was expected to be very similar to Coating 57, as these two panels were different preparations of the same coating.

Coating 404 had disbonded extensively. As the panel was being analyzed, it was observed that the surface profile was unexpectedly low. Based on comparison with a Keane Tator Comparator, the profile was about 0.5 mils, rather than the required 1.5 to 3 mils.

Consequently, it became of interest to determine the profile on some of the other test panels. The surface profile was determined for Coatings 402, 403, 405, and 57, as described in Section 3.13.

The surface profile under coatings 402, 403, and 405 ranged from 0 to 0.5 mils, considerably below the required profile of 1.5 to 3 mils. Most areas of the panel had a surface morphology typical of abrasive blast cleaning. The performance of Coatings 402, 403, and 405 was therefore outstanding when the low profile is taken into account.

The surface profile under Coating 57 was about 3 mils, which is acceptable, compared to the Coating 404 panel which had a profile of 0.5 mils.

It was further observed that Panels 402, 403, and 405 retained very tough adhering residues that survived the 850°F burn-off. In comparison, Coating 57 was charred and ashed and could easily be brushed off the surface.

5 CONCLUSION

Overall, Coating 403 performed the best of the seven panels tested. This coating showed no blistering, excellent adhesion in all three of the phases, no undercreep and excellent barrier properties.

The performance of Coatings 402, 403, and 405 can be considered outstanding based on the low profile (0.5 mils) of the steel surface used to prepare these panels. The performance would be expected to be even better with a higher profile.

The overall performance of the coatings is ranked below based on blistering, adhesion, undercreep and impedance results;

403 > 401, 402 > 320 > 405, 57 >> 404

Table 2a: Autoclave Test Results for Panels 57 and 320

HIGH LAND Panel #	RAE Panel #		Test Phase	Pre-Test Dry Film Thickness (mils)	Post-Test Dry Film Thickness (mils)	Adhesion (NACE TMO185)	Blistering (ASTM D714)	Undercreep (NACE TMO185)	Foam	Color Change (TMO185)	Log Impedance @ 0.1 Hz (ohm*cm^2)	Comments
57	1516	001	control	16		A			S/F		10.6	Coating appeared slightly bleached in gas and aqueous phases, severe bleaching occurred in h.c. phase. Blisters (1.5 mm to 3 mm) were observed in h.c. phase which were dry inside and did not penetrate to the substrate. No blistering in gas or aqueous phases. Control panel had excellent adhesion (no disbondment). 2 mm to 4 mm pieces could be removed mostly adhesively in all phases on the test panel, until entire test area was removed, showing a severe loss of adhesion. Undercreep testing showed adhesive removal in each phase.
		101	gas	16	12	D	None	2 mm	S/F	Slight	10.7	
			h.c.		16	D	#4F	1 mm	S/F	Severe	10.6	
			aqueous		17	D	None	2 mm	S/F	Slight	10.0	
320	1516	003	control	26		A			S/M		11.1	Coating appeared slightly bleached in gas phase, and slight yellowing occurred in h.c. and aqueous phases. Disbondment occurred at one corner in gas phase, appeared to be caused by drilled hole. Blisters (2 mm to 3 mm) were observed in the gas and h.c. phases. Tiny (<1 mm) blisters and indents (resembled pinholes not open to atmosphere) were observed in aqueous phase. All blisters were dry inside and did not penetrate to the substrate. Control panel showed excellent adhesion. Test panel also showed excellent adhesion in all phases, although 2 mm to 6 mm pieces could be removed only cohesively until entire test area was removed in each phase. Undercreep testing showed adhesive removal in gas and h.c. phases. Undercreep testing showed only cohesive removal in each phase (3 mm in gas and h.c. phases, 1.5 mm in aqueous phase).
		103	gas	24	26	A	#4M	0 mm	S/M	Slight	10.8	
			h.c.		26	A	#4M	0 mm	S/M	Slight	10.8	
			aqueous		27	A	#8F	0 mm	S/M	Slight	10.7	

Table 2b: Autoclave Test Results for Panels 401, 402 and 403

HIGHLAND Panel #	RAE Panel #		Test Phase	Pre-Test Dry Film Thickness (mils)	Post-Test Dry Film Thickness (mils)	Adhesion (NACE TMO185)	Blistering (ASTM D714)	Undercreep (NACE TMO185)	Foam	Color Change (TMO185)	Log Impedance @ 0.1 Hz (ohm*cm^2)	Comments
401	1516	004	control	17		B			S/F		8.9	Coating appeared slightly bleached in gas phase and moderate yellowing occurred in h.c. and aqueous phases. No blistering was observed in gas, h.c. or aqueous phases. Control panel and test panel (all phases) showed good adhesion, 2 mm pieces could be removed adhesively along the scribe lines. Undercreep testing showed adhesive removal in aqueous phase.
		104	gas	16	13	B	None	0 mm	S/F	Slight	10.7	
			h.c.		15	B	None	0 mm	S/F	Moderate	10.6	
			aqueous		17	B	None	0.5 mm	S/F	Moderate	9.6	
402	1516	005	control	13		A			S/F		7.6	Circular staining was observed in gas phase. Coating appeared slightly darker in h.c. phase and moderately darker in aqueous phase. No blistering was observed in gas or h.c. phases. Several tiny (<1 mm) blisters were observed in aqueous phase which were dry inside and did not penetrate to the substrate. Control panel and test panel (all phases) showed excellent adhesion, although 1 mm pieces were removed only cohesively. Undercreep testing showed adhesive removal in gas, h.c. and aqueous phases.
		105	gas	13	13	A	None	1 mm	S/F	None	8.0	
			h.c.		13	A	None	0.5 mm	S/F	Slight	5.6	
			aqueous		13	A	#8MD	0.5 mm	S/F	Moderate	5.1	
403	1516	006	control	14		A			S/F		9.9	Coating appeared slightly darker in gas and h.c. phases, slight bleaching occurred in aqueous phase. No blistering was observed in gas, h.c. or aqueous phases. Control panel and test panel (all phases) showed excellent adhesion, although 1 mm to 2 mm pieces were removed only cohesively. Undercreep testing showed only cohesive removal (1 mm to 2mm) in each phase.
		106	gas	14	13	A	None	0 mm	S/F	Slight	10.7	
			h.c.		14	A	None	0 mm	S/F	Slight	9.9	
			aqueous		17	A	None	0 mm	S/F	Slight	8.4	

Table 2c: Autoclave Test Results for Panels 404 and 405

HIGHLAND Panel #	RAE Panel #		Phase	Pre-Test Dry Film Thickness (mils)	Post-Test Dry Film Thickness (mils)	Adhesion (NACE TMO185)	Blistering (ASTM D714)	Undercreep (NACE TMO185)	Foam	Color Change (TMO185)	Log Impedance @ 0.1 Hz (ohm*cm^2)	Comments
404	1516	007	control	12		B			S/F		10.2	Slight yellowing of the coating occurred in h.c. and aqueous phases, no change in gas phase. No blistering could be observed in gas, h.c. or aqueous phases due to the severe disbondment. The coating was completely disbonded in gas phase and mostly disbonded in h.c. phase. Control panel showed good adhesion, 2 mm pieces removed adhesively. Test panel showed severe loss of adhesion, coating was removed adhesively in single strip across the entire panel (all phases). EIS measurements could not be performed in the gas and h.c. phases due to severe disbondment. Undercreep testing could not be performed.
		107	gas	12	19*	E	N/A	N/A	S/F	None	N/A	
			h.c.		14*	E	N/A	N/A	S/F	Slight	N/A	
			aqueous		12	E	N/A	N/A	S/F	Slight	8.3	
405	1516	008	control	17		B			S/F		10.1	Coating appeared slightly bleached in gas and h.c. phases, and moderately bleached in aqueous phases. Severe cracking occurred across entire coating surface in gas phase and a single crack was observed diagonally across h.c. phase. Cracking penetrated to substrate but surrounding coating remained adhered to substrate surface. A 3 mm blister (cracked across top) and a few smaller blisters were observed in h.c. phase, which were damp inside and penetrated to the substrate. Control panel showed good adhesion, 2 mm pieces removed adhesively. Test panel also showed good adhesion in h.c. and aqueous phases (2 mm to 3 mm pieces removed adhesively). Gas phase showed poor adhesion, with 2 mm to 4 mm pieces removed adhesively until most of the area was disbonded. EIS measurements could not be performed in the gas phase due to severe cracking. Undercreep testing showed only cohesive removal (1 mm to 2mm) in h.c. and aqueous phases.
		108	gas	20	19*	C	None	3 mm	S/F	Slight	N/A	
			h.c.		20	B	<#2F	0 mm	S/F	Slight	8.1	
			aqueous		20	B	None	0 mm	S/F	Moderate	9.4	

* Cracking, DFT measurement attempted

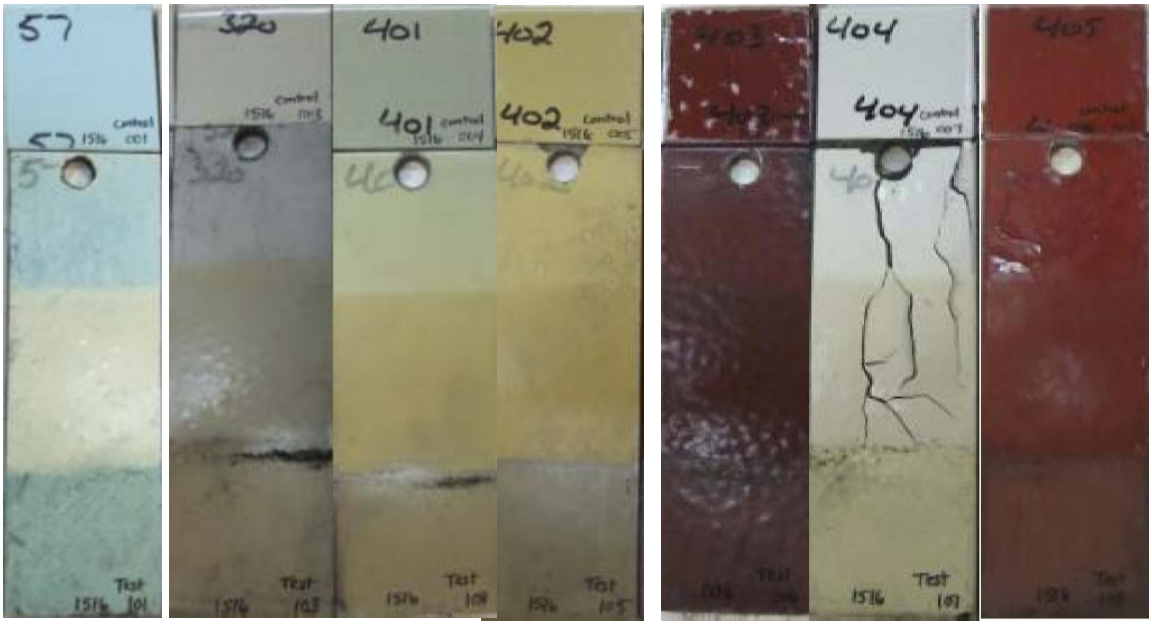


Figure 2: Autoclave Samples – After Exposure
(Left to Right: 57, 320, 401, 402, 403, 404 and 405)

Figure 3a: Blister Results - Panel 57 @ 7.5X Magnification
(hydrocarbon phase)

Figure 3b: Blister Results - Panel 320 @ 20X Magnification
(Left to Right: gas phase, hydrocarbon phase, aqueous phase)

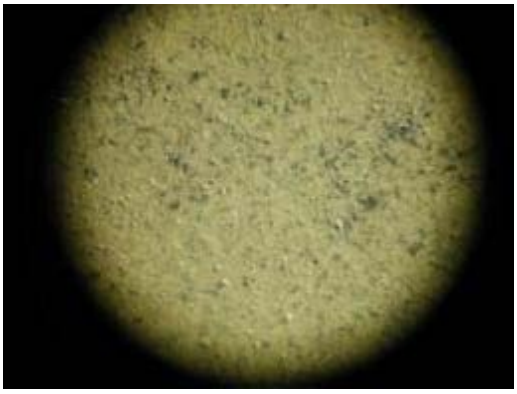


Figure 3c: Blister Results - Panel 402 @ 40X Magnification
(aqueous phase)

Figure 3d: Blister Results - Panel 405 @ 40X Magnification
(hydrocarbon phase)

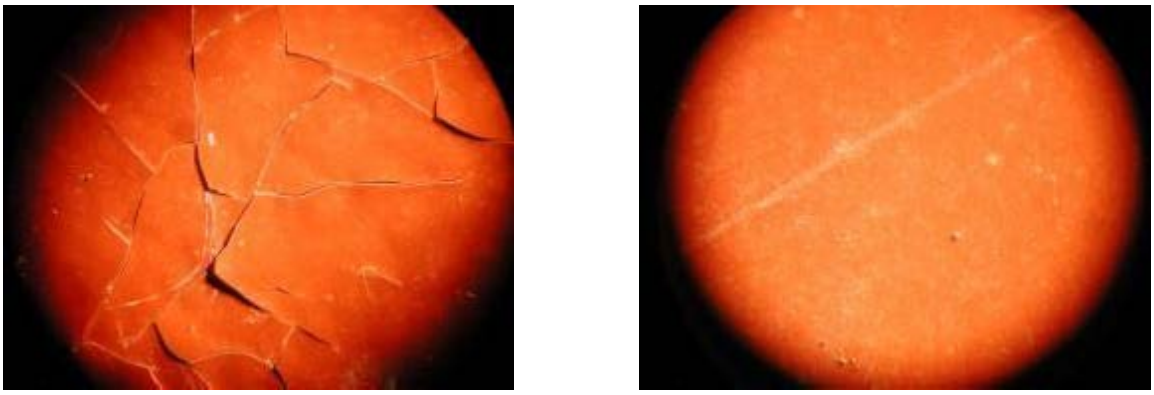


Figure 3e: Cracking Results - Panel 405 @ 40X Magnification
(Left: gas phase, Right: hydrocarbon phase)

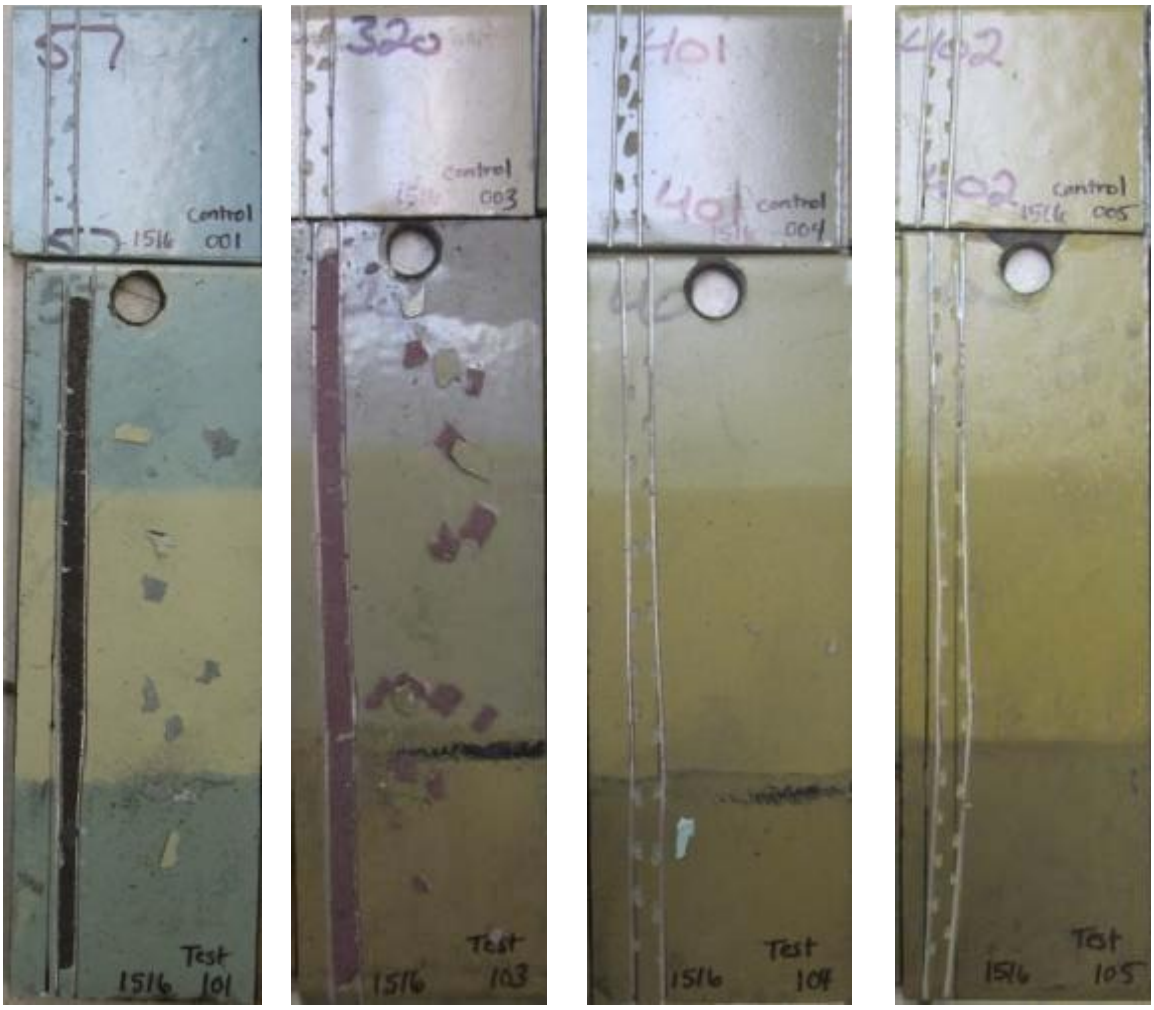


Figure 4a: Adhesion Test Results
(Left to Right: 57, 320, 401, 402)

Figure 4b: Adhesion Test Results
(Left to Right: 403, 404 and 405)

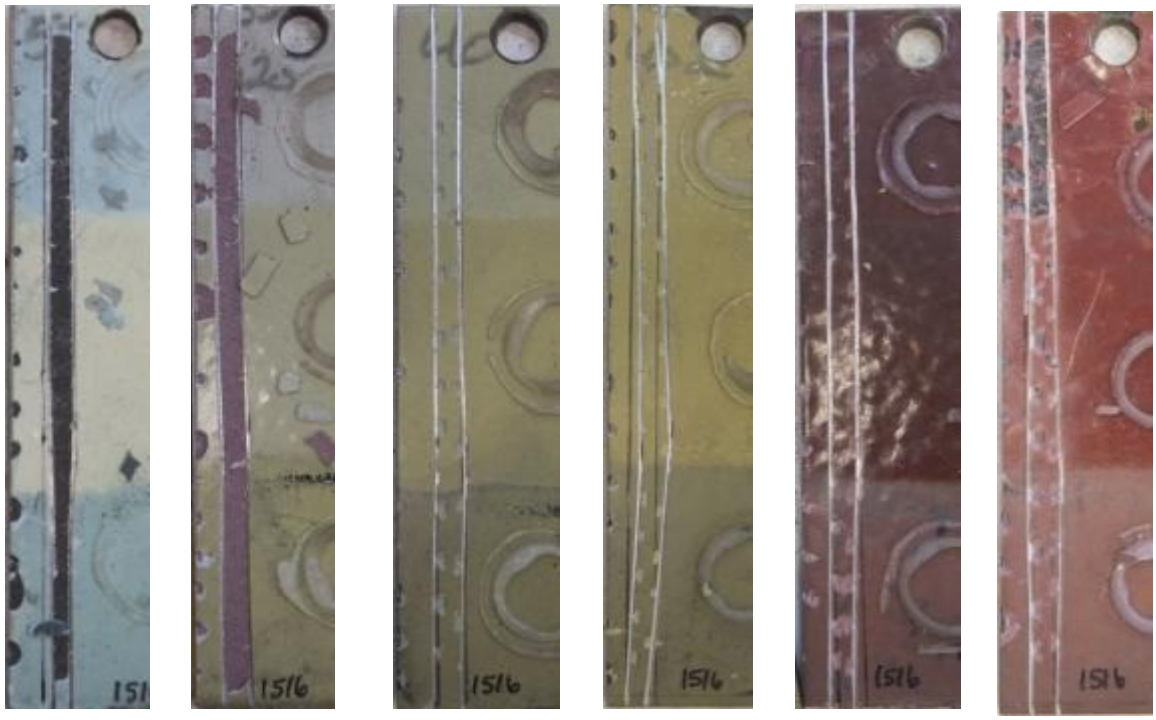


Figure 5: Undercreep Results
(Left to Right: 57, 320, 401, 402, 403 and 405)

Figure 6a: Foam Results for 57 @ 40X Magnification
(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

Figure 6b: Foam Results for 320 @ 40X Magnification

(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

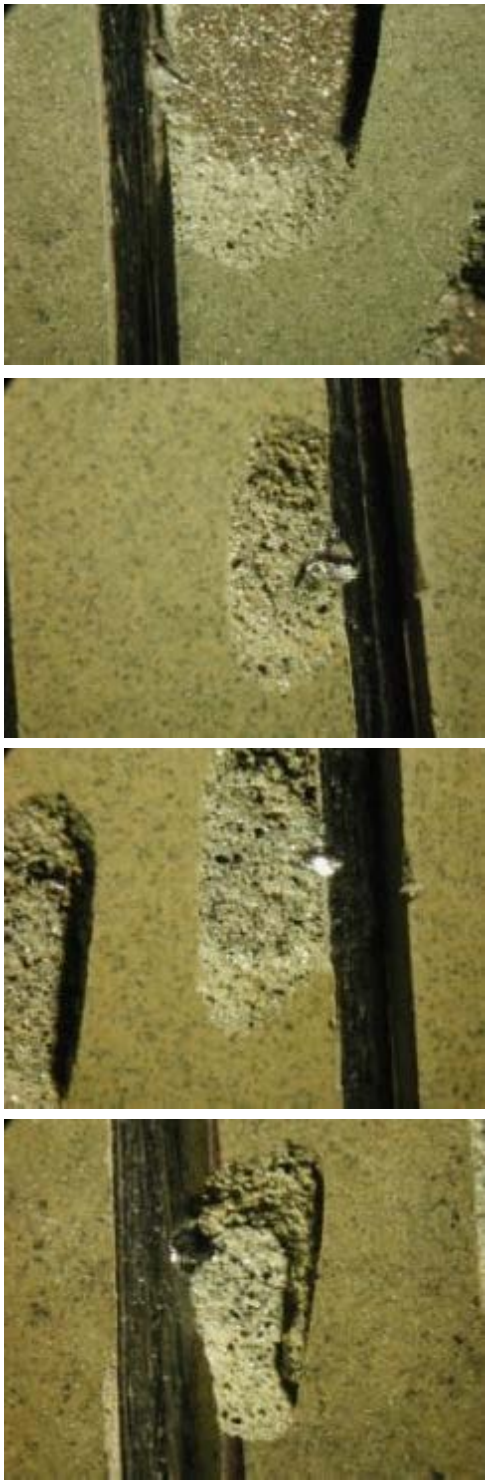


Figure 6c: Foam Results for 401@ 40X Magnification

(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

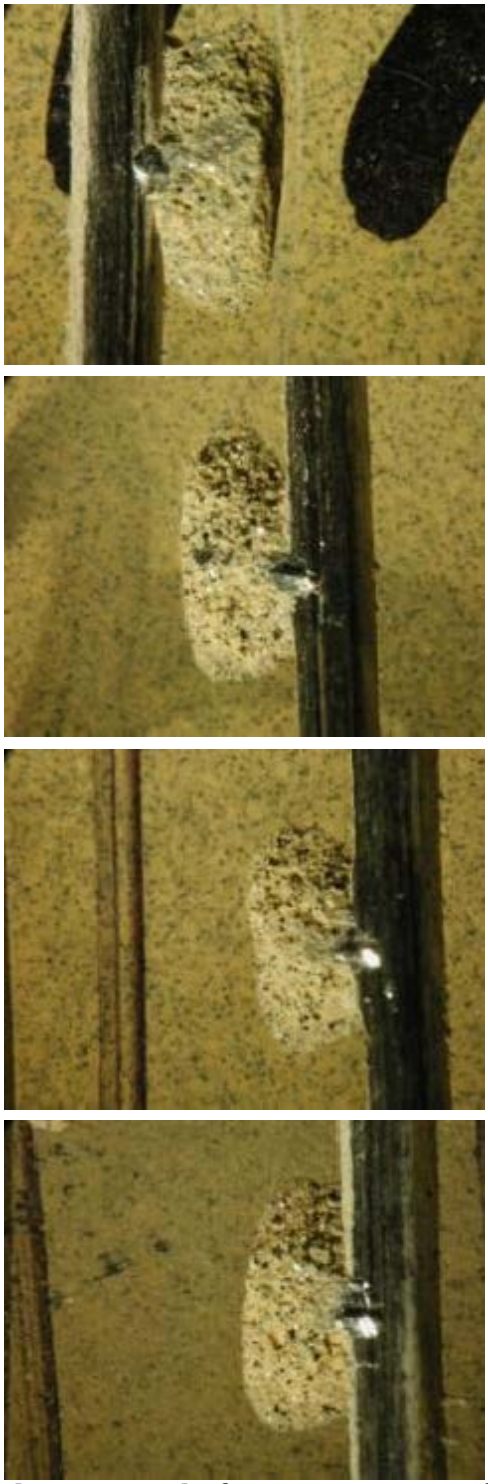


Figure 6d: Foam Results for 402@ 40X Magnification

(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

Figure 6e: Foam Results for 403@ 40X Magnification

(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

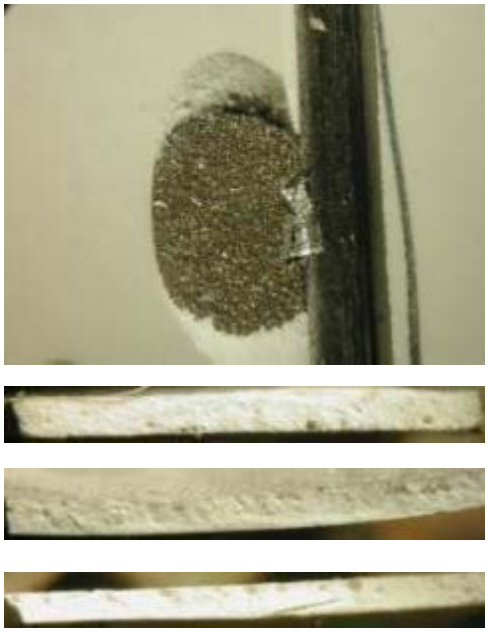


Figure 6f: Foam Results for 404@ 40X Magnification
(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

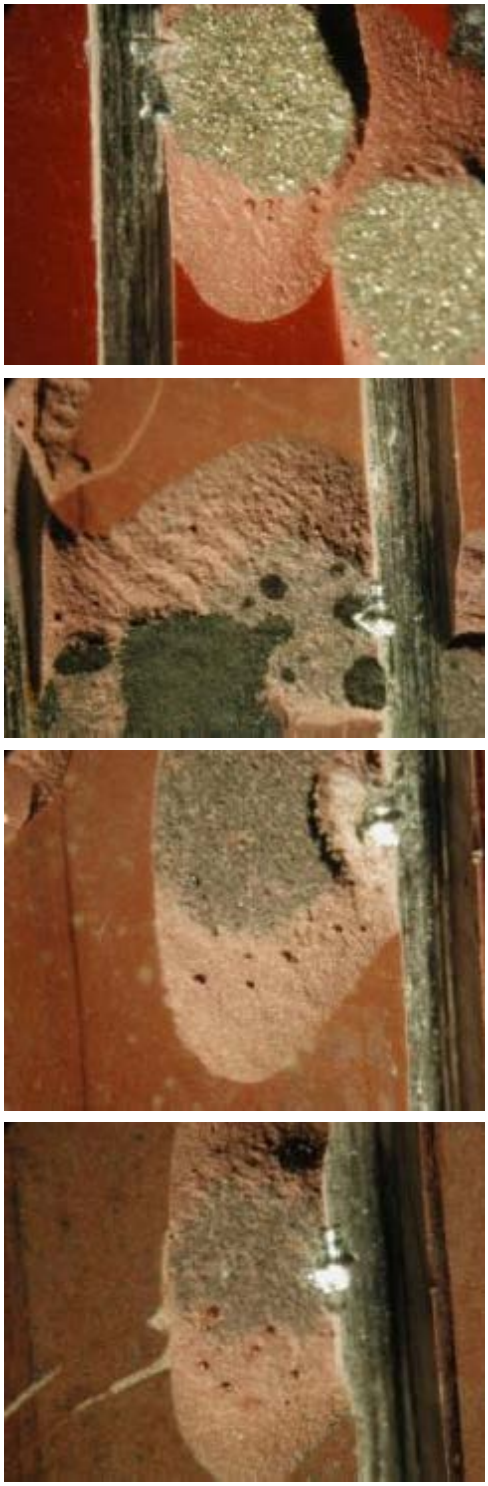


Figure 6g: Foam Results for 405@ 40X Magnification
(Top to Bottom: control, gas phase, hydrocarbon phase and aqueous phase)

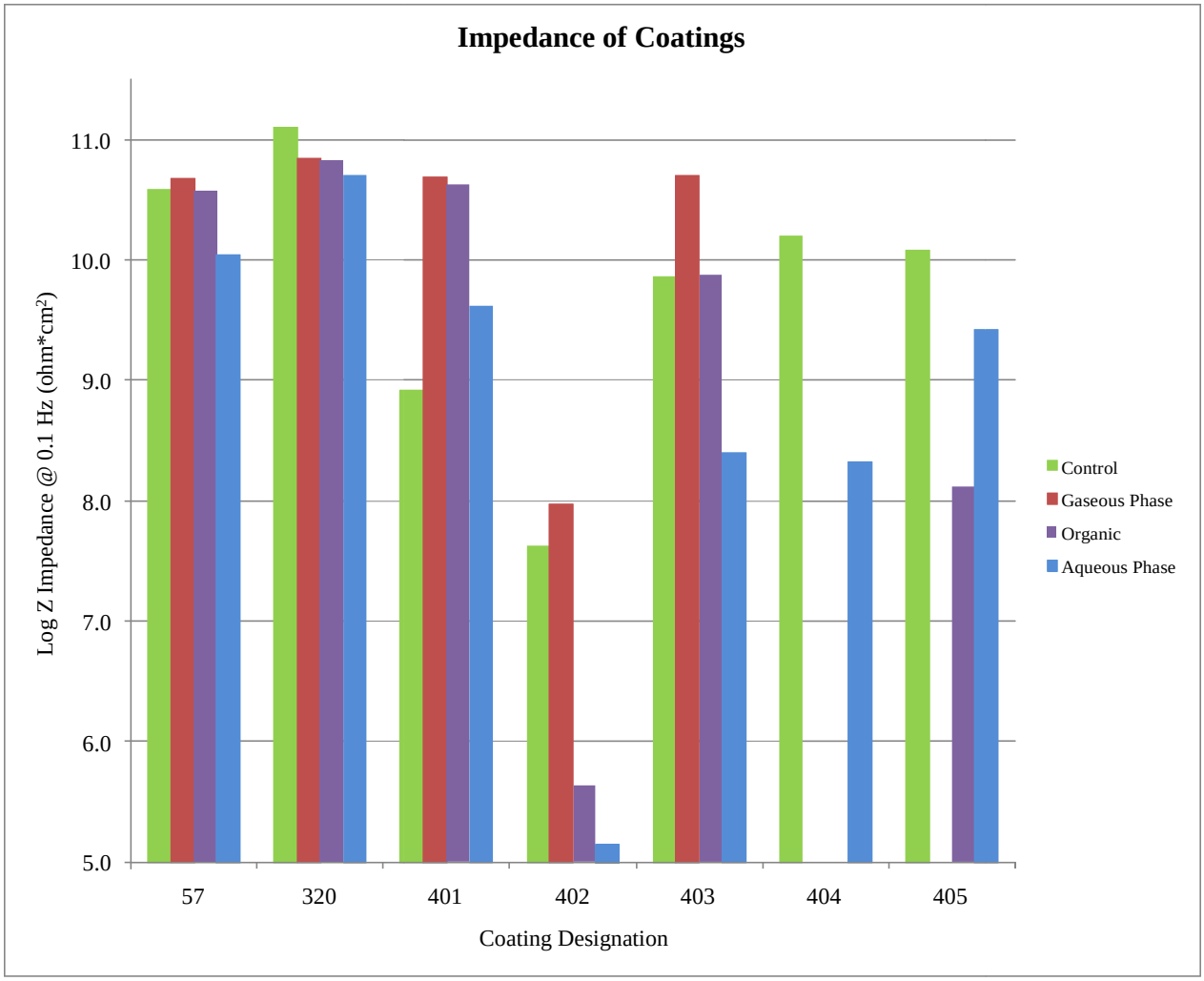


Table 3: EIS Results