Research Report
TIR 12-194

QUALIFICATION OF ASTM F1136 NON CHROME COATING (GEOMET® 321) FOR USE ON ASTM A490 HIGH STRENGTH STRUCTURAL BOLTS

TEST METHODOLOGY PER IFI-144

By

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EXECUTIVE SUMMARY

The objective of this investigation is to qualify ASTM F1136 non-chrome (NC) coatings, commercially known as GEOMET® 321, for use with ASTM A490 and A490M high strength structural bolts which are characterized by a tensile strength ranging from 150 to 173 ksi. ASTM F1136/F1136M, "*Zinc/Aluminum Corrosion Protective Coatings for Fasteners*," was previously approved in 2009 by Committee F16 on Fasteners for use on A490 structural bolts [1]. This current study is intended to supplement previous work [1,2] by conducting a full qualification in accordance with IFI-144 "*Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts*" of the non-chrome F1136 coatings, specifically GEOMET® 321. ASTM F1136/F1136M is a standard specification that is applicable to zinc-aluminum dispersion coatings, namely chrome containing DACROMET® and non-chrome GEOMET® licensed by NOF Metal Coatings North America, headquartered in Chardon, OH.

The GEOMET® 321 coating system satisfied the performance criteria tests specified in IFI-144, notably: coating thickness, coating adhesion, paintability, rotational capacity, salt spray exposure, and cyclic exposure tests. The most significant test results obtained in this study relate to the risk of internal hydrogen embrittlement (IHE) and environmental hydrogen embrittlement (EHE). Process qualification using ASTM F1940 methodology demonstrated that there is no risk of IHE. Product environmental testing of high strength specimen bolts, performed in accordance with ATSM F2660 exceeded the acceptance criteria thresholds established. These results demonstrated that GEOMET® 321 does not promote environmental hydrogen embrittlement (EHE) on standard ASTM A490 high strength structural bolts, regardless of size. The findings of this study support previous findings regarding GEOMET® [2]. Although no action is required by Committee F16 with respect to the current wording in ASTM A490 and A490M, it is recommended that the next revision of ASTM A490 and A490M, specifically in the form of a combined structural bolt standard, explicitly list both chrome and non-chrome versions of F1136 coatings as being permissible. This report is presented for review to committee F16 and subsequently will be made available via the ASTM F16 web page along with previous reports on the qualification of coatings A490 high strength structural bolts.

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1. OBJECTIVE

The objective of this investigation is to qualify ASTM F1136, non-chrome (NC) coatings for use with ASTM A490 and A490M[1] high strength structural bolts which are characterized by a tensile strength ranging from 150 to 173 ksi. ASTM F1136/F1136M, "*Zinc/Aluminum Corrosion Protective Coatings for Fasteners*," was previously approved in 2009 by Committee F16 on Fasteners for use on A490 structural bolts [1]. This current study is intended to supplement previous work [1,2] by conducting a full qualification in accordance with IFI-144 "*Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts*" of the non-chrome F1136 coatings, specifically GEOMET® 321, a zinc-aluminum dispersion coating licensed by NOF Metal Coatings North America, headquartered in Chardon, OH.

The primary concern that is intended to be addressed in qualifying coatings for use on A490 bolts is the risk of hydrogen embrittlement (HE), more precisely the risk of internal hydrogen embrittlement and environmental hydrogen embrittlement (EHE). IHE is a consequence of hydrogen introduced during the coating process. EHE can be accelerated by cathodically generated hydrogen during the corrosion reaction of a sacrificial coating. In the context of IFI-144, the risk of IHE is evaluated using ASTM F1940 "*Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners;*" EHE is evaluated using the methodology prescribed in ASTM F2660 "*Qualifying Coatings for Use on A490 Structural Bolts Relative to Hydrogen Embrittlement.*" Other tests required by IFI-144 are essentially methods for benchmarking the performance of a coating. They do not constitute acceptance criteria.

2. COATED SAMPLES

For the purpose of this study bolts, nuts and washers in two nominal inch sizes: 1-8 UNC, and 3/4-10 UNC were utilized. The test pieces were coated by the GEOMET® 321 process, in accordance with ASTM F1136 (NC). More precisely, bolts and washers were

[1] Any reference to ASTM 490 in this document also applies to ASTM A490M.

coated with GEOMET® 321 PLUS® (F1136 Grade 3 NC). Nuts were coated with GEOMET® 321 PLUS® XL (F1136 Grade 5 NC).

In addition to testing specimen bolts, the coating process itself was qualified in accordance with ASTM F1940 using certified notch bars that were coated at the same time as the bolts, nuts and washers.

2.1 Specimen bolts

All testing related to hydrogen embrittlement (HE) in accordance with ASTM F2660 was carried out using "*specimen bolts*" especially heat treated to achieve strength and hardness values that are slightly greater than the upper limits for A490 bolts. The specimen bolts were designed to simulate a worst case material condition with respect to susceptibility to environmental hydrogen embrittlement (EHE). For this reason, hardness and tensile values for specimen bolts exceed the maximum limits for standard A490 bolts. Additionally, "*standard A490 bolts"* of same sizes were used for other tests required by IFI-144. Examples of the specimen bolts are shown in Figure 1.

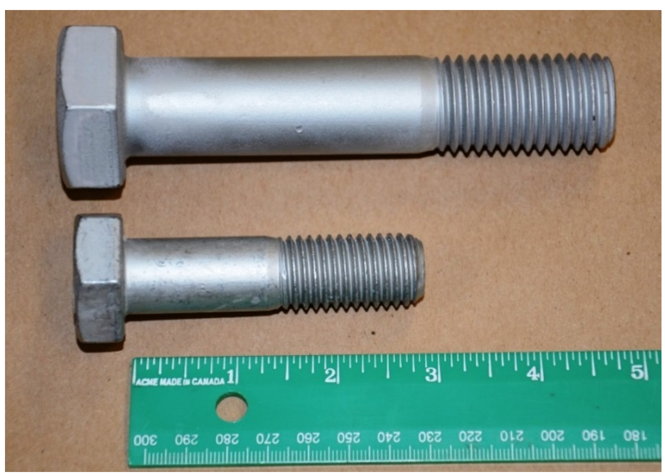


Figure 1 – Coated specimen bolts 1-8 x 5 and 3/4-10 x 3

Specimen bolts and standard A490 bolts used in this study comprised two nominal inch sizes: 1-8 x 5, and 3/4-10 x 3 and each of the four conditions were from homogeneous lots traceable to mill heats of *alloy* steel. Chemical composition for the specimens bolts are given in Table 1. Alloy steel grades were AISI 1340V and AISI 5135 for the 1 inch and 3/4 inch specimen bolts respectively, and AISI 1340V and AISI 1335V for the 1 inch and 3/4 inch standard A490 bolts respectively.

Table 1 – Chemical composition of test bolts

	Wt % Conc.			
	Specimen Bolts		Standard A490 Bolts	
	1-8 x 5	3/4-10 x 3	1-8 x 5	3/4-10 x 3
Carbon	0.40	0.36	0.40	0.38
Manganese	1.65	0.71	1.61	1.65
Phosphorus	0.012	0.020	0.011	0.015
Sulfur	0.013	0.015	0.018	0.016
Silicon	0.21	0.23	0.20	0.24
Molybdenum	0.023	-	-	-
Chromium	0.10	0.85	-	-
Vanadium	0.023	-	0.030	0.030
AISI Designation	**1340V**	**5135**	**1340V**	**1335V**

Mechanical properties for the specimen bolts and A490 bolts are given in tables 2-3 and 5-6. The average measured mid-radius hardness values for the 1 inch and 3/4 inch specimen bolts were 38.5 and 40.9 HRC respectively. The specified hardness range in ASTM A490 is 33-38. Average wedge tensile strength values for the specimen bolts were roughly 178 and 182 ksi respectively. The maximum allowable wedge tensile strength in ASTM A490 is 173 ksi. In both cases, the hardness and tensile strength of the specimen bolts exceed the hardness and tensile strength of standard A490 bolts. Based on the measured hardness and tensile strength values, it is clear that the specimen bolts significantly exceed the worst case scenario in terms of susceptibility to environmental hydrogen embrittlement for standard A490 bolts. Wedge tensile strength values after coating was verified for the 3/4-10 x 3 specimen bolts, confirming that the GEOMET® 321 coating process does not alter the mechanical properties of the specimen bolts (Table 4).

Table 2 – Hardness values of specimen bolts

	1-8 x 5	
Sample	**Mid-radius (HRC)**	**Surface (HR 30N)**
1	38.6	58
2	38.4	59
Avg.	**38.5**	**58.5**

	3/4-10 x 3	
Sample	**Mid-radius (HRC)**	**Surface (HR 30N)**
1	41.0	61.0
2	40.8	60.5
3	40.7	60.9
4	40.8	59.6
5	41.1	59.8
Avg.	**40.9**	**60.4**

Table 3 – Full sized wedge tensile test values of specimen bolts

Sample	1-8 x 5	
	Load (lbf)	Stress (psi)
1	108,510	179,148
2	107,580	177,613
Avg.	**108,045**	**178,381**

Tensile Stress Area 0.6057 in^2

Sample	3/4-10 x 3	
	Load (lbf)	Stress (psi)
1	61,530	184,222
2	59,660	178,623
3	60,530	181,228
4	61,390	183,802
5	61,590	184,401
Avg.	**60,940**	**182,455**

Tensile Stress Area 0.3340 in^2

Table 4 – Full sized wedge tensile test values on 3/4-10 x 3 specimen bolts after coating illustrating no change in tensile strength

Sample	3/4-10 x 3	
	Load (lbf)	Stress (psi)
1	60,350	180,689
2	60,920	182,395
3	60,580	181,377
4	60,250	180,389
5	61,700	184,731
Avg.	**60,760**	**181,916**

Tensile Stress Area 0.3340 in^2

Table 5 – Hardness values of standard A490 bolts

1-8 x 5	
Sample	Mid-radius (HRC)
1	34.5
2	34.4
3	34.6
Avg.	**34.5**

3/4-10 x 3	
Sample	Mid-radius (HRC)
1	34.8
2	35.9
3	35.3
Avg.	**35.3**

Table 6 – Full sized wedge tensile test values of standard A490 bolts

	1-8 x 5	
Sample	Load (lbf)	Stress (psi)
1	99,306	159,000
2	99,335	164,000
Avg.	**99,321**	**163,976**

Tensile Stress Area 0.6057 in^2

	3/4-10 x 3	
Sample	Load (lbf)	Stress (psi)
1	54,776	164,000
2	54,765	163,967
Avg.	**54,771**	**163,984**

Tensile Stress Area 0.3340 in^2

2.2 Notch bar specimens

In addition to testing specimen bolts, the coating process itself was qualified in accordance with ASTM F1940, "*Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners*." In this procedure, standard notch bar specimens made of AISI 4340 steel, heat treated to 50-52 HRC were coated along with production bolts, and were tested using the incremental step load protocol prescribed in ASTM F1940 (Fig. 2). The dimensions and appearance of the notch bar specimens are shown in figures 2-3. The chemical composition of the lot of notch bars used in this study is given in Table 7.

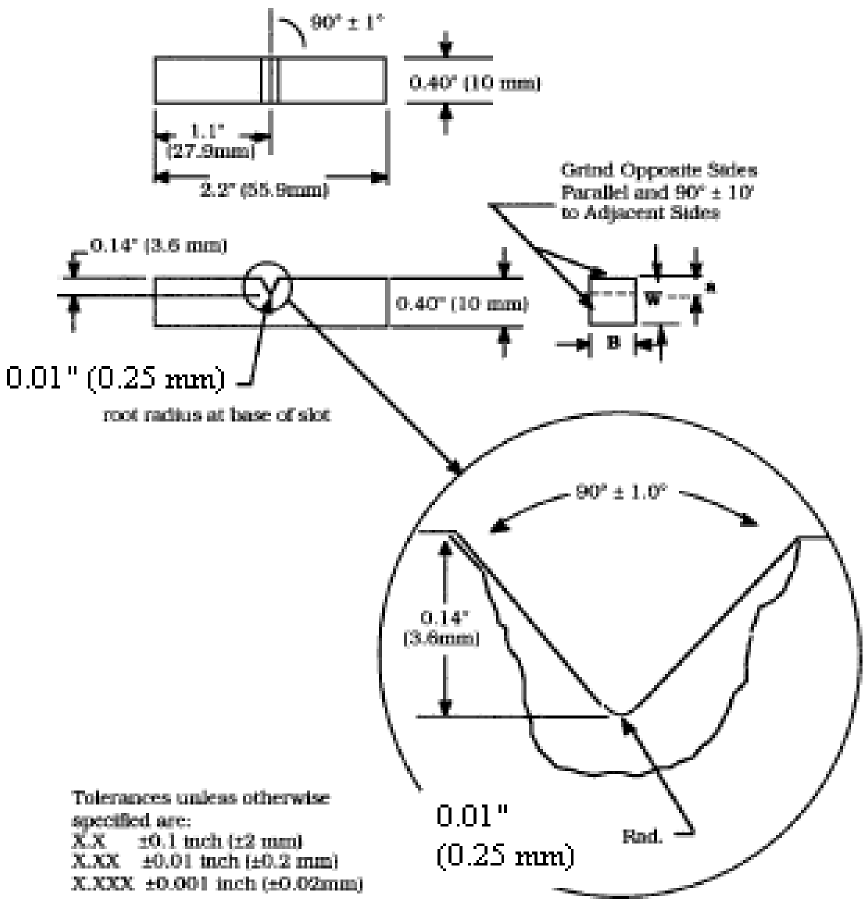


Figure 2 – ASTM F1940 notch bar specimen configuration

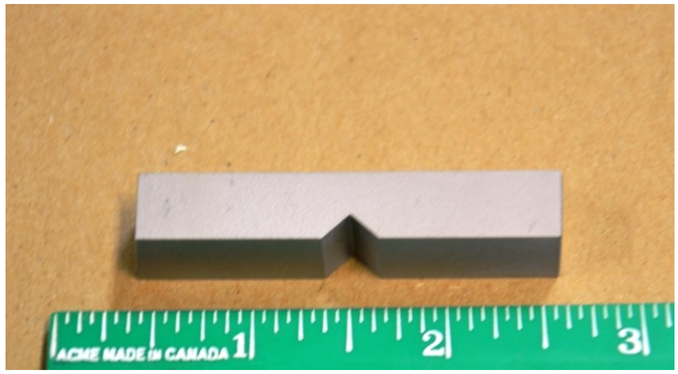


Figure 3 – Coated ASTM F1940 notch bar specimen

Table 7 – Chemical composition of ASTM F1940 notch bar specimens

	Actual	**AISI 4340 requirements**
Carbon	0.42	0.38-0.43
Manganese	0.75	0.65-0.85
Phosphorus	0.006	0.025 max.
Sulphur	0.003	0.025 max.
Silicon	0.27	0.15-0.30
Copper	0.21	-
Nickel	1.75	1.65-2.00
Chromium	0.79	0.70-0.90
Molybdenum	0.25	0.20-0.30

Note: The average measured hardness of the lot of notch bars used in this study was 52.2 HRC.

3. GEOMET® 321 COATING PROCESS

GEOMET® 321 is a proprietary coating system licensed by NOF Metal Coatings North America based in Chardon, OH. This coating system applies a zinc/aluminum inorganic base coat that can be combined with an additional topcoat that can act as friction modifier and additional passivation layer. The coating thickness by this process can range from 10 to 20 µm, and the coverage is relatively uniform.

The test bolts (i.e., specimen bolts and standard A490 bolts), nuts, and washers were coated by the GEOMET® 321 process, in accordance with ASTM F1136 (NC). For the purpose of this study a rack spray process was used to coat the 1 inch bolts, and a dip spin process was used for the 3/4 inch bolts. The nuts and washers (1 inch and 3/4 inch) were coated by the dip spin process. The GEOMET® basecoat was applied in two layers for the rack-spray application and in three layers for the dip spin application. The coatings were performed under normal operating conditions in accordance with the procedures prescribed by NOF Metal Coatings North America for GEOMET® 321, and in accordance with ASTM F1136. In each process, the test parts were accompanied by *witness* ASTM F1940 notch bars that were coated at the same time. Note that surface preparation consisted of alkaline degreasing followed by grit blasting. Therefore the parts were not exposed to any hydrogen during the coating process. Figures 4 and 5 illustrate the process flowcharts for the dip spin and rack GEOMET® 321 processes. Coating details are provided below.

Coatings:

1 inch Bolts: GEOMET® 321/PLUS per ASTM F1136 Grade 3
Basecoat + clear sealer (PLUS®)
Applied by spray coating

3/4 inch Bolts: GEOMET® 321/PLUS® per ASTM F1136 Grade 3
Basecoat + clear sealer (PLUS®)
Applied by dip spin coating

All nuts: GEOMET® 321/PLUS® XL per ASTM F1136M Grade 5
Basecoat + lubricated sealer (PLUS® XL)
Applied by dip spin coating

All washers: GEOMET® 321/PLUS® per ASTM F1136M Grade 3
Basecoat coating + clear sealer (PLUS®)

Applied by dip spin coating

Coating applicators:

Dip Spin:
Michigan Metal Coatings Company, Port Huron, MI
www.michiganmetalcoatings.com

Rack spray:
Allegheny Coatings, Ridgway, PA
www.alleghenycoatings.com

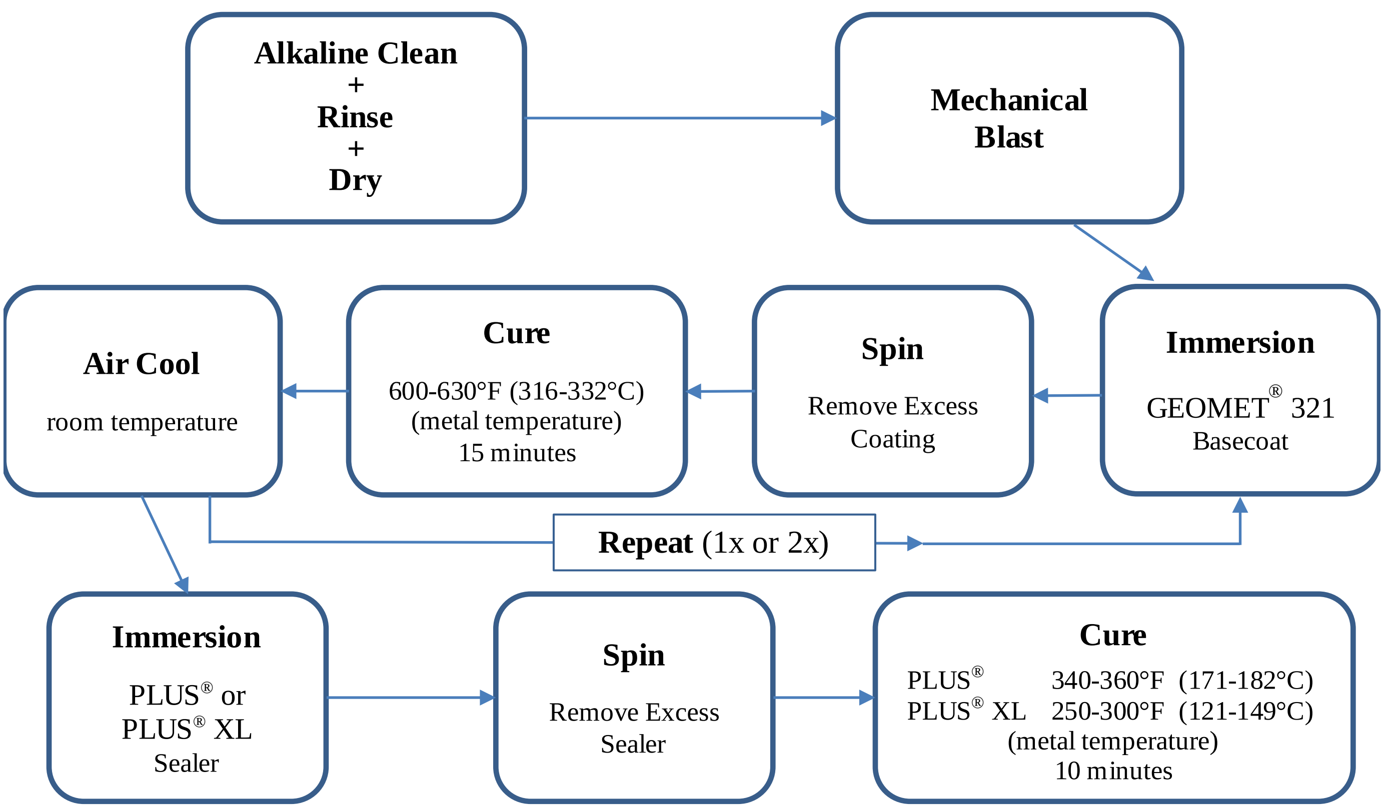


Figure 4 –Dip Spin GEOMET® 321 /Sealer Processing Steps

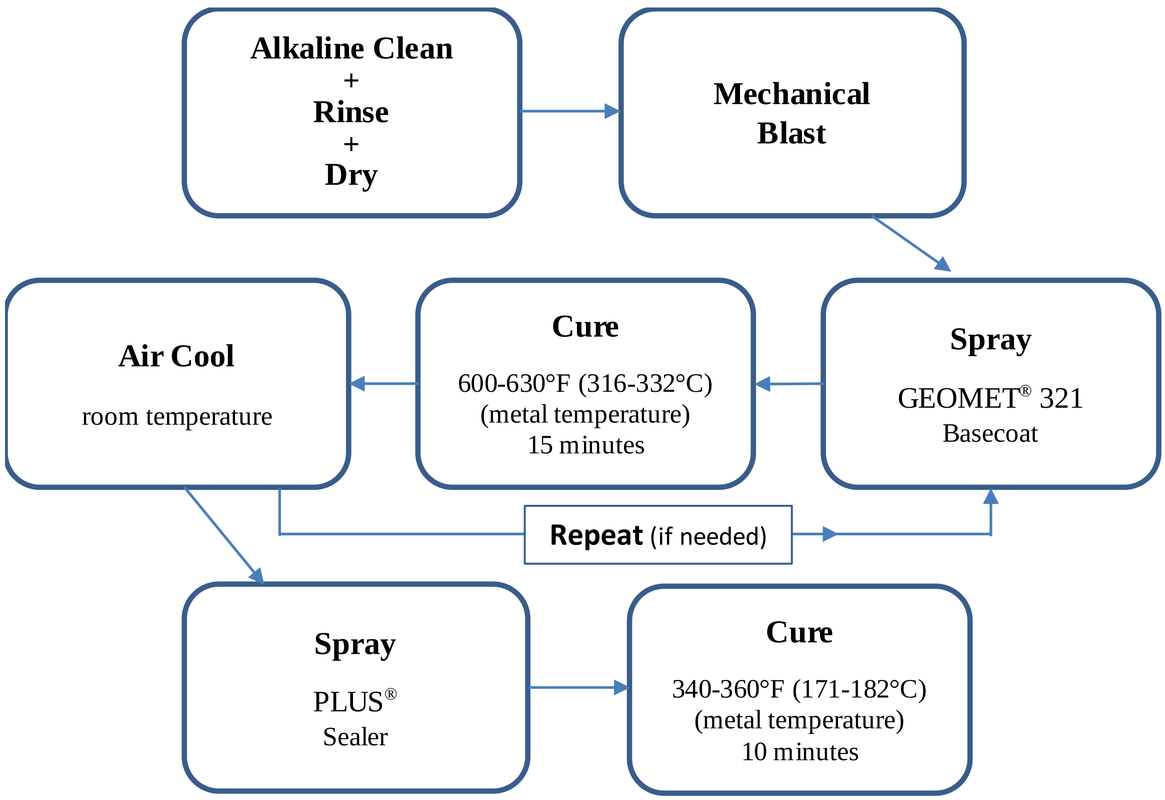


Figure 5 – Rack Spray GEOMET® 321 /Sealer Processing Steps

4. EXPERIMENTAL METHODS

The following table (Table 8) lists the test methodologies required by IFI-144 and applied in this study. Relevant descriptive information about each test method will be given in the “Results” section.

Table 8 – IFI-144 qualification test methods

Sect.	Test	Specification	Test Parts Used
2	Hardness	ASTM F606	Specimens & Standard A490
2	Chemical Analysis	ASTM A751	Specimens & Standard A490
5.1	Microstructure	ASTM E3	Specimens
5.2	Coating Thickness	ASTM D1186	Standard A490
5.3	Adhesion	ASTM B571	Standard A490
5.4	Paintability	Visual	Standard A490
5.5	Rotational Capacity	FHWA/AASHTO	Standard A490
5.6	Salt Spray Exposure	ASTM B117 / ASTM D1654	Standard A490
5.7	Cyclic Exposure (conducted on bolts assembled in test blocks)	GM9540P	Standard A490
5.8	Post Cyclic Exposure Tensile Pull	ASTM F606	Standard A490
5.9	Hydrogen Embrittlement (process)	ASTM F1940	F1940 notch square bars
5.10	Hydrogen Embrittlement (product)	ASTM F2660	Specimens

5. RESULTS & DISCUSSION

5.1 Microstructure

The metallurgical structure of the specimen bolts was fully transformed tempered martensite, which is per the requirements for A490 bolts. SEM images of the microstructures are given in Figure 6 and 7 respectively.

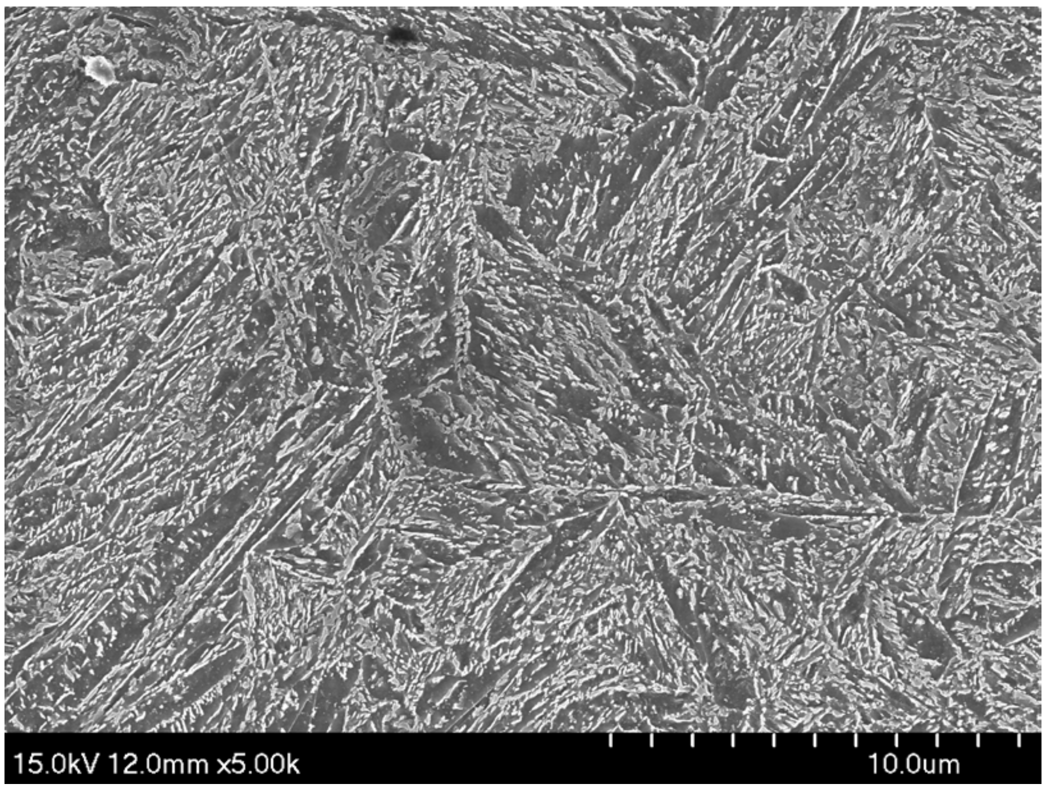


Figure 6 – Microstructure 1-8 x 5 specimen bolts (5,000 X)

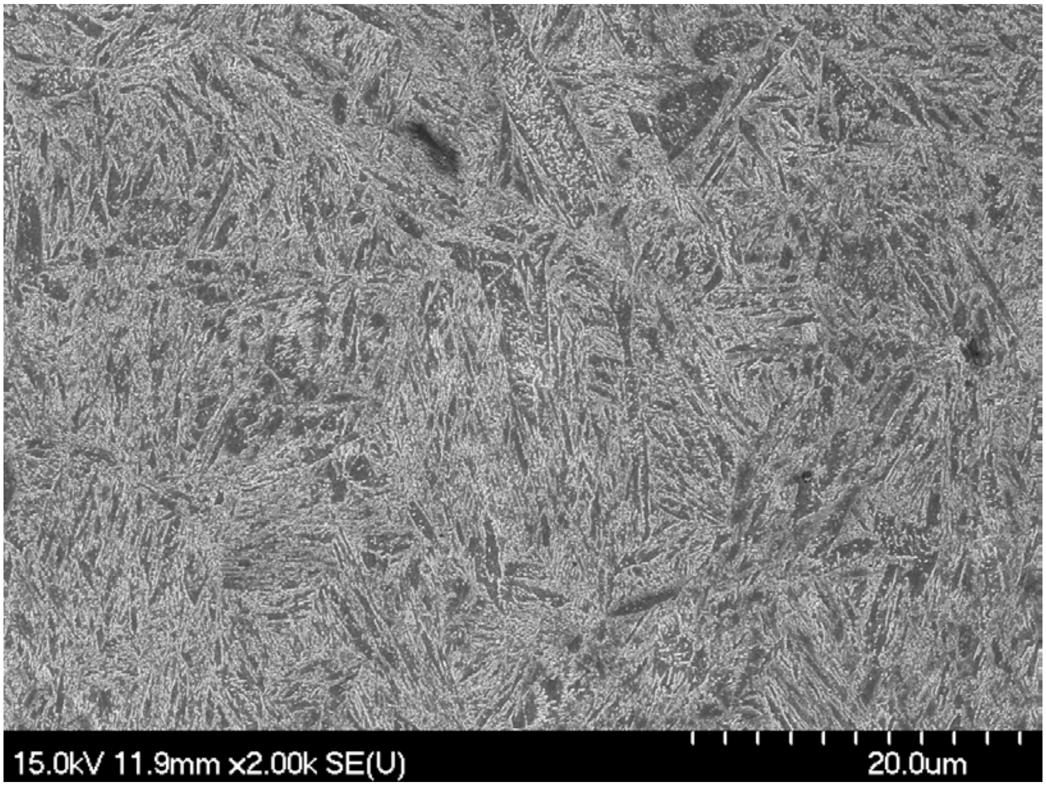


Figure 7 – Microstructure 3/4-10 x 3 specimen bolts (5,000 X)

5.2 Coating thickness

Coating thickness was measured in accordance with ASTM D1186 using a Fisherscope MMS magnetic induction tester. The sampling size consisted of ten 1 inch bolts and six of all other part types and sizes. Surfaces measured were the top of the head and each hexagonal face of 3/4 inch bolts, the unthreaded portion of the shaft of 1-inch bolts, the six hex faces of all nuts, and both flat sides of all washers. A minimum of 10 individual measurements were done per surface. For all parts, the average thickness exceeded the required minimum of 0.24 mil (6 microns) specified in F1136 Grade 3 (bolts and washers) and F1136 Grade 5 (nuts) (Figure 8). The specified coating thickness range in ASTM F1136 for Grade 3 and Grade 5 coatings is 0.24-0.47 mil (6-12 µm).

The measured thickness values are consistent with the basecoat coating weight of roughly 40 g/m^2 which is greater than the typical target of 24 g/m^2. It was anticipated that thicker coating would improve the corrosion performance, but it would also maximize total galvanic current available over the life of the coating, thus making the environmental hydrogen embrittlement (EHE) conditions more severe.

Ensuring satisfactory thread fit between mating nuts and bolts must be accommodated at the discretion of the manufacturer of the nuts by providing an oversize allowance, and further verified by the after application of the coating. At the time of writing of this report, committee F16 was undertaking an initiative to develop standard oversize allowances for nuts used with A490 bolts coated by Zn/Al dispersion coatings.

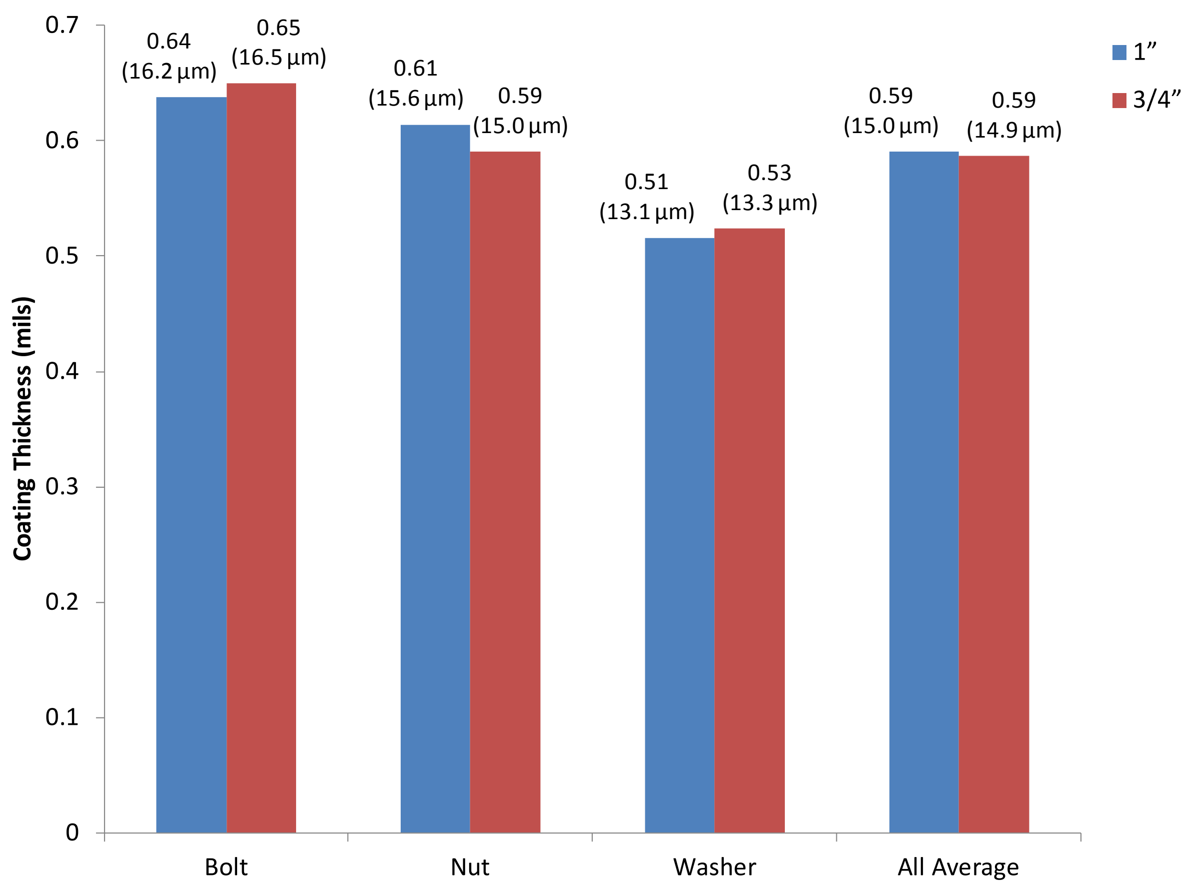


Figure 8 – Average coating thickness values – bolts, nuts, washers

5.3 Adhesion

IFI-144 stipulates adhesion testing in accordance with ASTM B571. Five coated bolts of each size (3/4 inch and 1 inch) were scribed in 3 parallel lines with sufficient pressure to penetrate the coating to the steel substrate as shown in Figure 9, followed by application of 3M 610 clear tape with firm finger pressure and rapid removal at an angle of 180°. The bond strength of 3M 610 tape is 47N/100mm width. No continuous portion of the coating between the parallel lines broke away from the substrate, indicating satisfactory adhesion, as shown in figures 10-11.

Figure 9 – Example of scribed bolt

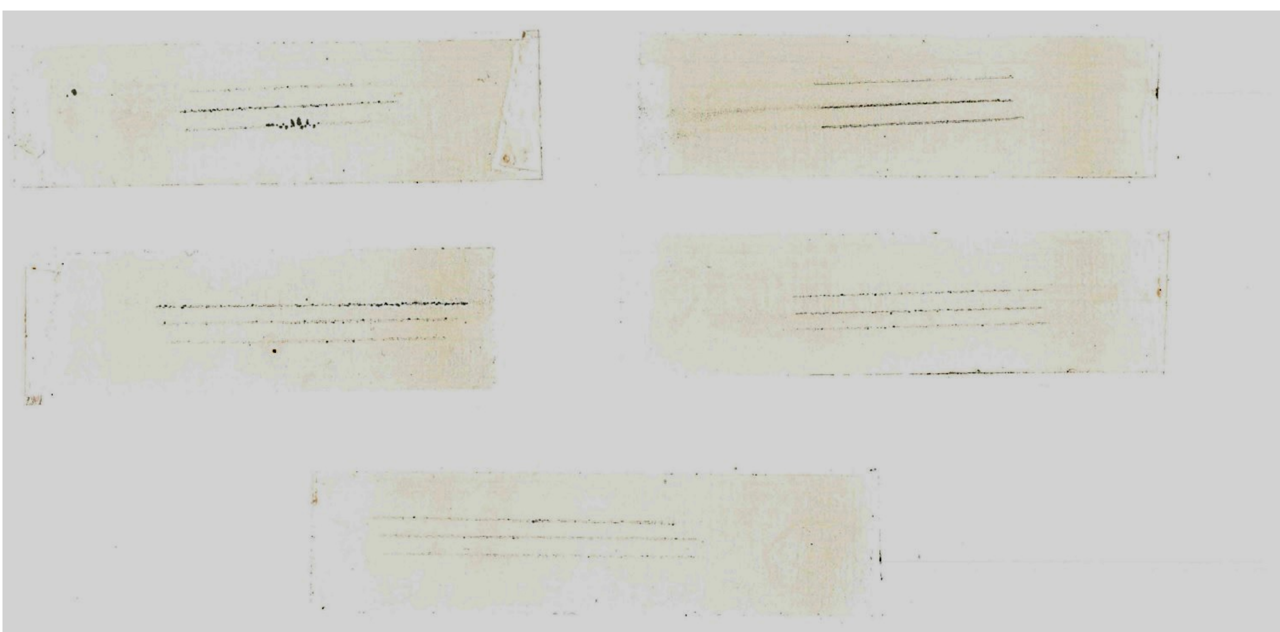


Figure 10 – 3M 610 tape samples showing satisfactory adhesion on 1-8 x 5 bolts

Figure 11 – 3M 610 tape samples showing satisfactory adhesion on 3/4-10 x 3 bolts

5.4 Paintability

IFI-144 stipulates, “Paint shall be applied to the fastener which is coated with the proposed material coating seeking qualification. Paint may be applied by spraying or brushing. After 48 hours, the painted fastener shall be dry to the touch.”

The paint system used for this test was Carbozinc 11 primer with Carboxane 2000 topcoat, which is commonly used in structural applications such as bridges. This is the same coating system that was used for A490 approval of the DACROMET P coating. The primer and topcoat paint were each applied to the GEOMET® 321P coated bolts (heads, threads, and shaft) and to the GEOMET® 321XL coated nuts by brushing. Average total coating thickness, measured by magnetic induction using a Fisherscope MMS instrument, was 175 microns (6.9 mils). After 48 hours at room temperature, the coating was verified to be dry to the touch, fulfilling the requirement (Fig. 12).

Although not required by IFI-144, an additional adhesion test was done per ASTM D3359 Procedure A. An X-scribe was made on the head of bolts and the hex flat of nuts, penetrating through the paint layers to expose the substrate (Fig. 12). The 3M 610 tape was then applied over the cut and removed rapidly at an angle close to 180°. The tapes were rated in accordance with ASTM D3359. Bolts were rated 4A to 5A, and nuts were rated 3A to 5A. This adhesion is acceptable by industry standards.[2]

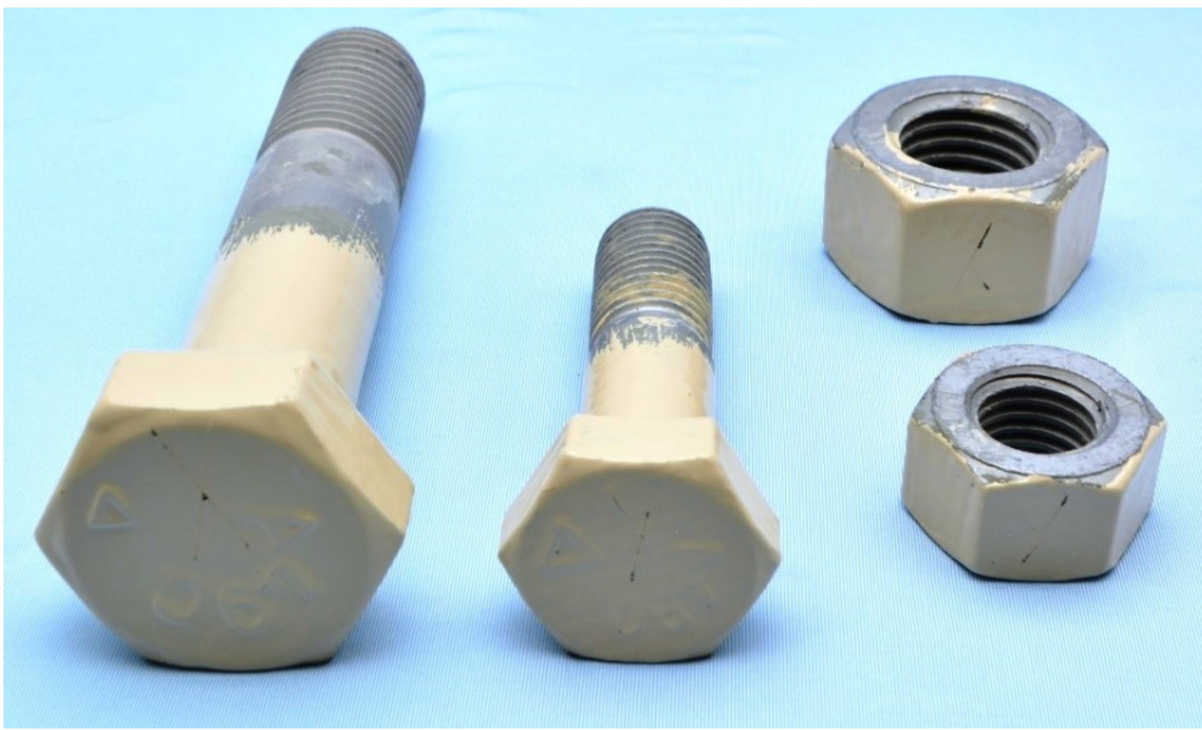


Figure 12 – Primed and painted bolt were dry to the touch after 48 h curing at room temperature. Scribes are visible on the head

[2] Rating of 5A means "no removal," 4A means "trace peeling or removal along incisions or at their intersections," and 3A means "jagged removal along incisions up to 1.6 mm on either side," 2A means "jagged removal along most of incisions up to 3.2 mm on either side," 1A means "removal from most of the area of the X," 0A means "removal beyond the area of the X."

5.5 Rotational capacity

The rotational capacity (RC) tests were performed in accordance with industry standards, notably in accordance with requirements by The American Association of State Highway and Transportation Officials (AASHTO) and by the Federal Highway Administration (FHWA). The RC tests were performed on GEOMET® 321P coated *standard A490* bolts, nuts and washers. The test parameters for *pre-installation verification* were in accordance with the Research Council on Structural Connections (RCSC) Specification for Structural Joints Using High-Strength Structural Bolts. The length (L) to diameter (d) ratios for the specimen bolts are shown in Table 9. The test parameters for *rotational capacity* testing were in accordance with AASHTO and FHWA (see Table 10).

The coated parts comfortably passed the RC test criteria, despite the fact that no allowance was made for coating thickness between the mating bolts and nuts. In other words, the conditions tested were more severe in terms of friction. A closer look at measured nut factor (K) values at installation tension illustrates the friction reducing effect of GEOMET® 321P (bolts-washers) used in combination with GEOMET® 321XL (nuts). The K value range at installation tension was 0.106 - 0.113 for 1 inch parts, and 0.121 – 0.137 for 3/4 inch parts. These relatively low K values explain the ease with which the assemblies satisfied the RC test requirements. It follows that torque values were well below maximum allowable torque at installation tension, as defined per AASHTO and FHWA[3]. The results for both test sizes are shown in Figures 13 and 14 respectively. For each size the data are presented in two charts: (i) angle vs. tension, and (ii) torque vs. tension.

Table 9 – length over diameter ratios for specimen bolts

Bolt diameter d (in)	Length L (in)	L/d
1.00	5	5
0.75	3	4

[3] Maximum allowable torque values at the end of the RC test are calculated using a K value of 0.25. Refer to Table 12.

Table 10 – Pre-installation verification testing and acceptance criteria

Bolt size (in)	Snug tension[4] (kips)	Min. installation tension[5] (kips)	Min. pre-installation verification tension[6] (kips)
1-8	10	64	67
3/4-10	5	35	37

Bolt Length (L)	Nut rotation from snug tension[7]
$L \leq 4d$ (not more than 4d)	120° (1/3 turn)
$4d < L \leq 8d$ (more than 4d but not more than 8d)	180° (1/2 turn)
$8d < L \leq 12d$ (more than 8d but not more than 12d)	240° (2/3 turn)

Table 11 – Rotational capacity testing and acceptance criteria

Bolt size (in)	Initial tension[8] (kips)	Installation tension[9] (kips)	Max. torque at installation tension[10] (ft-lb)	Min. final tension[11] (kips)
1-8	6	64	1,333	74
3/4-10	4	35	547	40

Bolt length (L)	Nut rotation from initial tension
$L \leq 4d$ (not more than 4d)	240˚ (2/3 turn)
$4d < L \leq 8d$ (more than 4d but not more than 8d)	360˚ (1 turn)
$8d < L \leq 12d$ (more than 8d but not more than 12d)[12]	480˚ (1-1/3 turn) or 420˚ (1-1/6 turn)

[4] Equal to 15% of minimum installation tension, rounded off to nearest kip.

[5] Equal to 70% of minimum specified tensile strength of bolts, rounded off to nearest kip. Used for design, actual installation and inspection.

[6] Equal to 1.05 times the minimum installation tension values, rounded to the nearest kip.

[7] Nut rotation tolerance -0 +30 degrees, per Caltrans Special Provisions.

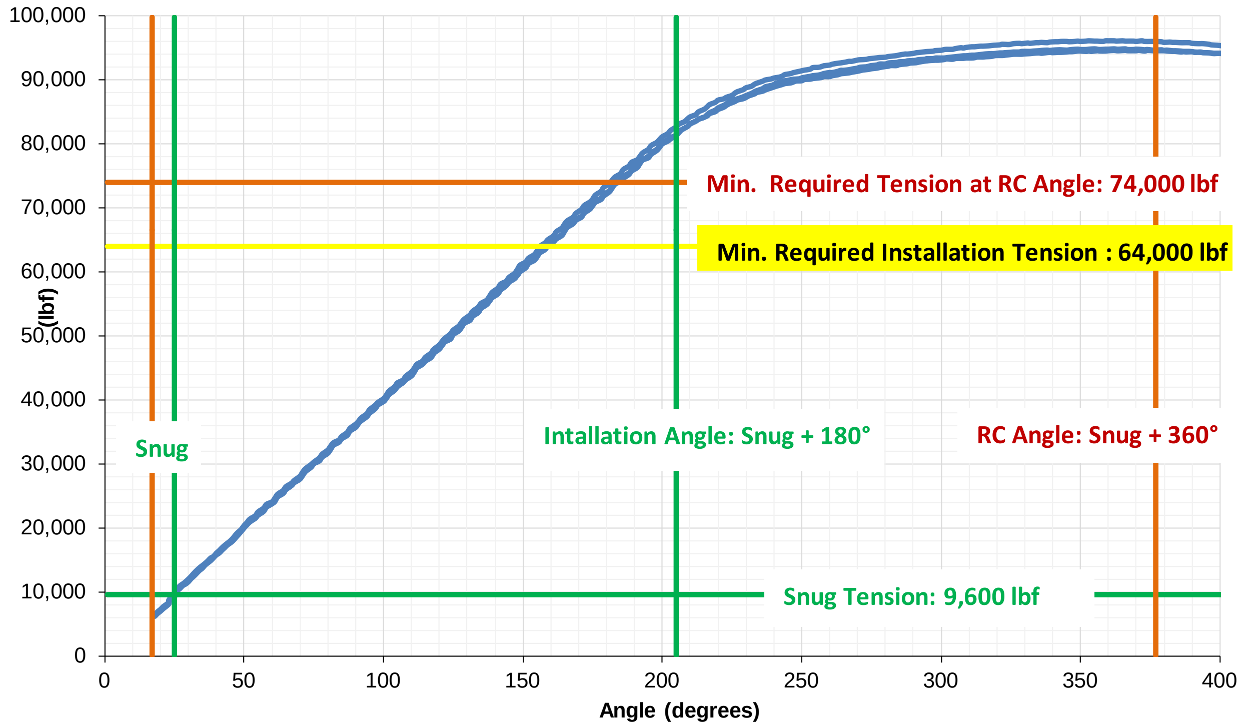
[8] Equal to 10% of installation tension, rounded off to nearest kip.

[9] Equal to 70% of minimum tensile strength of bolts, rounded off to nearest kip.

[10] T= 0.25 PD, where T= max. torque (ft-lb) P= tension (lbf) D= bolt diameter (ft).

[11] Equal to 1.15 times the minimum installation tension values, rounded to the nearest kip.

[12] At the time of this investigation, ASSHTO and FHWA requirements were identical with the exception of angles of rotation for bolt lengths greater than 8 diameters which were: 480° by AASHTO versus 420° by FHWA.



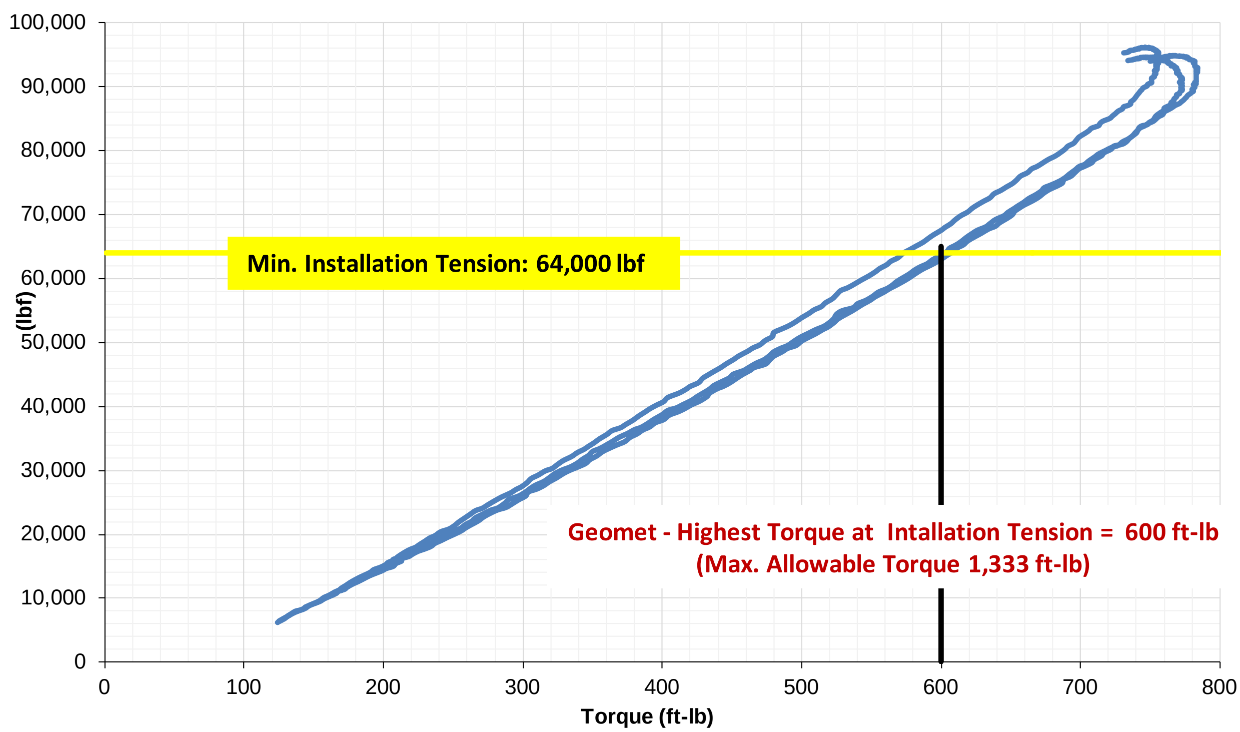
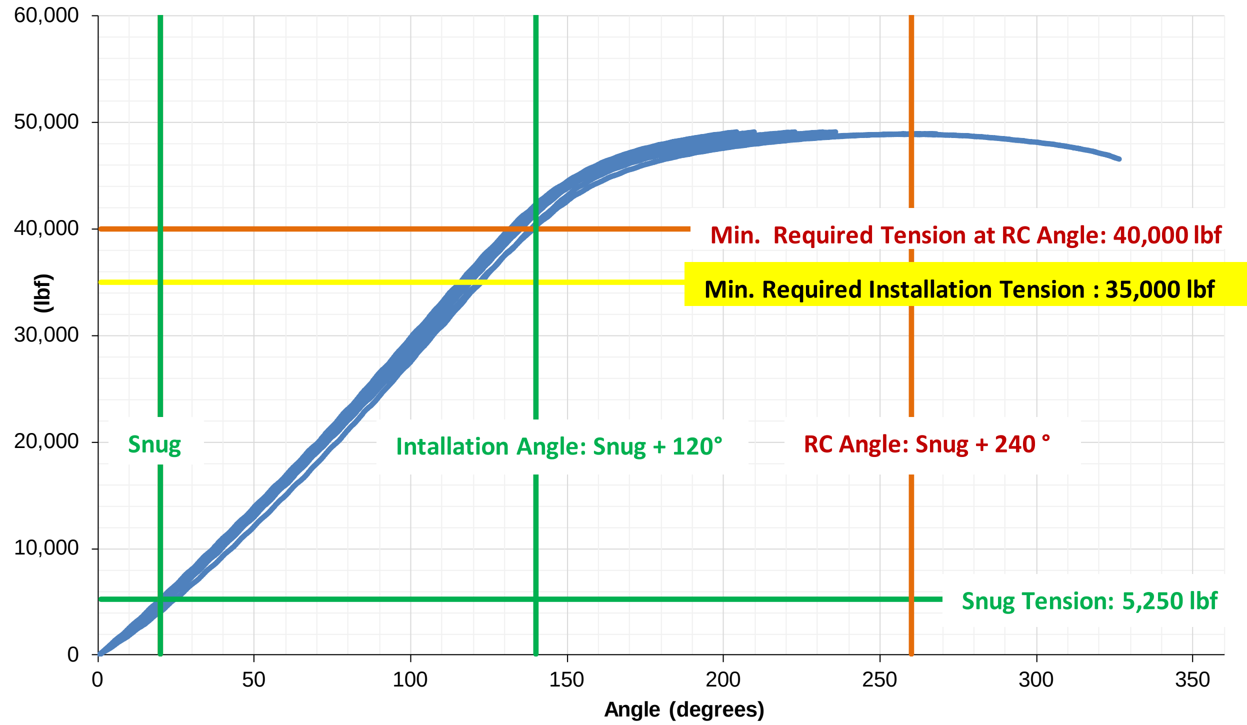


Figure 13 – RC test data for 1 inch parts: (i) angle vs. tension, and (ii) torque vs. tension



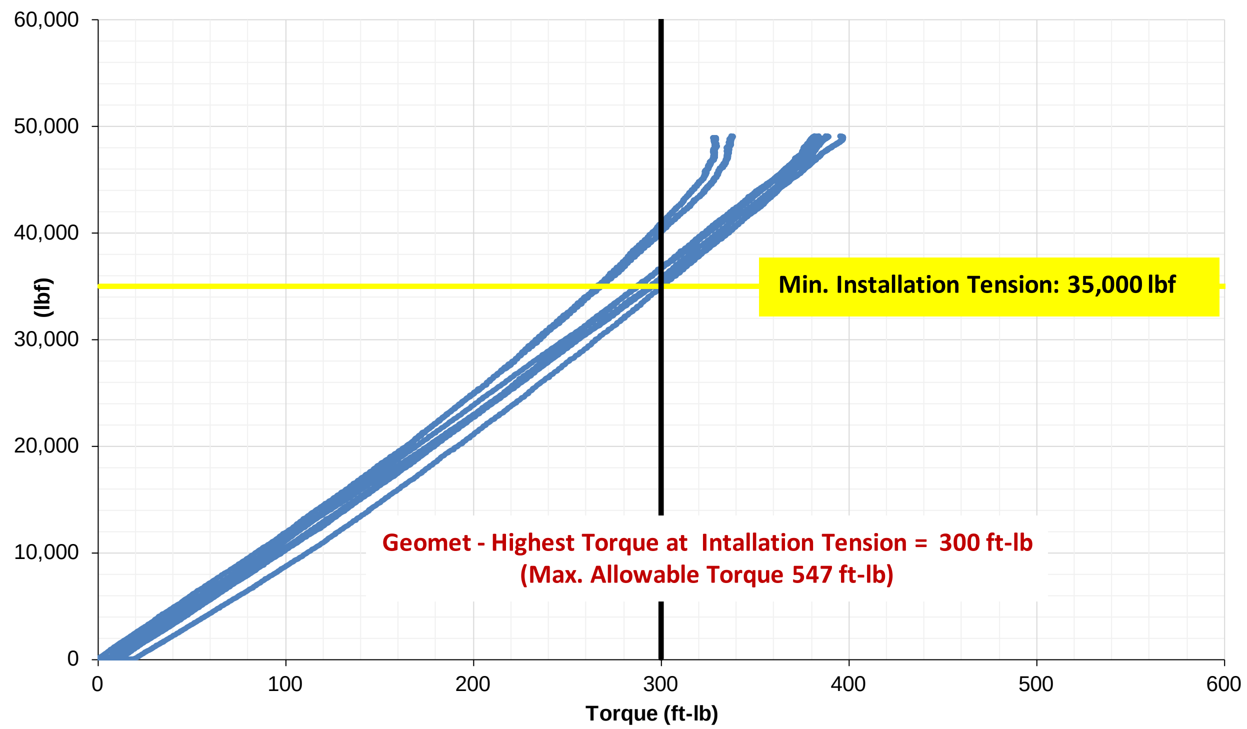


Figure 14 – RC test data for 3/4 inch parts: (i) angle vs. tension, and (ii) torque vs. tension

5.6 Salt spray exposure

IFI-144 requires salt spray testing (SST) in accordance with ASTM B117 for 1000 hours followed by visual evaluation for the percentage of red rust on significant surfaces.

Ten parts of each size (1 inch and 3/4 inch) bolts, nuts and washers were tested. After 1008 continuous hours of exposure, parts were rinsed with warm water to remove salt residue, allowed to dry, evaluated for red rust, and photographed. No red rust was observed on significant surfaces of any samples.

The test was then extended to 5000 hours, as had been done for previous IFI-144 testing of DACROMET® P and Magni 565 coatings. At the end of 5000 hours of exposure, parts were rinsed, allowed to dry, evaluated and photographed (Figs. 15-16). Minimal red rust was observed on less than 1% of the total area of any sample. The Location of red rust was primarily on the corners of the hexagonal heads. Detailed images are included in the Appendix.

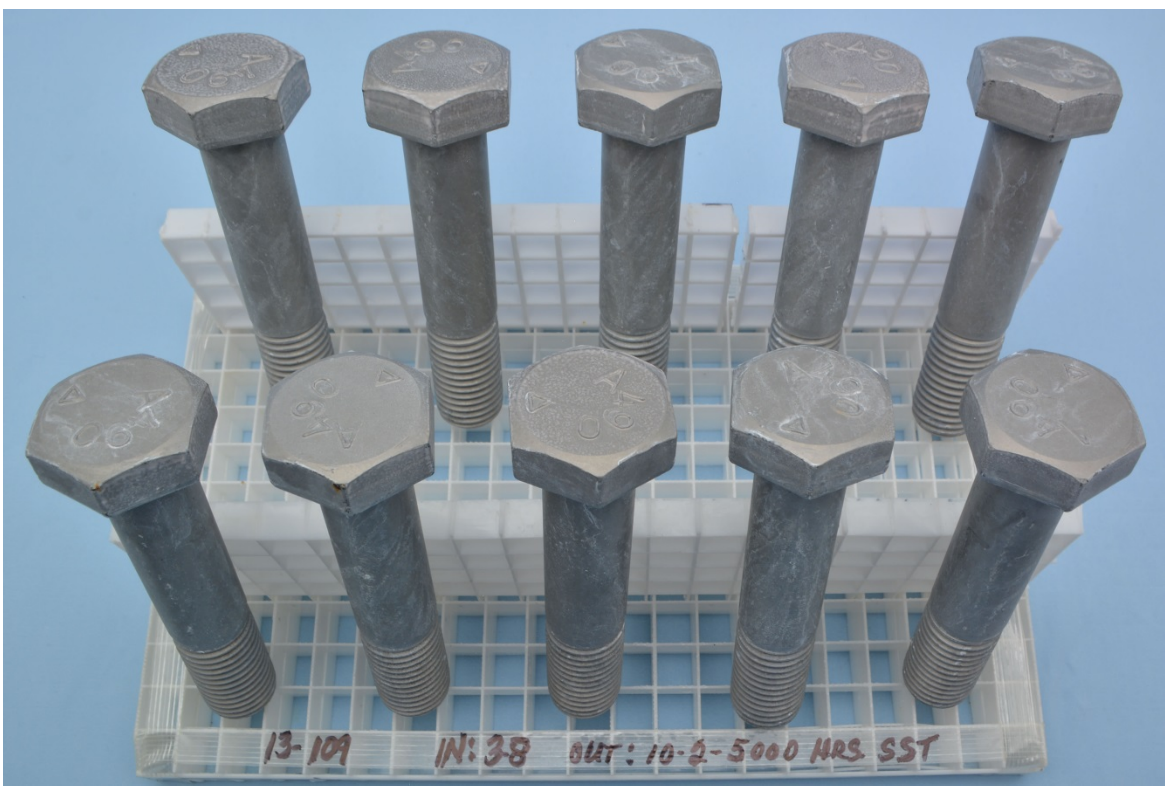


Figure 15 –1 inch bolts after 5000 h of salt spray exposure

Figure 16 –3/4 inch bolts after 5000 h of salt spray exposure

5.7 Cyclic exposure

IFI-144 requires that accelerated cyclic corrosion testing (CCT) be performed for an exposure period of 80 cycles on bolt/nut/washer assemblies mounted into fixtures in the *loaded* and *unloaded* conditions in order to evaluate the potential for stress corrosion cracking (SCC) resulting from galvanic corrosion generated hydrogen. The cyclic corrosion testing was performed done in accordance with GMW 14872[13], which unlike continuous salt spray, may be used to predict corrosion service life for automobiles in given conditions. Although the eventual corrosion service life of a vehicle will depend on specific conditions to which it is exposed, cycles of electrolyte (salt) exposure, humidity, heat, and drying are more representative of service conditions than continuous salt spray exposure. Table 12 and figures 17-18 illustrate the weight loss rate of witness test coupons, indicating that

[13] In 2010, General Motors revised and renumbered GM 9540P to GMW 14872. This latest revision was used for the current study. As with the earlier revision, corrosiveness of the test environment is monitored by simultaneously exposing witness test coupons, made of AISI 1006-1010 bare steel, and measuring mass loss (after removal of corrosion products) periodically during the test. It should be noted that the mass loss targets in GMW 14872 are somewhat more aggressive than in GM 9540P.

conditions prescribed in GMW 14872 are more aggressive than those prescribed in GM 9540P. The distinction made for “loaded” and “unloaded” conditions is intended to isolate the effect of stress, specifically under the turn-of-the-nut method resulting in over-elastic tightening applied to structural bolts, to simulate “worst case” service conditions for SCC.

The test fixtures consisted of hardened AISI 4140 rectangular steel blocks. The fixtures were coated with the same coating as the bolts to avoid any galvanic corrosion effects. For the purpose of this test, 1 inch and 3/4 inch *standard A490 bolts*, nuts and washers were utilized for cyclic testing. The test pieces were exposed for a total of 120 cycles[14] and evaluated for visual corrosion and weight loss.

As indicated earlier, the loaded condition of the bolts/nuts/washers assembled in fixtures was achieved by the turn-of-nut method. More precisely, the assembly procedure was as follows: tighten to snug tension (15% of the minimum installation tension) followed by tightening to 30° beyond the minimum RCSC pre-installation angle. This procedure resulted in 1/2 turn + 30° (i.e., 210° total) beyond snug tension for the 1-8 x 5 inch bolts, and 1/3 turn + 30° (150° total) beyond snug tension for the 3/4-10 x 3 inch bolts. The turn-of-the-nut tightening was performed by Gene Mitchell at GWY Inc., Greenfield, NH using assembly equipment designed for this purpose.

Following exposure to 80, and 120 cycles respectively, the parts were rinsed with warm water and visually examined. None of the bolts showed any signs of cracking or failure. With one exception, there were no indications of red rust on any of the bolts after 80 cycles. After 120 cycles, only very minor red rust was observed on the test parts. See Table 13 and figures 19-20. Detailed images are included in the Appendix.

The corrosion weight loss of exposed parts was measured by weighing the parts prior to and following exposure. The results showed that GEOMET® 321P coated parts did not experience any significant weight loss. Similar to previous findings with DACROMET® and Magni 565 coatings, a slight weight gain was observed which can be attributed to the presence of oxides and residues (Table 14).

[14] The exposure for 120 cycles exceeds the requirement for 80 cycles in IFI-144, but was intended to match previous studies performed on DACROMET and Magni 565.

Table 12 – Cyclic test conditions

Coating	Tightening Condition	Exposure	Sample Size
Test coupon	n/a	120 Cycles	22
GEOMET® 321P	Finger tightened	120 Cycles	5 per size
GEOMET® 321P	Turn-of-nut	120 Cycles	5 per size

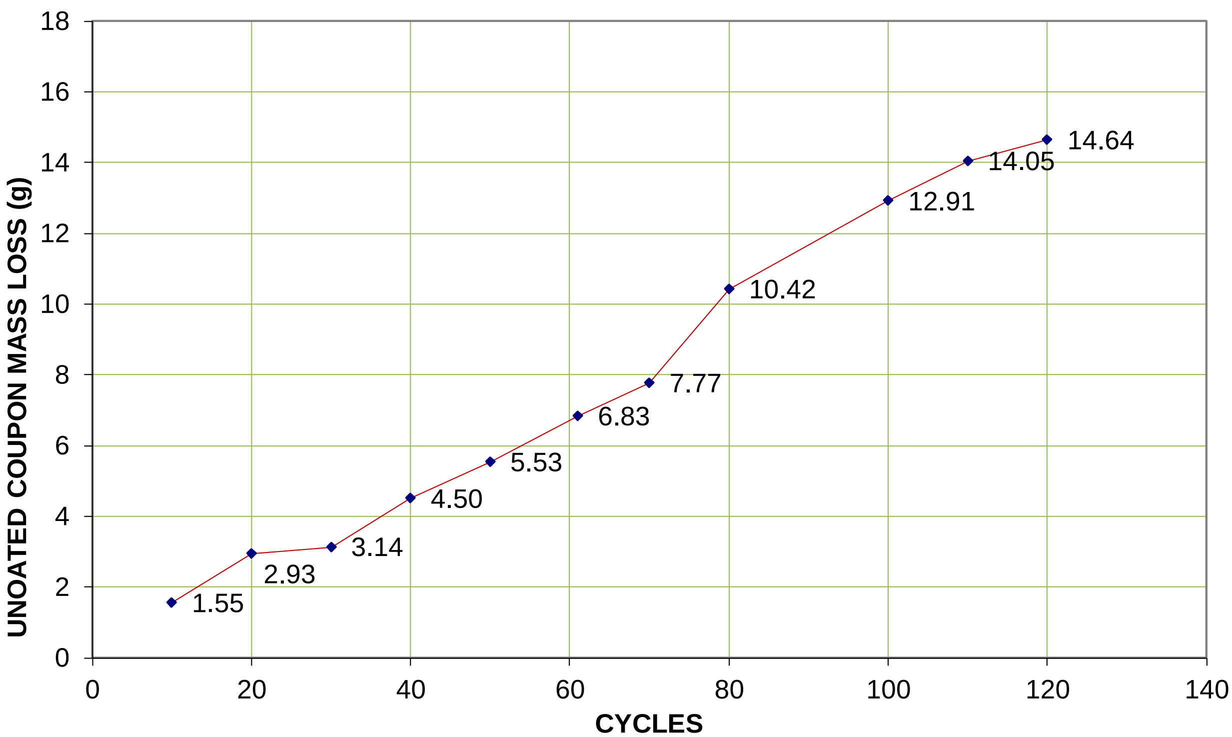


Figure 17 – Corrosion coupons 80 cycle mass loss: 10,423 mg
120 Cycle Mass Loss: 14,636 mg

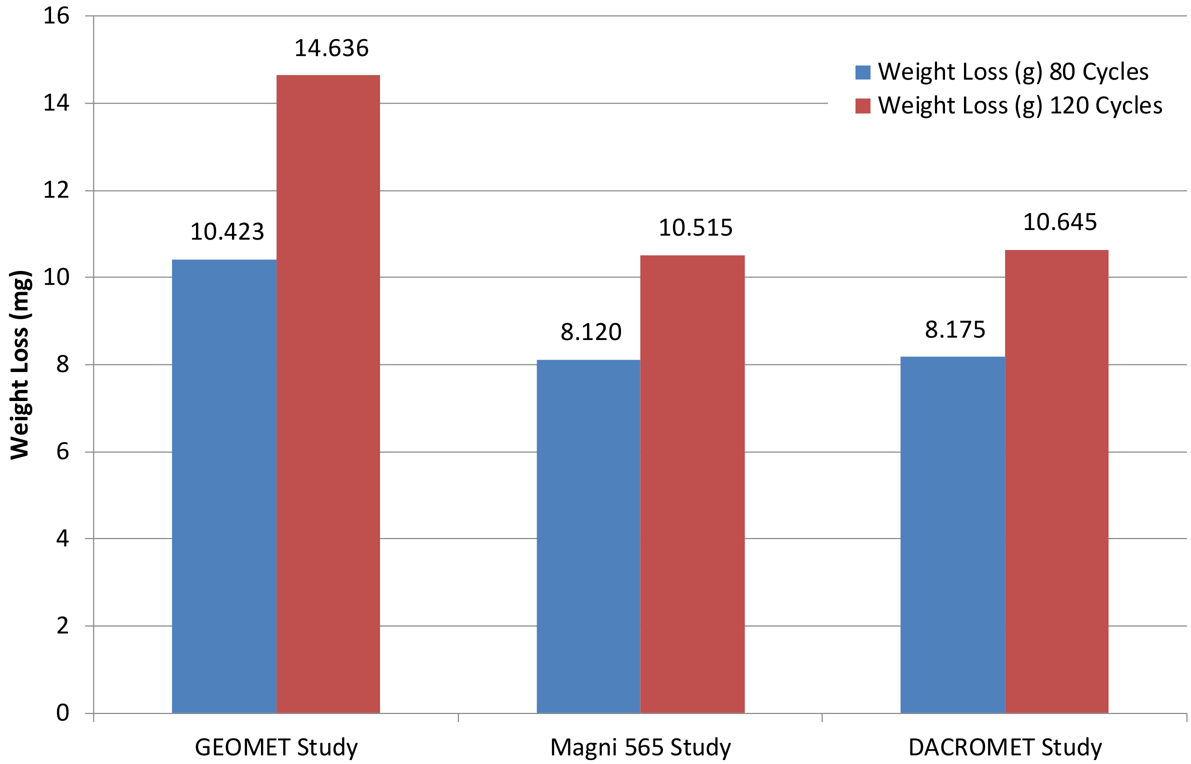


Figure 18 – Comparison illustrating more corrosive GMW14872 conditions in comparison GM 9540P condition used in previous studies

Table 13 – Visual estimation of red rust during cyclic testing

Sample No.	1 inch Bolts			
	No-load Bolts		Preload Bolts	
	80	120	80	120
1	0	<1	0	0
2	0	<1	<1	<1
3	0	1	0	<1
4	0	1	0	1
5	1	2	<1	<1

Sample No.	3/4 inch Bolts			
	No-load Bolts		Preload Bolts	
	80	120	80	120
1	0	<1	0	<1
2	0	<1	0	<1
3	0	<1	0	<1
4	0	<1	0	<1
5	0	<1	0	1

Table 14 – Weight change (g) on test parts following 120 cycles of exposure

			1 inch Assemblies (no load)		
Bolt	**Nut**	**W1**	**W2**	**Fixture**	**Assembly**
0.05	0.02	0.01	0.01	0.16	0.25

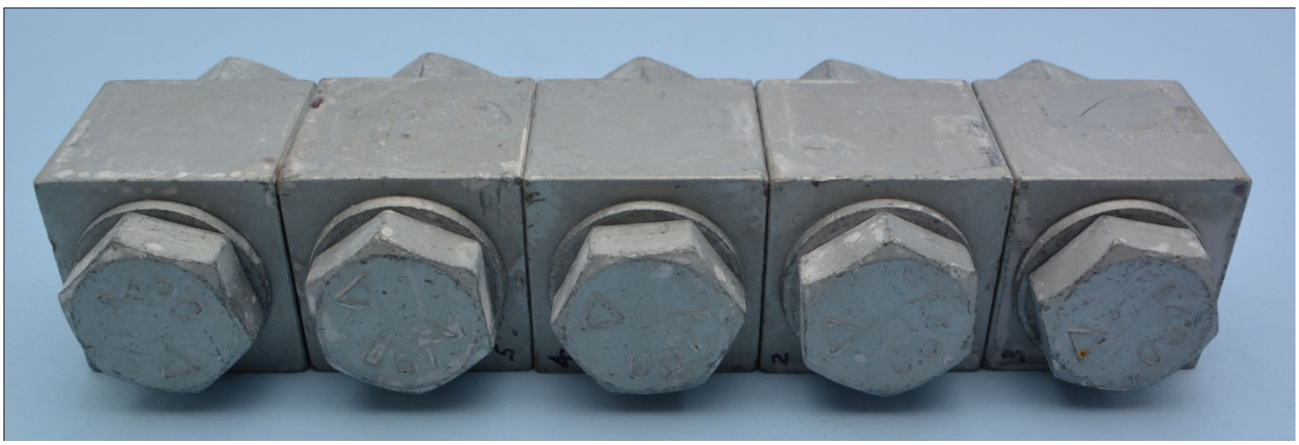
			3/4 inch Assemblies (no load)		
Bolt	**Nut**	**W1**	**W2**	**Fixture**	**Assembly**
0.04	0.05	0.01	0.01	0.06	0.17

(No-load bolts)

(Preloaded bolts)

Figure 19 –1 inch bolts after 120 cycles of exposure

(No-load bolts)



(Preloaded bolts)

Figure 20 –3/4 inch bolts after 120 cycles of exposure

5.8 Post exposure tensile tests

IFI-144 requires that bolts be axially tested after cyclic exposure to ensure that no degradation in strength has occurred as a result of the corrosion of the coated bolts. In this study, the 1-8 x 5 and 3/4-10 x 3 *standard A490* bolts were wedge tensile tested after 120 cycles of cyclic exposure in the preloaded (turn-of-the-nut) condition. Although IFI-144 specifies only axial testing, in this study, wedge tensile testing was performed in accordance with ASTM F606. The results were compared to the pre-exposure wedge tensile test results previously given in Section 2.1. Wedge tensile test results for all of the conditions are given in Tables 15 and 16. The results demonstrate that GEOMET® 321P coated bolts did not exhibit any measurable loss of strength following 120 cycles of cyclic exposure.

Table 15 – Pre and post exposure wedge tensile test results for 1-8 specimen bolts

	Uncoated	**120 Cycles**
Sample	**Load (lbf)**	**Load (lbf)**
1	96,306	100,200
2	99,335	99,500
3		100,000
4		102,100
5		100,400
Avg.	**99,321 (163,976 psi)**	**100,440 (165,825 psi)**

Tensile Stress Area 0.6057 in^2

Table 16 – Pre and post exposure tensile test results for 3/4-10 specimen bolts

	Uncoated	**120C**
Sample	**Load (lbf)**	**Load (lbf)**
1	54,776	54,300
2	54,765	54,600
3		54,200
4		54,600
5		54,300
Avg.	**54,771 (163,984 psi)**	**54,400 (162,874 psi)**

Tensile Stress Area 0.3340 in^2

5.9 Hydrogen embrittlement – process qualification

The incremental step load test method described in ASTM F1940 was used as the basis for quantifying the risk of internal hydrogen embrittlement (IHE) posed by the GEOMET® 321 coating process. In this method, a coated notch bar is subjected to a sustained four-point bending load and slow strain rate under displacement control. The test indirectly quantifies the amount of residual hydrogen introduced during the coating process by measuring the loss of fracture strength of the coated notch bar using a standardized loading protocol in air. The notch fracture strength (NFS) is defined as the maximum load at the onset of cracking that is identified by a 5 % drop in load under displacement control. Bare (uncoated) notch square bars are tested in the same manner to establish the *baseline* notch fracture strength. The ratio of the fracture strength of a coated notch bar over the baseline represents the percent notch fracture strength (%NFS), which is a measure of the degree of internal hydrogen embrittlement of the notch bar resulting from the coating process.

$$NFS\% = \frac{NFS_{(W)F1940}}{NFS_{(B)F1940}} \times 100 \qquad \text{Eq. 1}$$

Where:

NFS%	= Percent notch fracture strength
NFS(W)F1940	= Notch fracture load of coated (witness) notch bar
NFS(B)F1940	= Notch fracture load of bare notch bar

It should be noted that in this study NFS% values were obtained using a modified loading protocol (2% - 2 hour) that is more than 3 times slower than that the standard ASTM F1940 loading protocol. This very slow loading protocol was selected based on ASTM F1624 methodology to measure the true threshold given the anticipated decrease in hardness and fracture strength of the notch bars resulting from the curing temperatures of the GEOMET® process [5].

ASTM F1940 notch bars were coated by both GEOMET® 321 processing methods utilized for this study: the dip spin process at Michigan Metal Coatings and the rack spray process at Allegheny Coatings. As anticipated, the results showed marginal reduction of NFS% for both processes with average values of 91.2% and 92.6% respectively demonstrating that the two processes are equivalent. The overall average NFS% from both processes grouped together is 91.7% (Fig. 21).

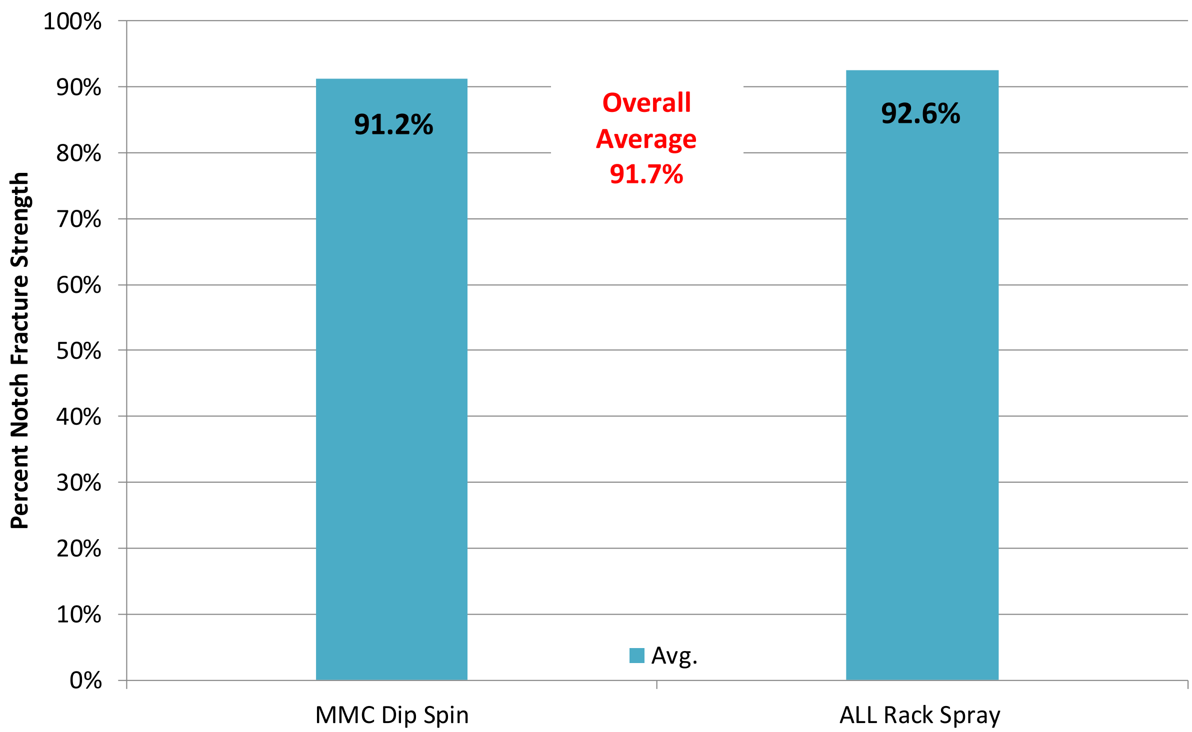


Figure 21 – Average NFS% values for the Michigan Metal Coating dip spin and Allegheny rack spray processes without any correction for loss notch bar fracture strength resulting from curing temperature

It is understood from previous work [4] that the marginal reduction in percent fracture load to 91.7% of baseline ISL fracture load of uncoated specimens is not solely related to hydrogen embrittlement phenomena. Rather, it is principally explained by a parallel reduction of specimen hardness resulting from the GEOMET® 321 curing cycle[15], which exceeds the specimen tempering temperature of 220 °C. The specimen hardness measured after GEOMET® 321 processing was 49.6 HRC, compared to 52.2 HRC for pristine notch bars, a reduction of 2.6 HRC points (Fig. 22).

The curing of the coated specimens effectively modifies the standard specimen material to a slightly lower hardness, *non-standard* material. Consequently, the fast fracture strength of GEOMET® coated specimen is the more appropriate baseline for

[15] Curing cycle consists of a minimum of 15 minutes at 316-330 °C per coat, for a minimum total of 30 minutes for the spray coat process and 45 minutes minimum for the dip spin process.

comparison with ISL results (i.e., denominator in Eq. 1). This alternate baseline eliminates hardness as a variable. Therefore to confirm that the drop in fracture strength is principally related to a lowering of specimen hardness rather than hydrogen embrittlement phenomena, GEOMET® 321 coated notch bars were tested by two methods: (i) fast fracture (FF), and (ii) incremental step loading (ISL). As indicated previously, the ISL loading protocol comprised 2% - 2 hour steps, and the loading rate for FF testing was 100 lb/min. With this approach, the average GEOMET® 321 coated ISL fracture strength was 93.4% of the baseline strength (Fig. 23). The remaining (relatively minor) ~6% difference is attributed to the effect of residual internal hydrogen from steelmaking processes. This effect of residual internal hydrogen in the steel typically causes a drop of ~10-12 % of fracture strength in standard F1940 notch bars. [4-5][16]

These results confirm previous findings that the GEOMET® 321 coating process, similar to the DACROMET® and Magni 565 processes does not introduce any residual hydrogen, and therefore does not pose any risk of internal hydrogen embrittlement.

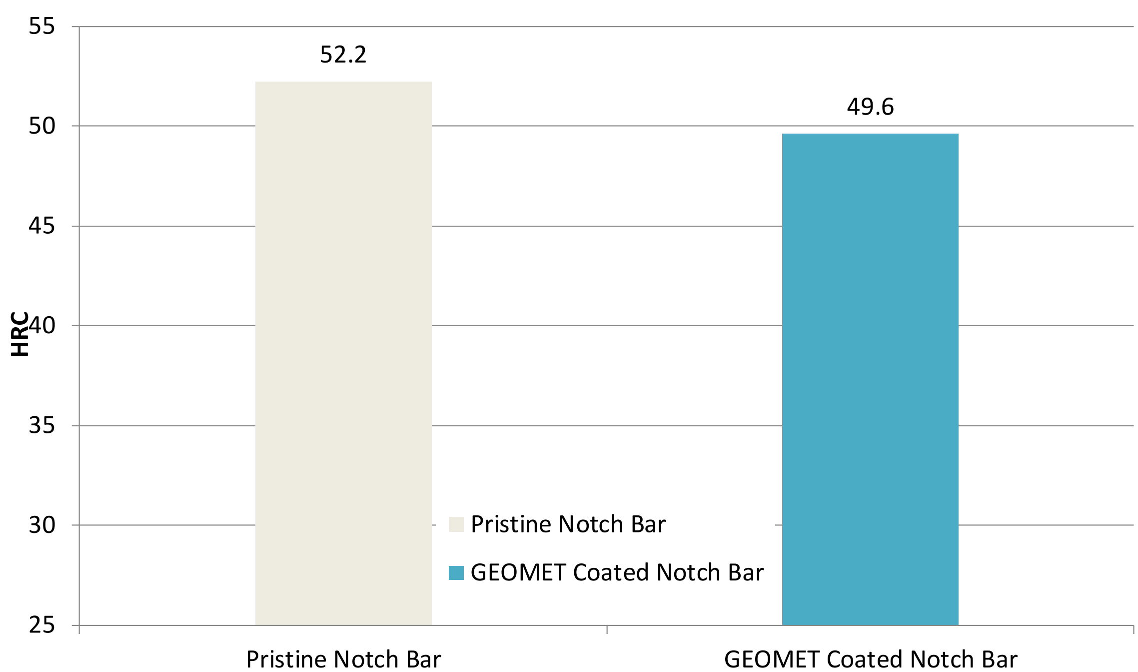


Figure 22 –Hardness comparison of ASTM F1940 notch bars before and after GEOMET® 321 coating, illustrating the effect of curing temperature on hardness

[16] The reported 10-12% reduction of fracture strengh occurs when comparing ISL strength to FF strength.

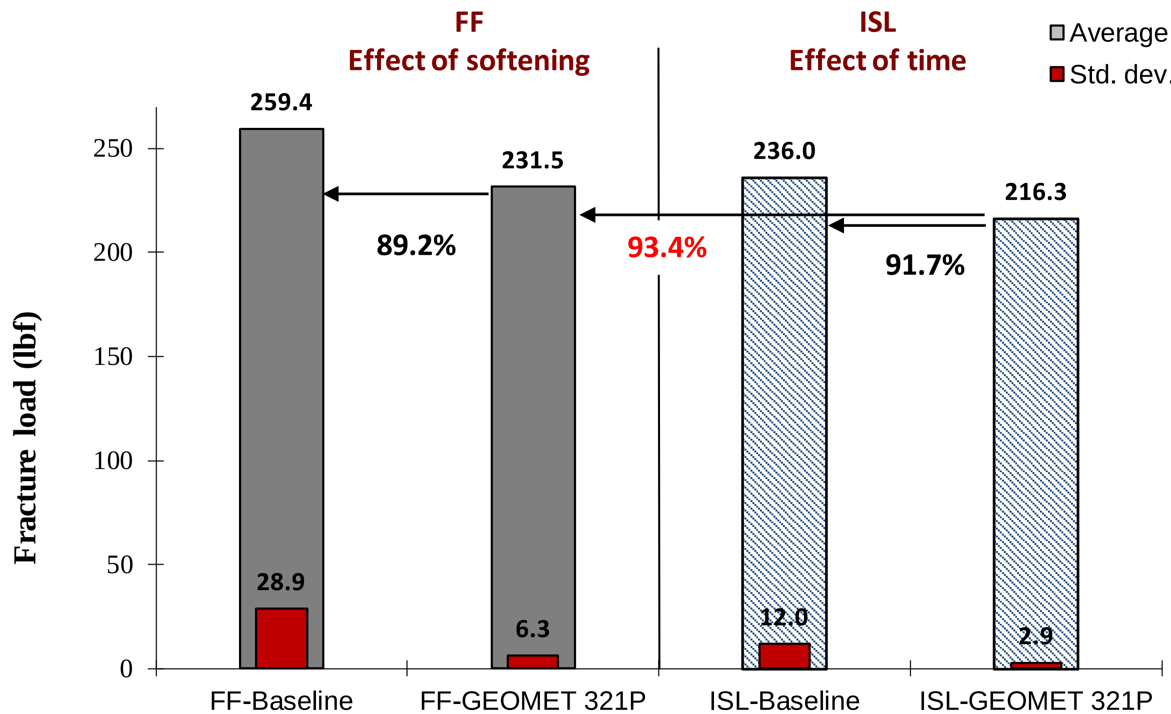


Figure 23 – IHE analysis considering material modification by using the fast fracture strength of a GEOMET® 321 coated specimen as the baseline

5.10 Hydrogen embrittlement – product qualification

The primary concern that is intended to be addressed in qualifying coatings for use on A490 bolts is the risk of environmental hydrogen embrittlement (EHE) that is accelerated by cathodically generated hydrogen during the corrosion process of a sacrificial coating. This risk is fundamentally a function of the susceptibility of the A490 bolt material to EHE. Susceptibility increases with increasing strength. By using *specimen* bolts heat treated to strengths above the specified strength for A490 bolts, the most severe material susceptibility condition was tested. The risk of EHE also increases with increasing quantities of absorbed hydrogen. The more active (more sacrificial) a coating, the more hydrogen is generated as it corrodes preferentially whilst it protects the steel substrate. The most sacrificial metallic coating is zinc, with an open circuit potential (OCP) of -1.20 V. The OCP for steel is roughly -0.65 V. In this study, the OCP for GEOMET® 321 on a coated specimen bolt was measured and then compared to previous result obtained for DACROMET® and Magni 565. [6] As can be seen from figures 24-25, OCP values for GEOMET® 321, after 24h of stabilization corresponding to the consumption of the topcoat, was in the order of -1.06 V, which is marginally lower than for the previously studied coatings. Nevertheless, this difference is not considered significant. GEOMET® 321 on steel corrodes at a slower rate than zinc on steel, consequently it generates significantly lower amounts of hydrogen than a pure zinc coating. This characteristic of having an OCP value that is closer to that of steel is beneficial, because it reduces the risk of EHE.

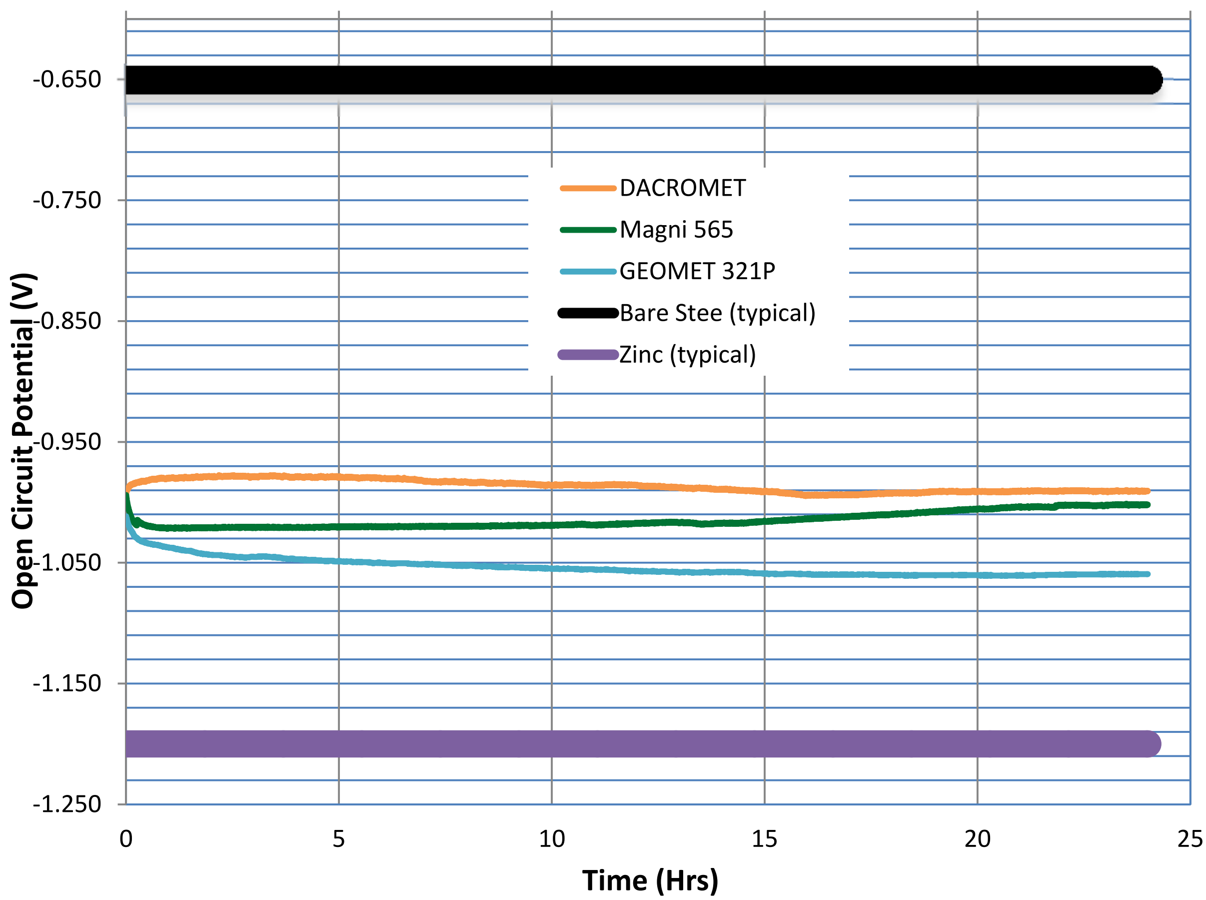


Figure 24 – OCP test progression for GEOMET® 321 compared to previously published results for Magni 565 and DACROMET® over 24 h period

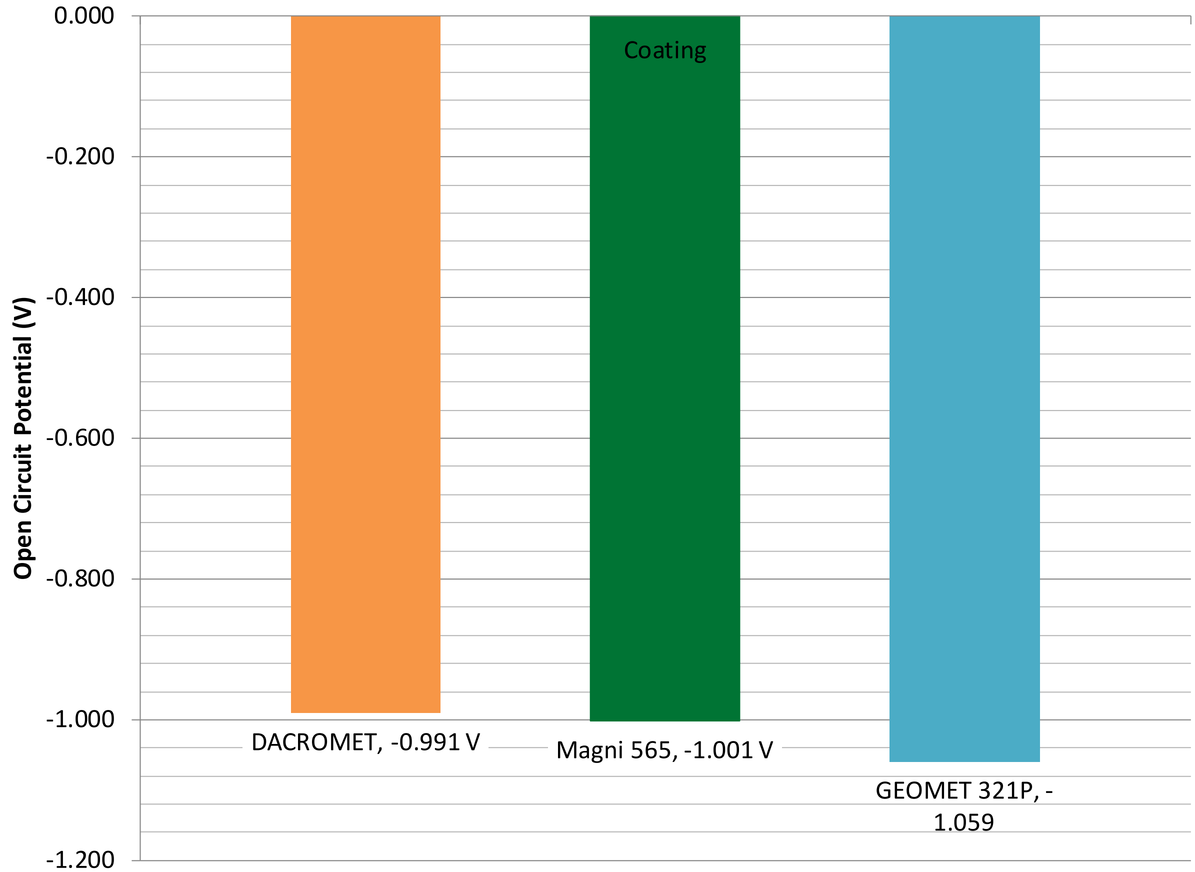


Figure 25 – OPC values for GEOMET® 321 compared to previously published results for Magni 565 and DACROMET® after 24 h of stabilization

Having established the corrosion potential for GEOMET® 32P, the methodology described in ASTM F2660 "*Qualifying Coatings for Use on A490 Structural Bolts Relative to Hydrogen Embrittlement.*" This test method was developed in order to define a standard methodology for EHE testing required by IFI-144. The environmental hydrogen embrittlement test was conducted on 1-8 and 3/4-10 specimen bolts. The specimen bolts were truncated by removal of the bolt head. The thread length was then inserted into the thread gripping fixtures to achieve the four-point bending, with a minimum of two threads between the gripping devices. The loading method was four-point pure bending under displacement control. The moment arm length was more than doubled to achieve the loads required to testing the 1 inch bolts.

The first step in the testing sequence was a measurement of the *fast fracture* load of the specimen bolts in bending ($FFS(B)_{coated}$) by performing a test. The average of these results was used to determine the fast fracture strength of each condition.

To measure the EHE susceptibility of the fastener/coating system, bolts were tested in environmental conditions using the incremental step load (ISL) methodology described in ASTM F2660, to measure the hydrogen embrittlement threshold load P_{th}. The focal condition for determining the EHE susceptibility of the fastener/coating system is the simulation of a galvanic corrosion condition. A galvanic condition was created by inscribing a mark in the coating at the root of a bolt thread to expose the steel substrate. This condition simulates a damaged coating, also referred to as "*coating holiday*." The scribe mark was located in the exposed threads between the gripping fixtures and had a length of one diameter. For the environmental tests, the specimens were immersed in an environmental chamber filled with 3.5 % NaCl solution. The bolts and fixtures were each isolated from other metal contacts using a lacquer coating. A reference electrode was placed in close vicinity to the scribe mark.

The tests proceeded until the sample experienced a load drop of more than 5% during any single step in the load rate. Subsequent tests were performed at progressively decreasing loading rates by using the same methodology. The lowest threshold value established by consecutive tests was considered the threshold load for the condition tested. The minimum acceptance value of the threshold load for the galvanic condition was determined in accordance with ASTM F2660 which stipulates that the $P_{th} > 0.6\ FFS(B)_{coated}$.

The results obtained in this study for both specimen bolt sizes are shown in Figures 26-27. *The results indicate that the acceptance load level was comfortably exceeded***.** The acceptance level values which apply to the "coating holiday" or "scribed" condition (red bar) are illustrated in green on each chart. Given that ASTM A490 bolts can be manufactured up to nominal size of 1-1/2 inch, the equivalence values in ASTM F2660, Table 2 are used. For 1 inch specimen bolts the threshold value must equal or exceed 71.8 % of the baseline $FFS(B)_{coated}$ in order to be applicable to 1-1/2 inch bolts. From figure 26, a threshold value of 76% was obtained for the scribed condition. Consequently, the result satisfies the acceptance criterion up to 1-1/2 inch. These test results demonstrate the GEOMET® 321 coating satisfies the acceptance criteria for EHE on A490 bolts of all sizes.

Additional to the immediate objective of qualifying GEOMET® 321 for use on ASTM A490 bolts, the ISL tests performed in air exhibited threshold strengths at between 91 % and 96 % of baseline strength. Similar to the F1940 notch square bars, this loss of strength can be attributed to the residual hydrogen that already existed in the steel.

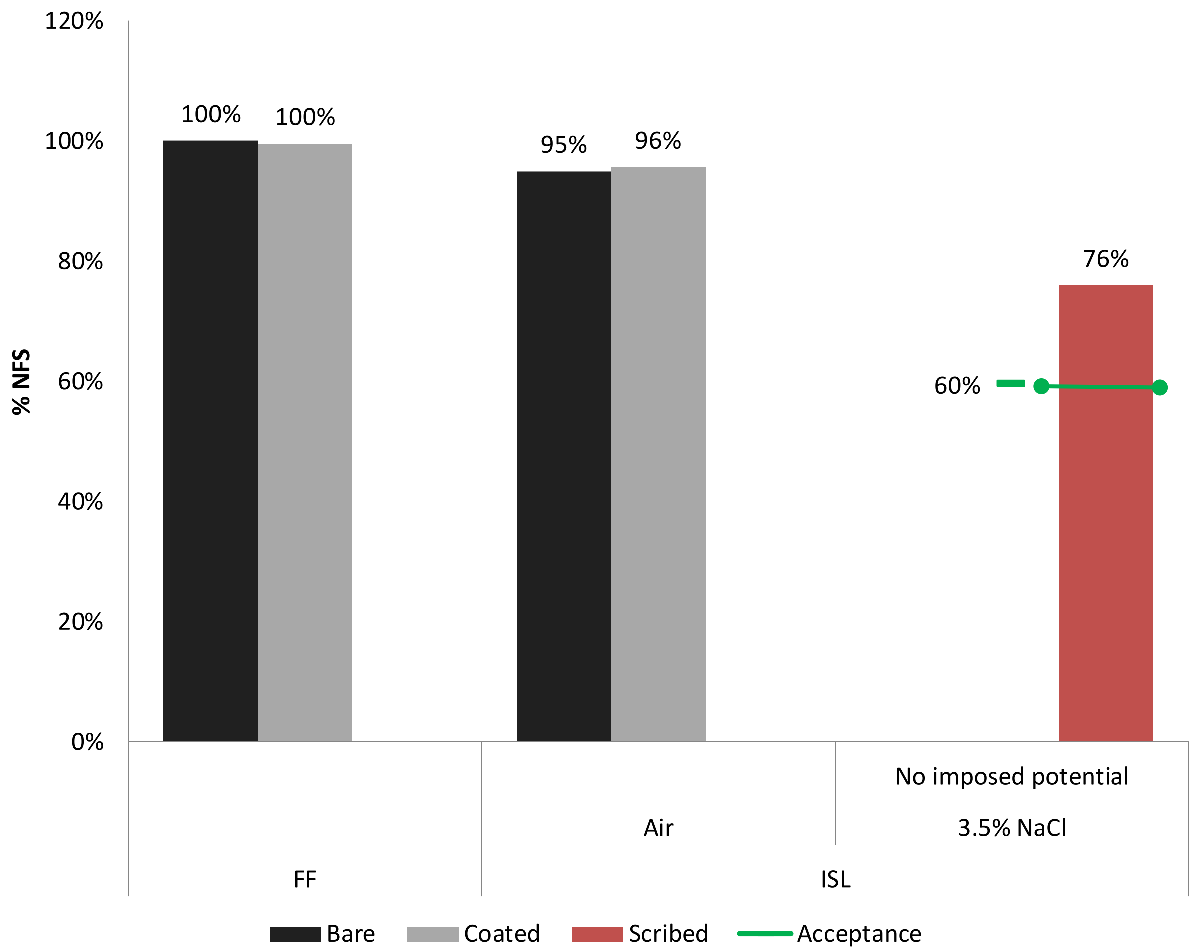


Figure 26 – 1-8 average threshold NFS% values

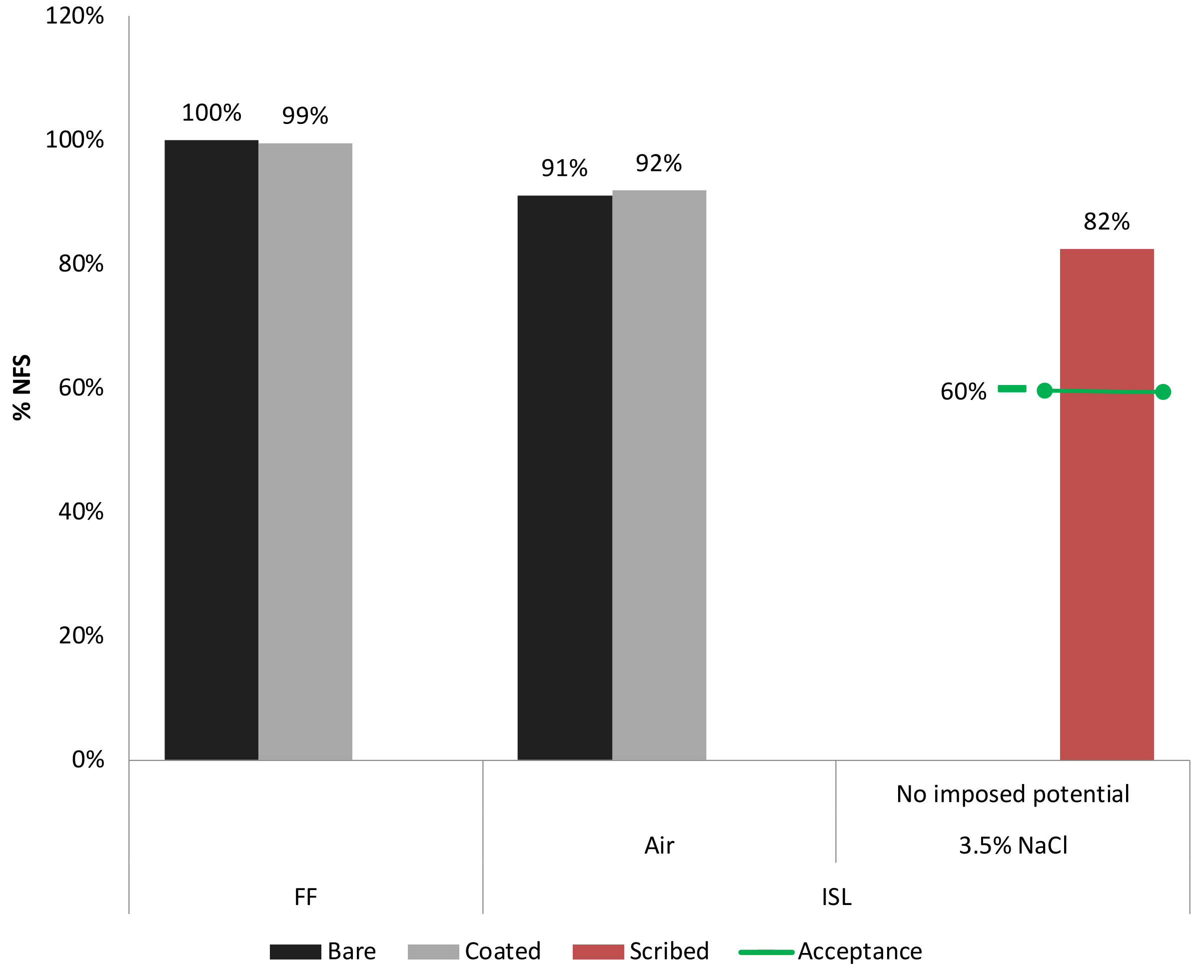


Figure 27 – 3/4-10 average threshold NFS% values

6. SYNOPSIS AND CONCLUSIONS

The GEOMET® 321 coating system satisfied the performance criteria tests specified in IFI-144, notably: coating thickness, coating adhesion, paintability, rotational capacity, salt spray exposure, and cyclic exposure tests. Furthermore cyclic exposure tests of standard A490 bolts tightened by the turn on the nut method constituted a qualitative test that demonstrated that GEOMET® 321 did not cause delayed hydrogen induced failure.

The most significant test results obtained in this study relate to the risk of internal hydrogen embrittlement (IHE) and environmental hydrogen embrittlement (EHE). Process qualification results performed using ASTM F1940 methodology demonstrated that there is no risk of IHE. Product environmental testing of high strength specimen bolts, performed in accordance with ASTM F2660, exceeded the acceptance criteria thresholds. These tests performed on high hardness specimen bolts demonstrated that GEOMET® 321 would not promote environmental hydrogen embrittlement (EHE) on standard ASTM A490 high strength structural bolts, regardless of size.

The findings of this study support previous findings regarding GEOMET®, and although no action is required by Committee F16 with respect to the current wording in ASTM A490 and A490M, it is recommended that the next revision of ASTM A490 and A490M, specifically in the form of a combined structural bolt standard, explicitly list both chrome and non-chrome versions of F1136 coatings as being permissible. This report is presented for review by Committee F16.

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4. S. Brahimi, S. Yue, *Effect of Surface Processing Variables and Coating Characteristics on Hydrogen Embrittlement of Steel Fasteners*, SURFIN 2009, Louisville, KY.

5. Brahimi S., *Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners - an Experimental Based Evaluation of the Test Method*, AESF SUR/FIN ’99, Cincinnati, OH, June 1999.

6. *Qualification of ASTM F2833 Coatings for use on ASTM A490 High-Strength Structural Bolts.* IBECA Research Report 08-12, www.astm.org, Commiteee F16 Documents, January, 2012.

REFERENCED SPECIFICATIONS

IFI

1. IFI-144 *Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts*.

ASTM

1. A325 *Standard Specification for Structural Bolts, Steel, Heat Treated, 120/105 ksi Minimum Tensile Strength*.

2. ASTM A490 *Standard Specification for Structural Bolts, Alloy Steel, Heat Treated, 150 ksi Minimum Tensile Strength*.

3. ASTM A490M *Standard Specification for High-Strength Steel Bolts, Classes 10.9 and 10.9.3, for Structural Steel Joints (Metric)*.

4. ASTM A751 *Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products*.

5. ASTM B117 *Standard Practice for Operating Salt Spray (Fog) Apparatus*.

6. ASTM B571 *Standard Practice for Qualitative Adhesion Testing of Metallic Coatings*.

7. ASTM B659 *Standard Guide for Measuring Thickness of Metallic and Inorganic Coatings*.

8. ASTM D1186 *Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base*.

9. ASTM D1654 *Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments*.

10. ASTM D3359 *Standard Test Methods for Measuring Adhesion by Tape Test*.

11. ASTM E3 *Practice of Preparation of Metallographic Specimens*.

12. ASTM E92 *Standard Test Method for Vickers Hardness of Metallic Materials*.

13. ASTM F606 *Test Methods for Determining the Mechanical Properties of Externally and Internally Threaded Fasteners, Washers, and Rivets*.

14. F1136 *Standard Specification for Zinc/Aluminum Corrosion Protective Coatings for Fasteners.*

15. F1136M *Standard Specification for Zinc/Aluminum Corrosion Protective Coatings for Fasteners (Metric)*.

16. ASTM *F1624 Standard Test Method for Measurement of Hydrogen Embrittlement in Steel by the incremental Loading Technique*.

17. ASTM F1940 *Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners*.

18. ASTM F2660 Q*ualifying Coatings for Use on A490 Structural Bolts Relative to Hydrogen Embrittlement*.

19. ASTM F2833 *Standard Specification for Corrosion Protective Fastener Coatings with Zinc Rich Base Coat and Aluminum Organic/Inorganic Type*

GM

1. GM 9540P *Accelerated Corrosion Test*.

2. GMW14872 *Cyclic Corrosion Laboratory Test.*

OTHERS

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2. FHWA, Procedure for performing rotational capacity test, long bolts in tension calibrator in Appendix A1 - A490. 2005, Federal Highway Administration.

3. RCSC, Specification for Structural Joints Using High-Strength Bolts, December 31, 2009.

APPENDICES