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Research Report
TIR 08-12

QUALIFICATION OF ASTM F2833 COATINGS 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 was to qualify ASTM F2833 coatings 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 F2833, "***Standard Specification for Corrosion Protective Fastener Coatings with Zinc Rich Base Coat and Aluminum Organic/Inorganic Type***," was recently adopted by Committee F16 on Fasteners, as a standard specification that is applicable to Magni 565 zinc-aluminum dispersion coating licensed by the Magni Group headquartered in Birmingham, MI. Following decades during which coatings were not permitted, the current edition of the A490 specification only permits the application of metallic coatings that comply with ASTM F1136. IFI-144 "***Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts***" prescribes a broad methodology to qualify coatings for use with ASTM A490 bolts. Consideration of any coating for use on A490 bolts must be based on results of testing performed in accordance with IFI 144, and submitted to Committee F16 for review.

The Magni 565 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 results performed in accordance with ASTM F1940 demonstrated that the risk of IHE is nil. Product environmental testing of super high strength specimen bolts, performed in accordance with ASTM F1624, exceeded the acceptance criteria thresholds established in ATSM WK13659. These results demonstrated that Magni 565 will not promote environmental hydrogen embrittlement (EHE) on standard ASTM A490 high strength structural bolts, regardless of size or steel alloy chemistry.

Based on the findings of this study, it is strongly recommended that ASTM Committee F16 on Fasteners approve the use of ASTM F2833 (Magni 565) on A490 high strength structural bolts.

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1. OBJECTIVE

The objective of this investigation was to qualify ASTM F2833 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 F2833, "***Standard Specification for Corrosion Protective Fastener Coatings with Zinc Rich Base Coat and Aluminum Organic/Inorganic Type***," was recently adopted by Committee F16 on Fasteners, as a standard specification that is applicable to the Magni 565 zinc-aluminum dispersion coating licensed by the Magni Group headquartered in Birmingham, MI.

Following decades during which coatings were not permitted, the current edition of the A490 specification only permits the application of metallic coatings that comply with ASTM F1136, "***Standard Specification for Zinc/Aluminum Corrosion Protective Coatings for Fasteners.***" Although the two specifications, ASTM F1136 and ASTM F2833, appear to cover similar Zn/Al dispersion coatings, they are in fact adapted specifically to two competing proprietary coating systems. Consequently, the two are treated as different coatings for the purpose of qualification on A490 bolts. The prohibition against metallic coatings was instituted as a precaution against the risk of environmental hydrogen embrittlement failures. IFI-144 "***Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts***" prescribes a broad methodology to qualify coatings for use with ASTM A490 bolts. In Section 4.3.2 of ASTM A490 it is stated: "Future consideration of any coating will be based on results of testing performed in accordance with the procedures in IFI 144, and submitted to Committee F16 for review." Consequently, this study applied the methodology prescribed in IFI-144.

Finally, it must be emphasized that 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). EHE can be accelerated by cathodically generated hydrogen during the corrosion reaction of a sacrificial coating. More precisely, if the substrate steel becomes exposed to the elements, the coating corrodes preferentially while preventing the substrate

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[1] Any reference to ASTM 490 in this document implies the inclusion of ASTM A490M.

steel from corroding. IFI-144 prescribes several methods to test the risk of hydrogen embrittlement. Other tests required by IFI-144 are essentially methods for benchmarking the performance of a coating. They do not constitute acceptance criteria per se.

2. COATED SAMPLES

For the purpose of this study bolts, nuts and washers in two nominal inch sizes: 1/2-13 UNC, and 3/4-10 UNC. The test pieces were coated by the Magni 565 process, in accordance with ASTM F2833 Grade 1. 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

Testing was carried out using specimen bolts especially heat treated to achieve strength and hardness values that are at or 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 exceeded the maximum limits for standard A490 bolts. Examples of the specimen bolts are shown in Figure 1.

Figure 1 – Coated specimen bolts 3/4-10 x 3 and 1/2-13 x 3 (left), and coated and uncoated 3/4-10 x 3 specimen bolts (right)

Specimen bolt used in this study comprised two nominal inch sizes: 1/2-13 x 3, and 3/4-10 x 3 and were from two 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 8640 and AISI 5135 for the 1/2 inch and 3/4 inch bolts respectively.

Table 1 – Specimen bolts chemical composition

	Wt % Conc.	
	1/2-13 x 3	**3/4-10 x 3**
Carbon	0.40	0.36
Manganese	0.82	0.71
Phosphorus	0.005	0.020
Sulfur	0.003	0.015
Silicon	0.24	0.23
Aluminum	0.028	0.021
Nickel	0.48	-
Chromium	0.50	0.85
Molybdenum	0.20	-
AISI Designation	**8640**	**5135**

Mechanical properties for the specimen bolts, coated and uncoated, are given in Tables 2 to 7. The average measured mid-radius hardness values for the 1/2 inch and 3/4 inch bolts were 40.4 and 40.9 HRC respectively. The specified hardness range for A490 bolts is 33-38. Note that at the time of manufacturing the specimen bolts, the maximum hardness for A490 bolts was 39 HRC[2]. In either case, the hardness of the specimen bolts significantly exceeds the hardness of standard A490 bolts.

Average wedge tensile strength values for the specimen bolts were roughly 192 and 182 ksi respectively. The maximum allowable wedge tensile strength for standard A490 bolts is 173 ksi.

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[2] ASTM A490 and A490M hardness requirements were modified early in 2010 to reduce maximum hardness from 39 to 38 HRC, which is more in line with the maximum tensile strength of 173 ksi.

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.

Finally, wedge tensile strength values for the specimen bolts were not statistically altered after being coated (Tables 4 and 7). These results confirmed that the Magni 565 coating process did not alter the mechanical properties of the specimen bolts.

Table 2 – Specimen bolts hardness values - 1/2-13 x 3

	1/2-13 x 3	
Sample	**Mid-radius (HRC)**	**Surface (HR 30N)**
1	40.4	58.8
2	39.9	60.5
3	40.5	58.9
4	40.6	57.8
5	40.7	58.6
Avg.	**40.4**	**58.9**

Table 3 – Specimen bolts hardness values - 3/4-10 x 3

	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 4 – Full sized wedge tensile test results
1/2-13 x 3 uncoated bolts

Sample	Load (lbf)	Stress (psi)
1	27,530	194,010
2	26,960	189,993
3	27,245	192,001
4	27,300	192,389
5	26,950	189,922
Avg.	**27,197**	**191,663**

Table 5 – Full sized wedge tensile test results
1/2-13 x 3 coated bolts

Sample	Load (lbf)	Stress (psi)
1	27,100	190,980
2	27,010	190,345
3	27,650	194,856
4	26,970	190,063
5	27,320	192,530
Avg.	**27,210**	**191,755**

Tensile Stress Area 0.1419 in^2

Table 6 – Full sized wedge tensile test results
3/4-10 x 3 uncoated bolts

	3/4-10 x 3	
Sample	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**

Table 7 – Full sized wedge tensile test results
3/4-10 x 3 coated bolts

	3/4-10 x 3	
Sample	**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

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, standardized 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 of the notch bar specimens are shown in Figure 3. The chemical composition of the lot if notch bars used in this study is given in Table 8. The average measured hardness of the lot of notch bars used in this study was 51.4 HRC.

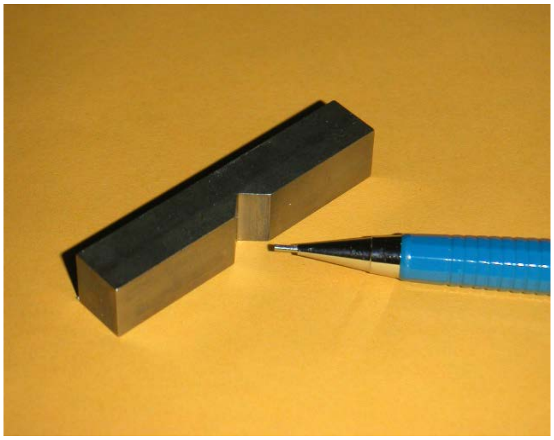

Figure 2 – ASTM F1940 notch bar specimen

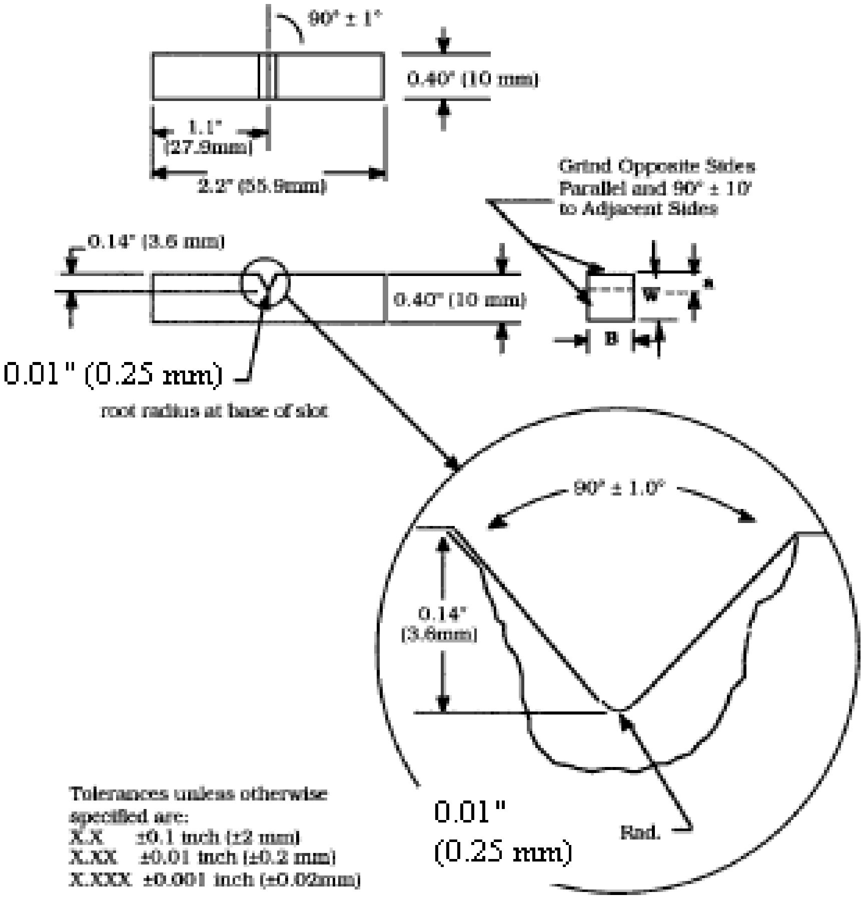

Figure 2 – ASTM F1940 notch bar specimen configuration

Table 8 – Chemical composition of ASTM F1940 notch bar specimens

	Actual	AISI 4340 requirements
Carbon	0.40	0.38-0.43
Manganese	0.78	0.65-0.85
Phosphorus	0.007	0.025 max.
Sulphur	0.030	0.025 max.
Silicon	0.22	0.15-0.30
Copper	0.18	-

Nickel	1.72	1.65-2.00
Chromium	0.82	0.70-0.90
Molybdenum	0.28	0.20-0.30
Aluminium	0.016	-
Vanadium	0.001	-

3. MAGNI 565 COATING PROCESS

Magni 565 is a proprietary coating system licensed by The Magni Group based in Birmingham, MI. This coating system, which originally targeted the automotive market, applies an aluminum/zinc organic topcoat (epoxy) over a zinc rich primer base coat. The basecoat is designed to provide sacrificial protection of the steel substrate. The topcoat is designed to provide an adherent, durable barrier that also has the effect of passivating the zinc basecoat. The coating coverage by this process is very consistent and uniform. Typical coating thickness can range from 12 to 20 microns.

The coating of specimen bolts and ASTM F1940 notch bar specimens used in this study was performed at Depor Industries, in Troy, MI. The coating process was performed under normal operating conditions in accordance with the procedures prescribed by The Magni Group for Magni 565, which is in turn in accordance with ASTM F2833, Grade 1.

Surface preparation consisted of alkaline degreasing followed by acid pickling in sulphuric acid at 42 °C for 10-15 minutes. This step was followed by the application of a medium weight zinc phosphate (Z24) to promote adhesion of the basecoat. This coating was applied to the parts by the dip-spin process. Large parts can be coated by the rack-spray process. Figure 3 illustrates the process flowchart for surface preparation.

The application of basecoat was followed by an 18 minute curing cycle at roughly 250 °C part metal temperature. Once the topcoat was applied, the parts were cured for 18 minutes at roughly 225 °C metal part temperature. Figure 4 illustrates the process flowchart for the Magni 565 process.

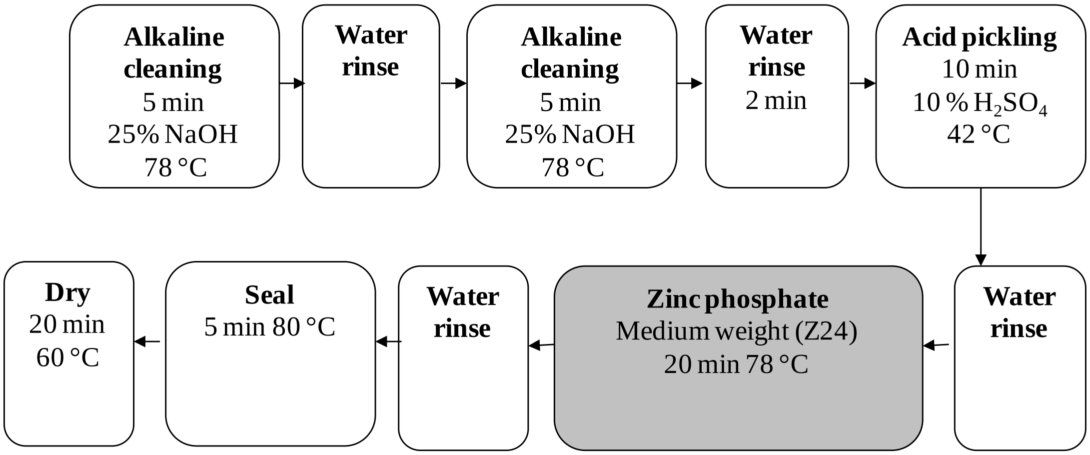

Figure 3 – Magni 565 phosphate pre-treatment flowchart

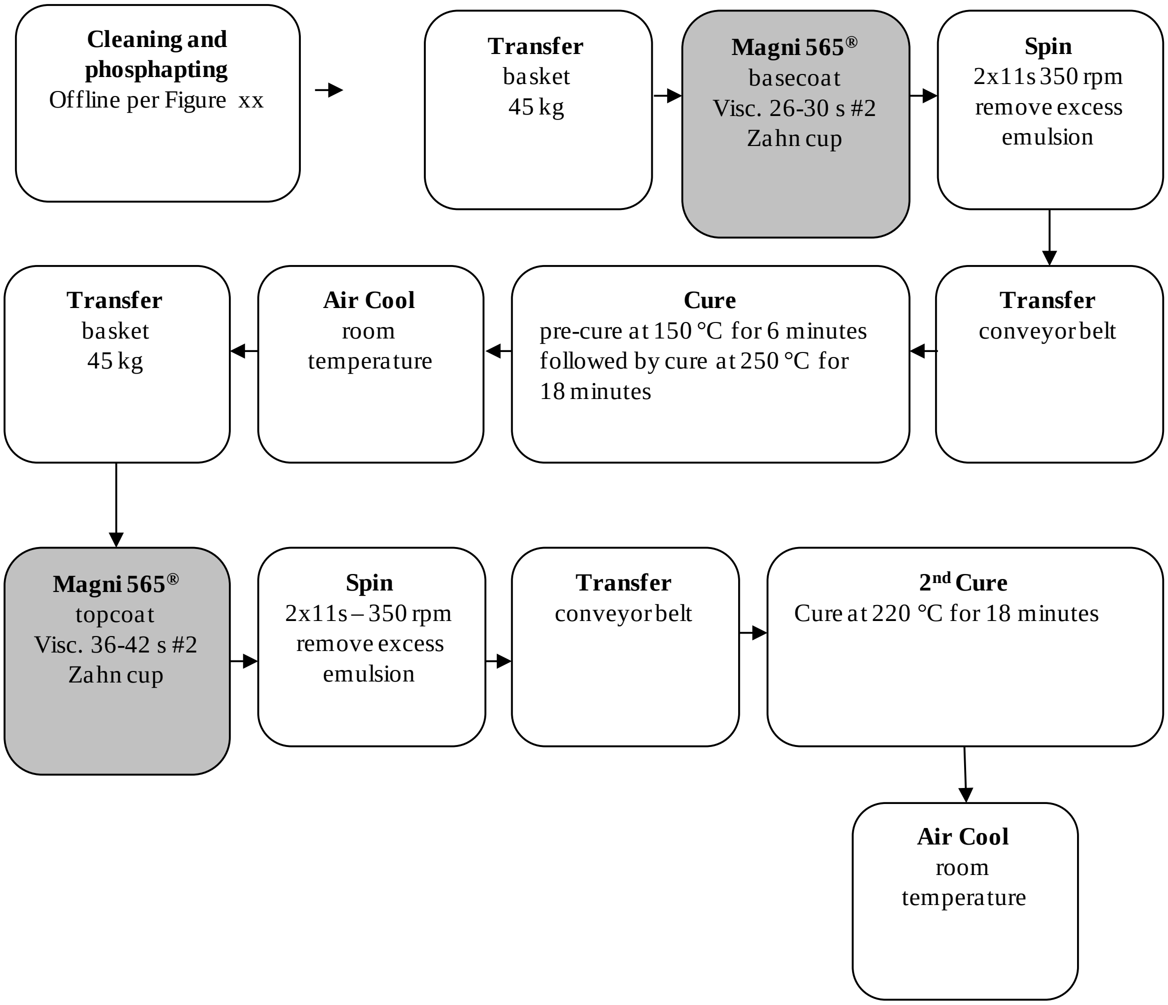

Figure 4 – Magni 565 process flowchart

4. EXPERIMENTAL METHODS

The following table lists the test methodologies in IFI-144, applied in this study. Relevant descriptive information about each test method will be given in the results section. In should be noted that the environmental hydrogen embrittlement test (Section 5.10) was performed in accordance with ASTM F1624, but the precise methodology and acceptance criteria was in accordance with a test method that has been drafted by a task group under Subcommittee F16.01 titled: "***Standard Test Method to Qualify Coatings for Use on A490 Structural Bolts Relative to Hydrogen Embrittlement***." This test method is pending final approval and is being developed under ASTM work item WK13659.

Table 9 – IFI-144 qualification test methods

Sect.	Test	Specification	Condition
2	Hardness	ASTM F606	N/A
2	Chemical Analysis	ASTM A751	N/A
5.1	Microstructure	ASTM E3	N/A
5.2	Coating Thickness	ASTM D1186	Coated
5.3	Adhesion	ASTM B571	Coated
5.4	Paintability	Visual	Coated
5.5	Rotational Capacity	FHWA/AASHTO	Coated
5.6	Salt Spray Exposure	ASTM B117 / ASTM D1654	Coated
5.7	Cyclic Exposure (conducted on bolts assembled in test blocks)	GM9540P	Bare
			Coated
5.8	Tensile Pull	ASTM F606	Bare (Pre Exposure)
			Coated (Post Exposure)
5.9	Hydrogen Embrittlement (process)	ASTM F1940	Bare
			Coated
5.10	Hydrogen Embrittlement (product)	ASTM F1624 – per F16.01 draft test method WK13659	Bare
			Coated

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 5 and 6 respectively.

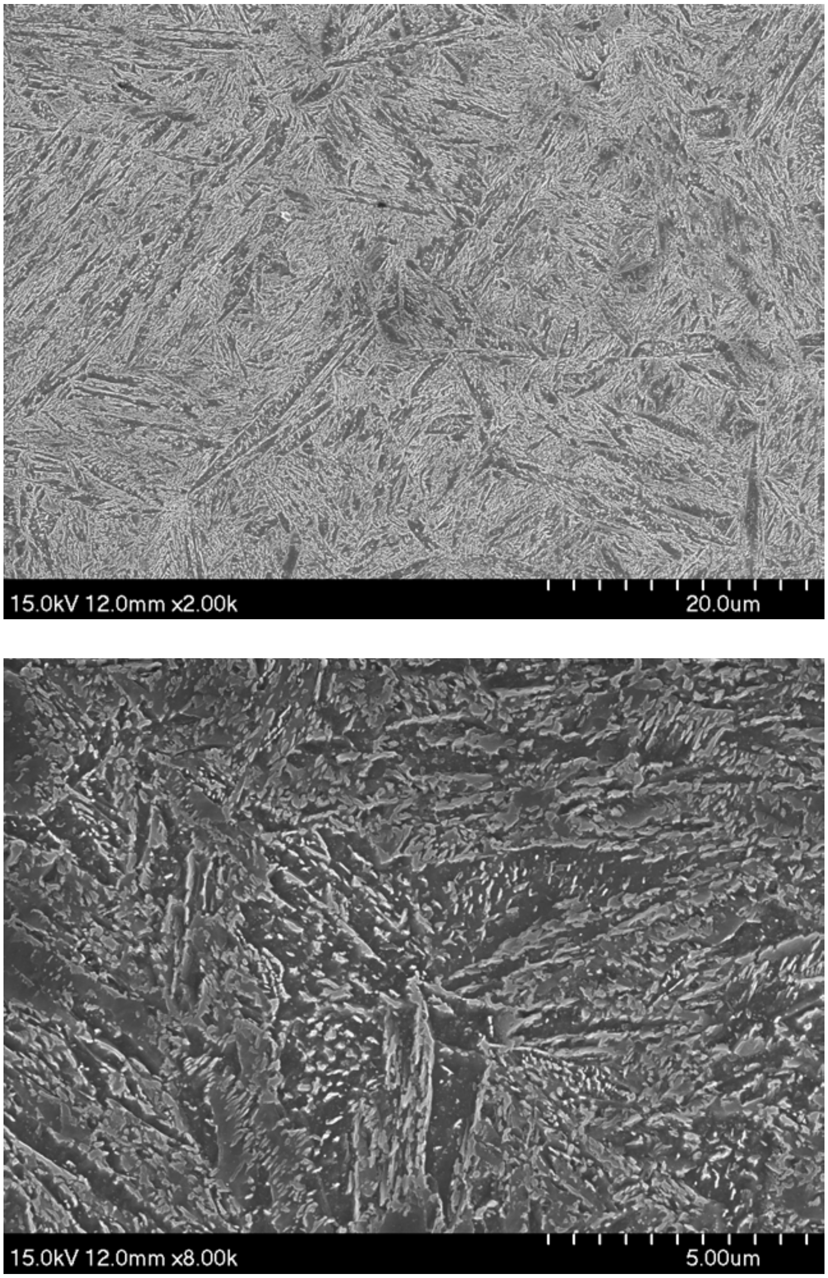

Figure 5 – Microstructure 1/2-13 x 3 specimen bolts (2,000 X and 8,000 X)

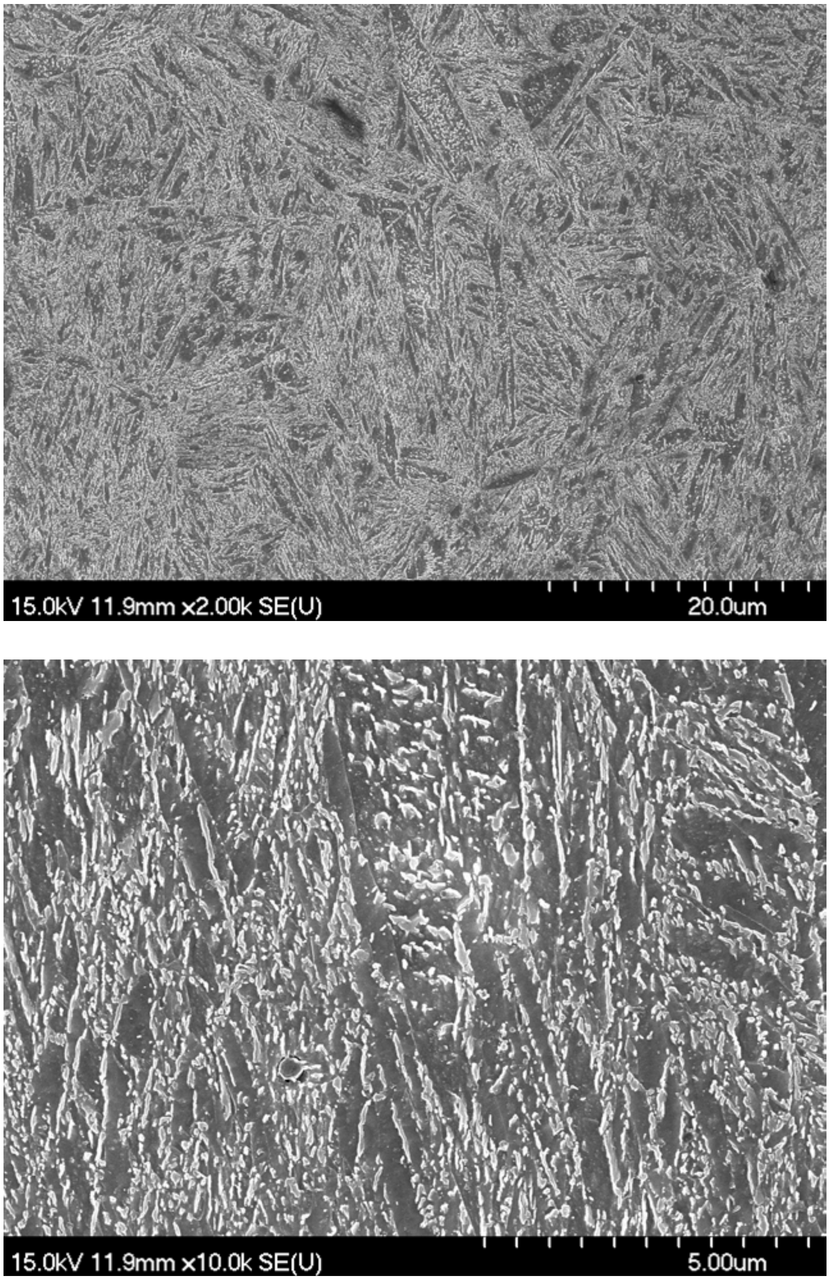

Figure 6 – Microstructure 3/4-10 x 3 specimen bolts (2,000 X and 10,000 X)

5.2 Coating thickness

Coating thickness was measured in accordance with ASTM D1186 using a magnetic induction tester, Elcometer model 355. Measurements were taken on the head of bolts and flats of nuts and washers. Sample size consisted of 10 pieces per lot, and 10 measurements per sample. The specified <u>average</u> thickness range for Grade 1 product specified in ASTM

F2833 is 0.47-0.59 mil. The results are presented in terms of average of average readings, and minimum and maximum readings on each test piece. The average thickness values for all bolts, nuts and washers were within the specified range. However, the standard deviation values represent roughly 50 % of the mean values. The variability of results may not only be a reflection of coating thickness variation. Readings may be affected by the measurement instrument and technique, and location of measurement. This degree of variability is large, but inherent to the coating process. The same degree of variability is present with ASTM F1136 coatings, and does not pose any concern with respect to the quality or resistance of the coating. The sole topic of concern is ensuring that coated nuts and bolts achieve satisfactory thread fit. These data reinforce the initiative being taken by committee F16 to develop standard oversize allowances for nuts used with A490 bolts coated by Zn/Al dispersion coatings.

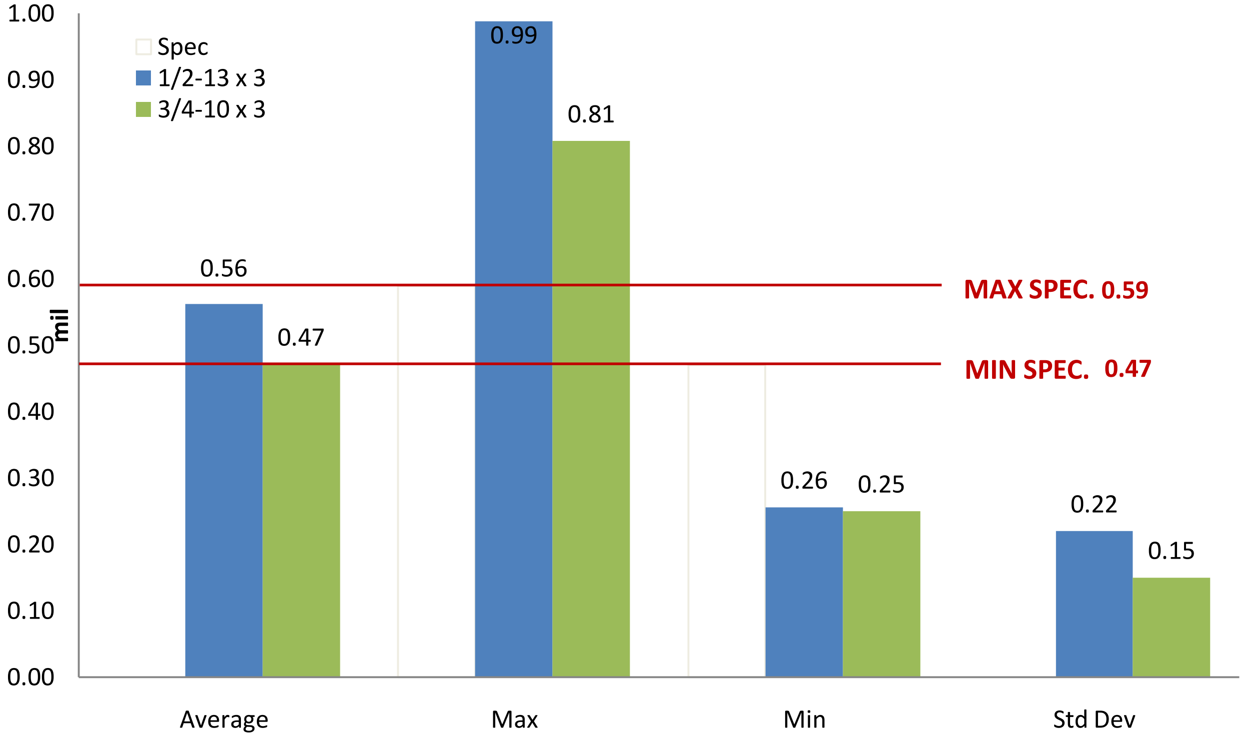

Figure 7 – Coating thickness results - bolts

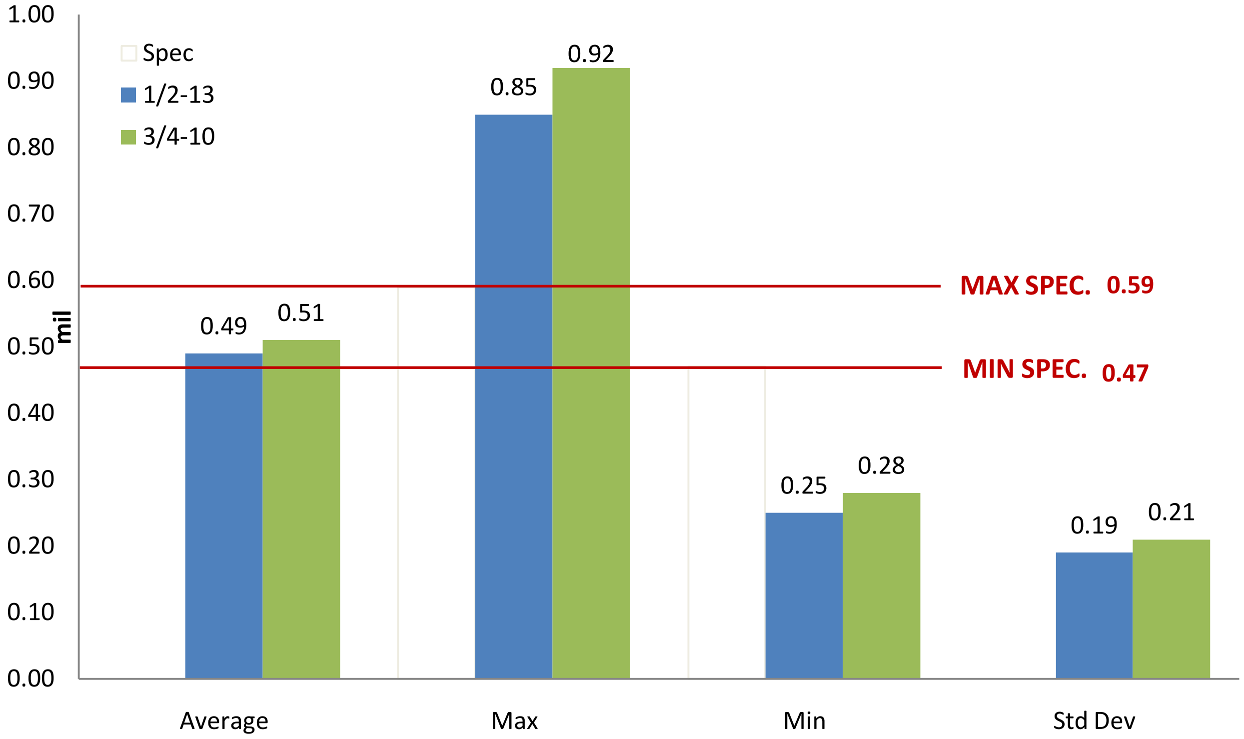

Figure 8 – Coating thickness results – nuts

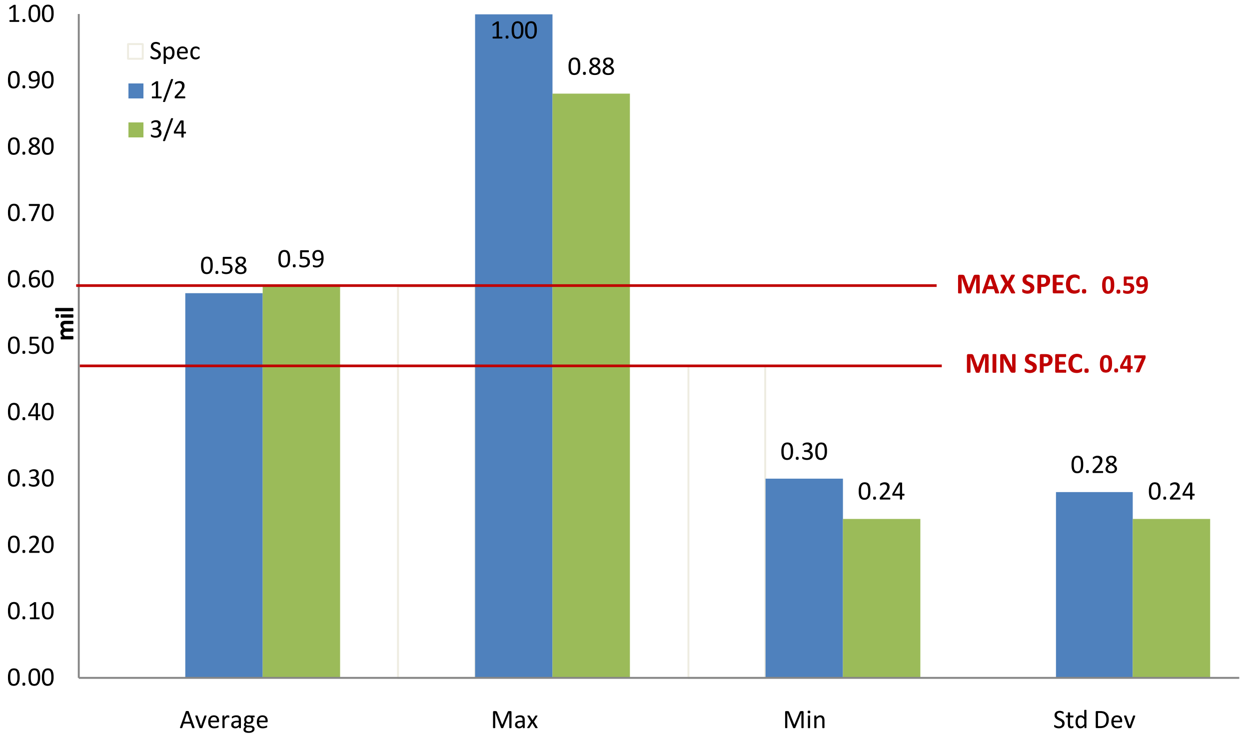

Figure 9 – Coating thickness results – washers

5.3 Adhesion

IFI-144 stipulates adhesion testing in accordance with ASTM B571. Similar to the qualification of ASTM F1136, the scribe-grid test (Test 13) was performed on the Magni 565 coated bolts. The sample bolts were scribed in three parallel lines with sufficient pressure to penetrate the coating and expose the substrate as shown in Figure 10. Five samples were tested, three in pristine state and two following handling and mechanical testing. Adhesion was visually satisfactory on all samples. The scribing was followed by removal of loose particles, followed by the application of tape (Permacel 99) to the area with firm finger pressure. The specified minimum bond strength of Permacel 99 is 45 g/mm. The tape was then removed rapidly at an angle of 180°. No part of the coating between the parallel lines broke away from the substrate, indicating satisfactory adhesion.

5.4 Paintability

IFI-144 requires paint to be applied to the coated fastener either by spraying or brushing. After 48 hours, the painted fastener is required to be dry to the touch. The paint system used for this test was Carbozinc 11 Primer and Carboxane 2000 TC, which are designed for use in structural applications. The same paint system was used during the qualification testing of ASTM F1136. Paint was applied to the entire surface of the Magni 565 coated bolts by brushing, and was allowed to cure for 48 hours at room temperature, after which it was verified to be dry to the touch. This satisfied the requirement in IFI-144. Images of primed and painted bolts are shown in Figure 11.

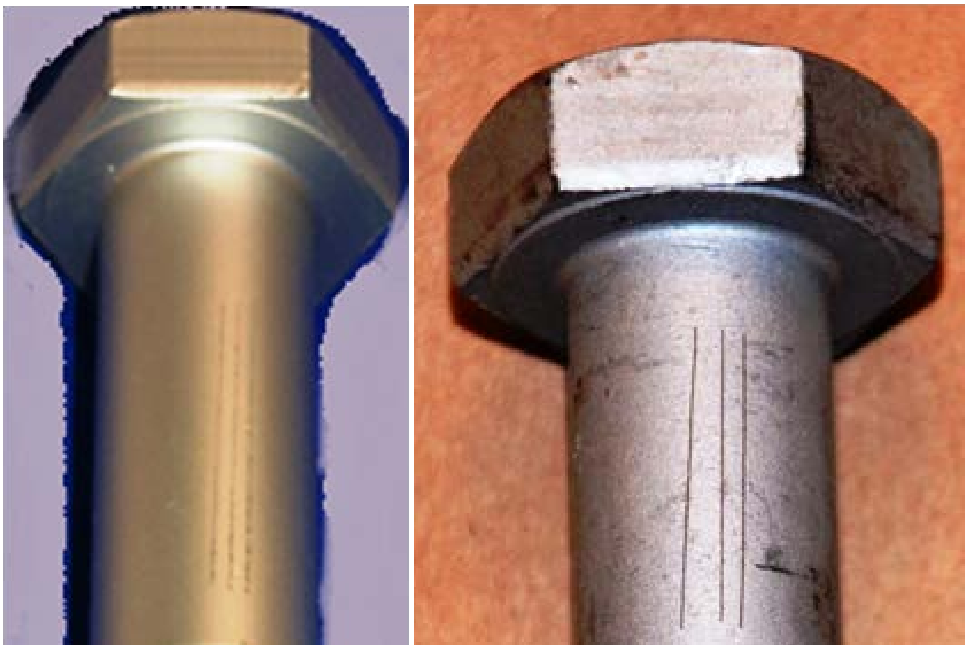

Figure 10a – bolts scribed before (left) and after mechanical testing (right)

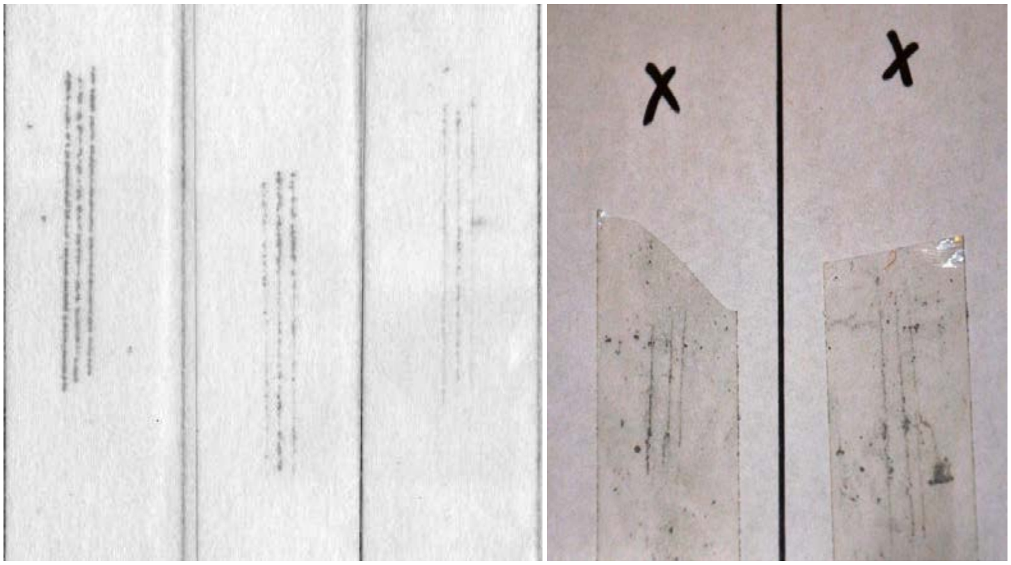

Figure 10b – Permacel tape samples showing satisfactory adhesion before (left) and after mechanical testing (right)

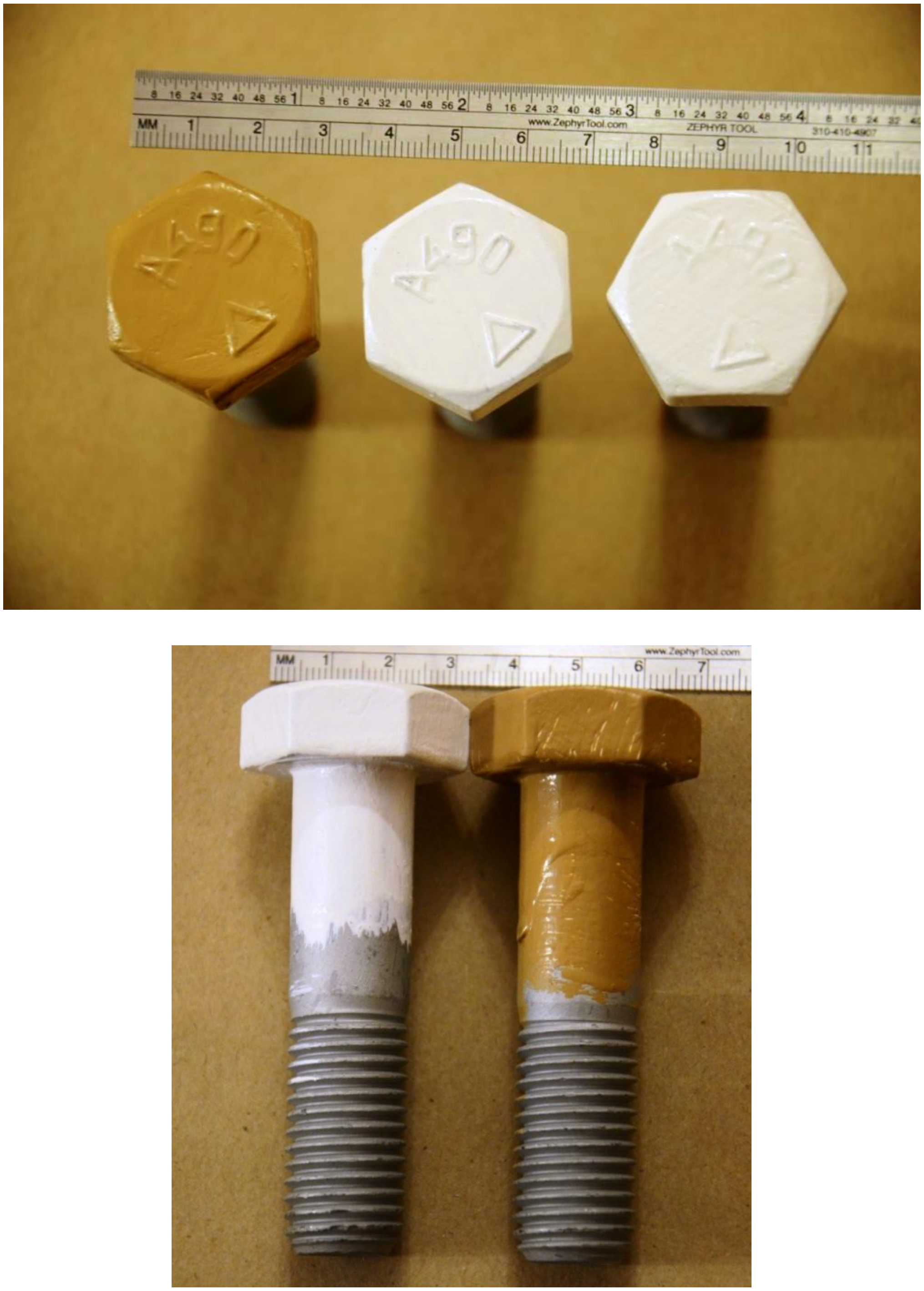

Figure 11 – Primed and painted bolt were dry to the touch after 48 h curing at room temperature

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). For the purpose of benchmarking, the RC tests were performed in both uncoated and Magni 565 coated conditions, using specimen bolts, nuts and washers. The Magni 565 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 coefficients of friction. Any coating on standard A490 bolts will normally be accommodated with oversized nuts to allow for free fitting assembly of coated bolts and nuts. The results for both test sizes are shown in Figures 12 and 13 respectively. For each size the data are presented in two charts: (i) angle vs. tension, and (ii) torque vs. tension.

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 test parameters for *rotational capacity* testing were in accordance with AASHTO and FHWA. The length (L) to diameter (d) ratios for the specimen bolts are as follows:

Table 10 – length over diameter ratios for specimen bolts

Bolt diameter d (in)	Length L (in)	L/d
0.50	3	6
0.75	3	4

Table 11 – Pre-installation verification testing and acceptance criteria

Bolt size (in)	Snug tension[3] (kips)	Min. installation tension[4] (kips)	Min. pre-installation verification tension[5] (kips)
1/2-13	2.25	15	16
3/4-10	5.25	35	37

Bolt Length (L)	Nut rotation from snug tension[6]
L ≤ 4d (not more than 4d)	120° (1/3 turn)
4d < L ≤ 8d (more than 4d but not more than 8d)	180° (1/2 turn)
8d < L ≤ 12d (more than 8d but not more than 12d)	240° (2/3 turn)

Table 12 – Rotational capacity testing and acceptance criteria

Bolt size (in)	Initial tension[7] (kips)	Installation tension[8] (kips)	Max. torque at installation tension[9] (ft-lb)	Min. final tension[10] (kips)
1/2-13	1.5	15	156	17.25
3/4-10	3.5	36	547	40.25

Bolt length (L)	Nut rotation from initial tension
L ≤ 4d (not more than 4d)	240˚ (2/3 turn)
4d < L ≤ 8d (more than 4d but not more than 8d)	360˚ (1 turn)
8d < L ≤ 12d (more than 8d but not more than 12d)[11]	480˚ (1-1/3 turn) or 420˚ (1-1/6 turn)

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

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

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

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

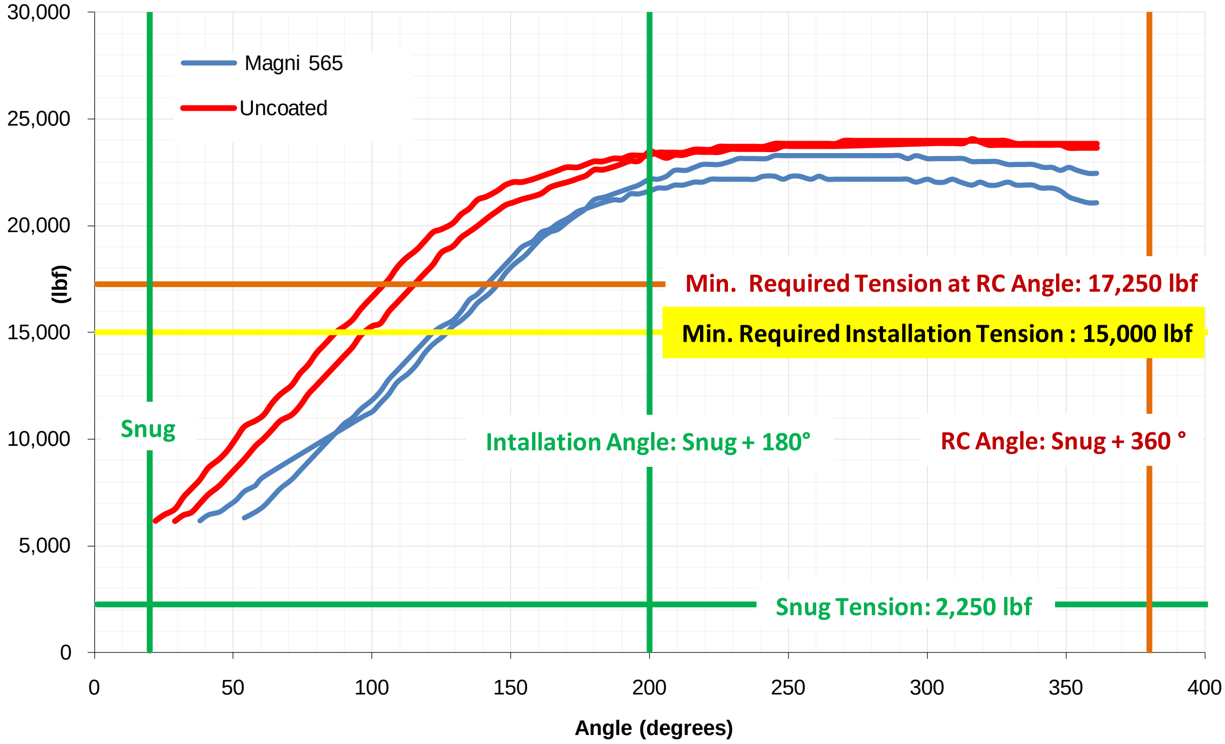
[7] Equal to 10% of installation tension, rounded off to nearest kip.

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

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

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

[11] 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.



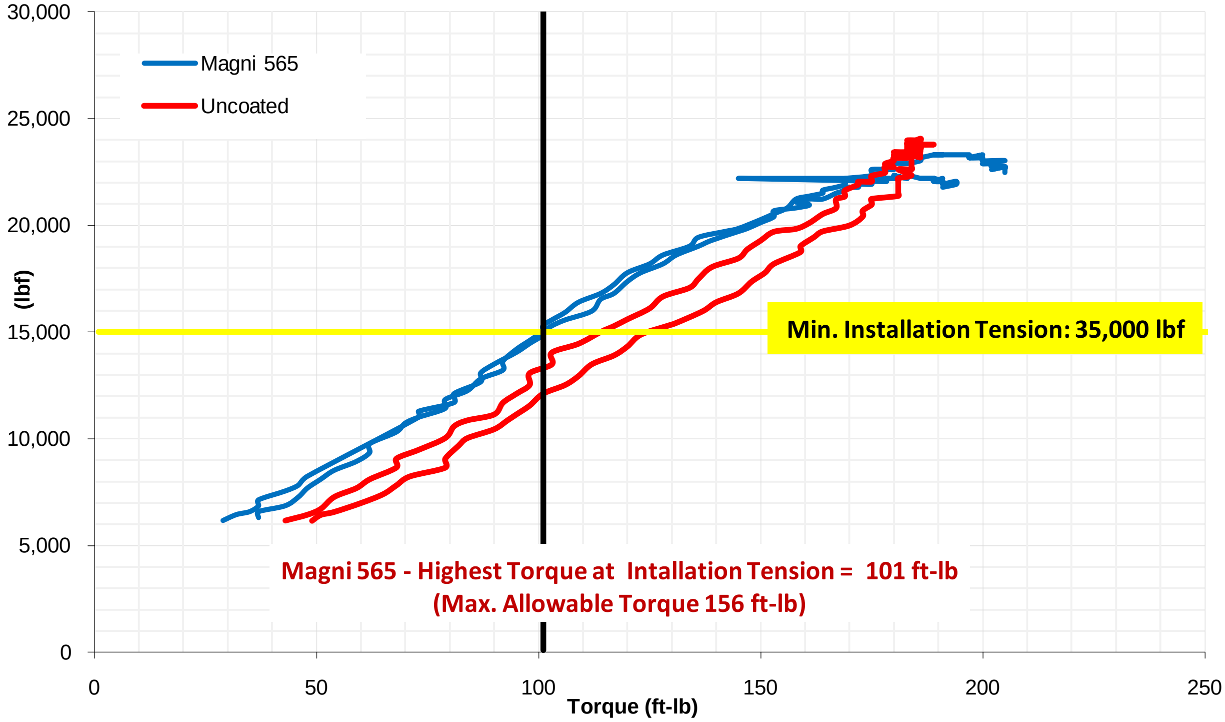
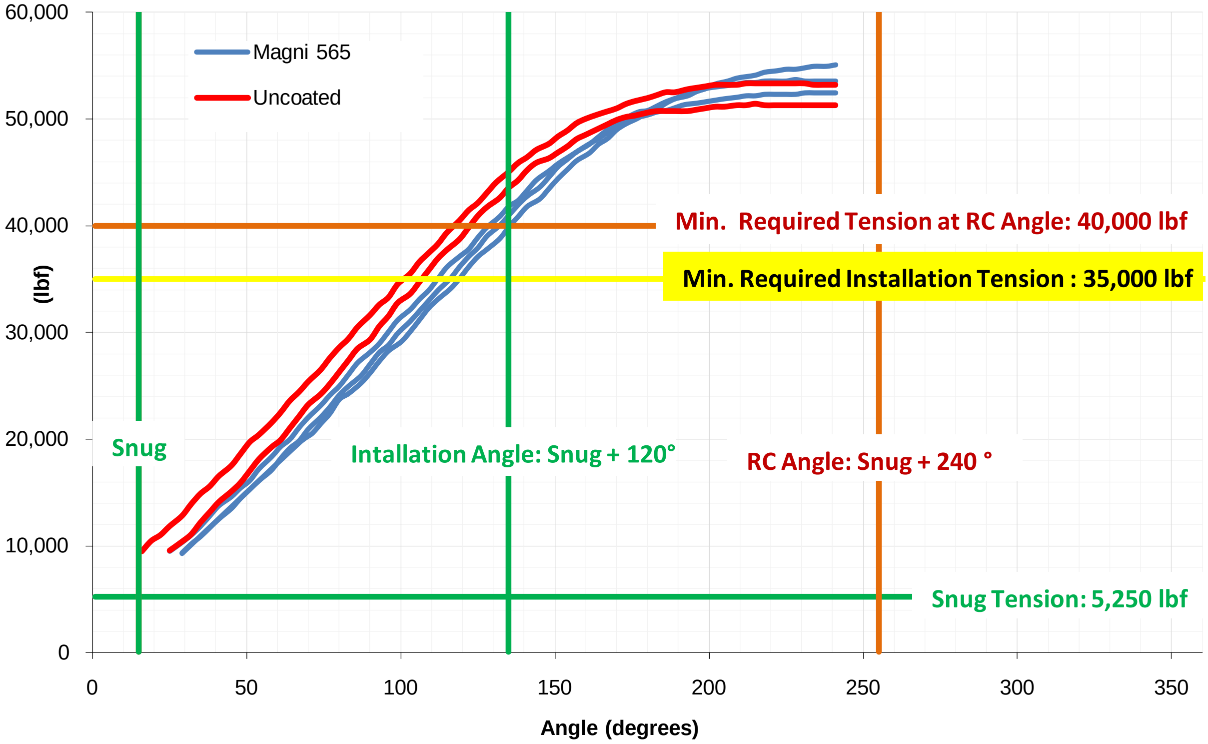


Figure 12 – RC test data for 1/2 inch parts: (i) angle vs. tension, and (ii) torque vs. tension



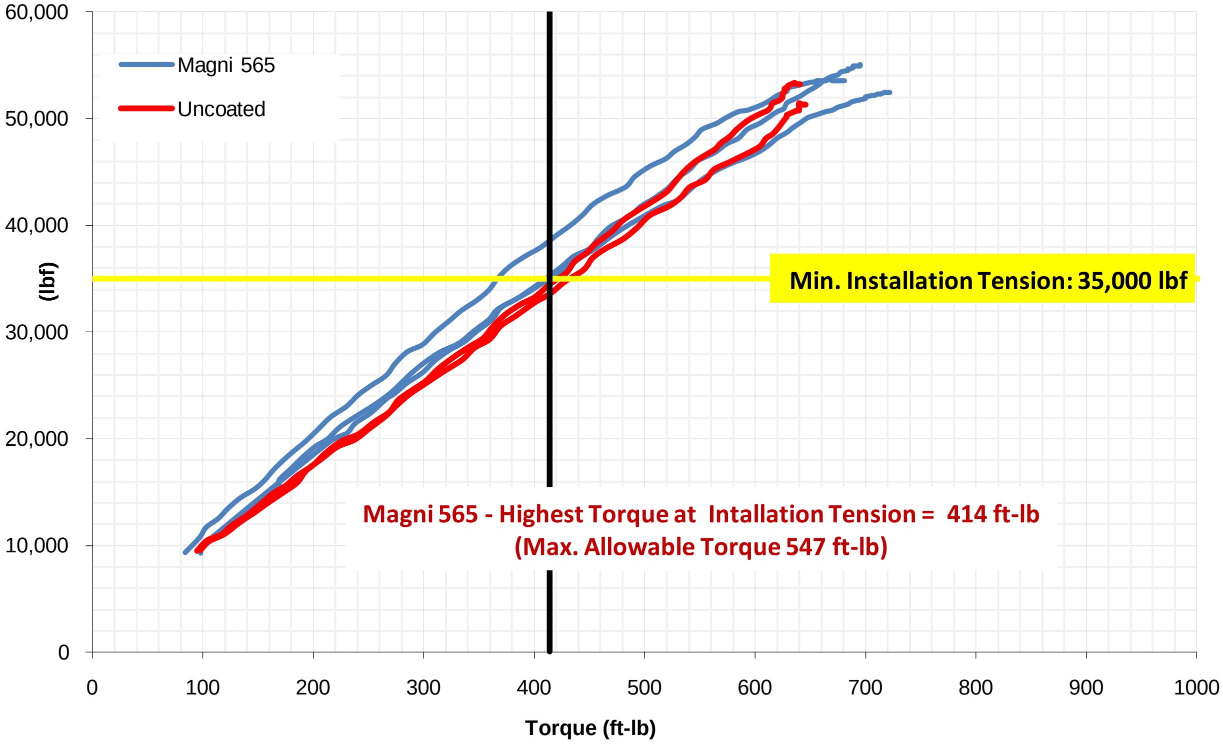


Figure 13 – RC test data for 3/4 inch parts: (i) angle vs. tension, and (ii) torque vs. tension

A closer look at average measured nut factor (K) values at installation tension illustrates the friction reducing effect of Magni 565. The average K value at installation tension for Magni 565 was 0.162 for 1/2 inch parts, and 0.181 for 3/4 inch parts. In comparison, average K values for uncoated parts ranged between 0.191 and 0.196. Again, it should be pointed out that the RC tests were performed without allowance for coating thickness between the mating nuts and bolts. Consequently, the K values for the Magni 565 are higher than if oversize nuts had been used, as would be expected for A490 bolts. Additionally, Magni 565 coated parts exhibited torque values that were well below maximum allowable torque at the end of the RC test rotation, as defined per AASHTO and FHWA[12].

The following is a brief commentary on the history and significance of rotational capacity testing with respect to A490 bolts. The rotational capacity test was initially instituted in ASTM A325 for galvanized product only. The purpose of the test was to ensure that mating bolts and nuts, especially the nuts, were sufficiently lubricated to overcome friction caused by the very rough galvanized surface. The test simply subjects the bolts to a nut tightening rotation that is typically (but not always) two times (2X) that which is needed for normal assembly. If the bolt can be readily disassembled without any sign of damage or failure, then it passes the test. The American Association of State Highway and Transportation Officials (AASHTO) followed by the Federal Highway Administration (FHWA) mandated RC testing for all A325 bolts, coated and uncoated. Additionally, the same A325 RC test procedures were extended to A490 bolts, this despite the fact that metallic coatings were not permitted on A490 bolts. Furthermore, the AASHTO and FHWA versions of the RC test contain additional acceptance criteria, namely maximum allowable torque readings and a minimum pretension at final rotation. The applicability of A325 performance criteria to A490 bolts has never been adequately addressed. The problem with extending the RC test to A490 bolts is that A490 bolts are significantly higher in strength than A325 bolts, 150 ksi min. versus 120 ksi min. Consequently, A490 bolts do not exhibit the same ductility and elongation. The degrees of rotation stipulated in A325 RC test procedures may not have the

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

intended outcome with A490 bolts. Despite the lack of experimental data, AASHTO and FHWA RC test specifications for uncoated A490 and A490M bolts are being applied as written with inconsistent and even questionable outcomes in the field. This situation has persisted due to the infrequent use of A490 bolts in road and bridge work. The application of a coating such as Magni 565, with its superior friction reduction/control characteristics will undoubtedly improve the capability of A490 and A490M bolts to pass the RC test criteria, pending the future evolution of RC test criteria towards being appropriately adapted to A490/A490M bolts.

5.6 Salt spray exposure

Magni 565 coated bolts of both 1/2" and 3/4" sizes were exposed in a salt spray chamber in accordance ASTM B117, for a total exposure time of 5000 h. Note that IFI-144 only requires 1000 h of exposure. The chamber was opened periodically for inspection, and photographs were taken at 820 h, 1000 h, 3000 h and 5000 h. There was no evidence of red rust after 1000 h of exposure on any of the samples for both sizes (Figures 14-15). The first signs of red rust were apparent only after 2000 h of exposure. At 3000 h red rust was visible, but primarily at the corners of the hex heads, covering 10 -15% to the head surface. After 5000 h of exposure, roughly 50-80% of the surface of the heads exhibited red rust. Detailed images are given in Appendix A. Estimation of percentage of red rust was performed in accordance with ASTM D1654.

Figure 14 –3/4 inch bolts after 1000 h of salt spray exposure

Figure 15 –1/2 inch bolts after 1000 h of salt spray exposure

5.7 Cyclic exposure

IFI-144 requires that cyclic testing be performed for an exposure period of 80 cycles on bolt/nut/washer assemblies mounted into fixtures in the loaded and unloaded conditions. Cyclic testing is to be done with in accordance with GM 9540P. The fixtures consisted of hardened steel cylinders that were painted with Carbozinc 11 Primer and Carboxane 2000 TC. In this study, only 3/4 inch bolts, nuts and washers were utilized for cyclic testing. The test pieces were exposed for a total of 120 cycles and evaluated for visual corrosion and weight loss. The intent of performing the test on bolts that are under service load is to simulate “worst case” service conditions for stress corrosion cracking (SCC). To achieve the loaded condition the bolts/nuts/washers were assembled in fixtures by the turn-of-nut method (see Table 11), while the unloaded bolts were finger tightened into the fixtures. Following exposure to 40, 80, and 120 cycles respectively, the parts were rinsed with warm water and visually evaluated for red rust. The corrosion weight loss of exposed parts, each component and the entire assembly were weighed prior to and following exposure. An uncoated control bolt (Bare) was used for baseline comparison with the Magni 565 coated parts. The results showed that Magni 565 coated parts did not experience any significant weight loss. Similar to previous findings with ASTM F1136 coatings, a slight weight gain was observed which can be attributed to the presence of oxides and residues. In comparison, the control bolt exhibited measurable weight loss in the order of 22 mg. Test conditions are given in Table 13. The visual corrosion results are given in Table 14, and weight loss measurements are given in Table 15.

Table 13 – Cyclic test conditions

Coating	Tightening Condition	Exposure	Sample Size
Bare	Finger tightened	120 Cycles	1
Magni 565	Finger tightened	120 Cycles	5
Magni 565	Turn-of-nut ½ turn	120 Cycles	5

Table 14 – Visual estimation of red rust during cyclic testing

Turn-of-Nut Tightened Red rust on head (rating scale: 0-10)			
	40	**80**	**120**
Magni 565	0	0+	<1
	0	0	<1
	0	0	<1
	0	0+	<1
	0	0	<1
No Load Red rust on head (rating scale: 0-10)			
	40	**80**	**120**
Magni 565	0	0	<1
	0	0+	<1
	0	0	<1
	0	0	<1
	0	0+	<1
Control (Bare)	8	10	10

Table 15 – Weight change (mg) following 120 cycles of exposure

	Bolt	Nut	W1	W2	Fixture	Assy
Magni - Turn-of-Nut Tightened	0.05	0.04	0.01	0.04	0.00	0.14
Magni - No Load	0.06	0.03	0.02	0.01	0.00	0.12
Bare – No load	(11.30)	(5.21)	(3.15)	(1.95)	0..00	(21.61)

Corrosion coupons: 80 cycle mass loss: 8,120 mg
120 Cycle Mass Loss: 10,515 mg

5.8 Post exposure tensile tests

IFI-144 requires that bolts be axially tested after salt spray and 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 1/2-13 x 3 specimen bolts were tensile tested after 5000 h salt spray exposure. The 3/4-10 x 3 specimen bolts were tensile tested after 5000 h of salt spray exposure and after 120 cycles of cyclic exposure. 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 16 and 17 and shown in Figures 16 and 17. The results demonstrate that Magni 565 coated bolts did not exhibit any measurable loss of strength following 5000 h of continuous salt spray exposure or 120 cycles of cyclic exposure.

Table 16 – Pre and post exposure tensile test results for 1/2-13 specimen bolts

	Uncoated	Coated	5000h SS
Sample	**Load (lbf)**	**Load (lbf)**	**Load (lbf)**
1	27,530	27,100	27,200
2	26,960	27,010	26,990
3	27,245	27,650	27,350
4	27,300	26,970	27,260
5	26,950	27,320	27,110
Avg.	**27,197**	**27,210**	**27,182**

	Uncoated	Coated	5000h SS
Sample	**Stress (psi)**	**Stress (psi)**	**Stress (psi)**
1	194,010	190,980	191,684
2	189,993	190,345	190,204
3	192,001	194,856	192,741
4	192,389	190,063	192,107
5	189,922	192,530	191,050
Avg.	**191,663**	**191,755**	**191,557**

Tensile Stress Area 0.1419 in^2

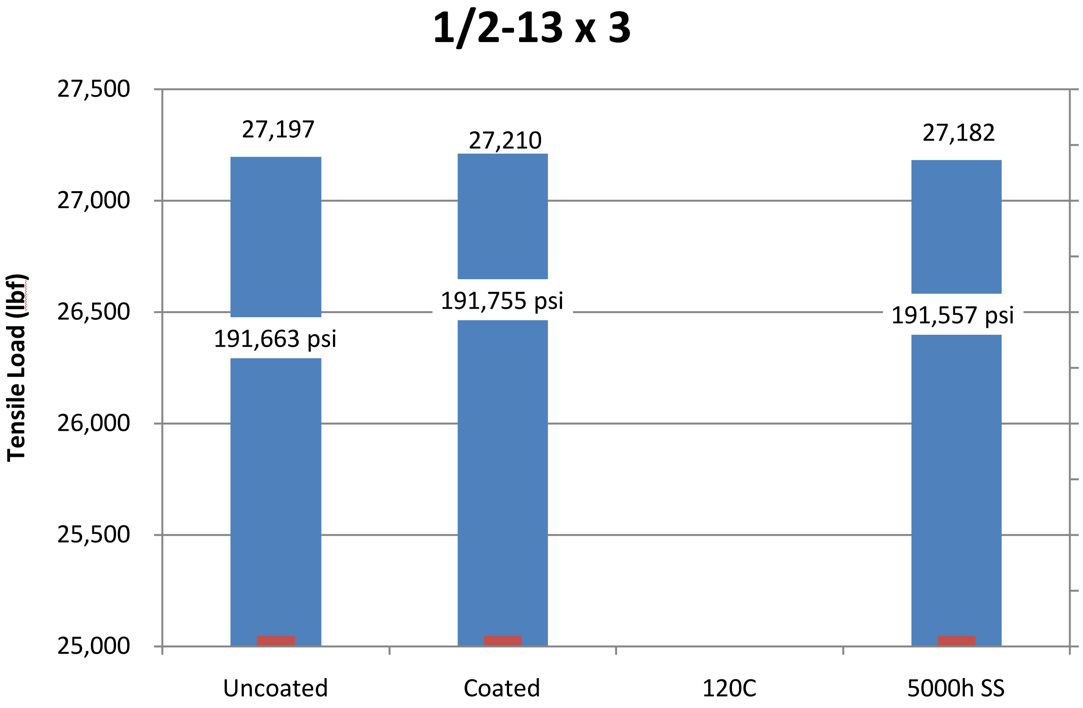


Figure 16 – Pre and post exposure tensile test results for 1/2-13 specimen bolts

Table 17 – Pre and post exposure tensile test results for 3/4-10 specimen bolts

	Uncoated	Coated	120C	5000h SS
Sample	**Load (lbf)**	**Load (lbf)**	**Load (lbf)**	**Load (lbf)**
1	61,530	60,350	60,510	61,150
2	59,660	60,920	60,730	60,650
3	60,530	60,580	61,410	60,940
4	61,390	60,250	60,350	60,420
5	61,590	61,700	60,200	60,550
Avg.	**60,940**	**60,760**	**60,640**	**60,742**

	Uncoated	Coated	120C	5000h SS
Sample	**Stress (psi)**	**Stress (psi)**	**Stress (psi)**	**Stress (psi)**
1	184,222	180,689	181,168	183,084
2	178,623	182,395	181,826	181,587
3	181,228	181,377	183,862	182,455
4	183,802	180,389	180,689	180,898
5	184,401	184,731	180,240	181,287
Avg.	**182,455**	**181,916**	**181,557**	**181,862**

Tensile Stress Area 0.3340 in^2

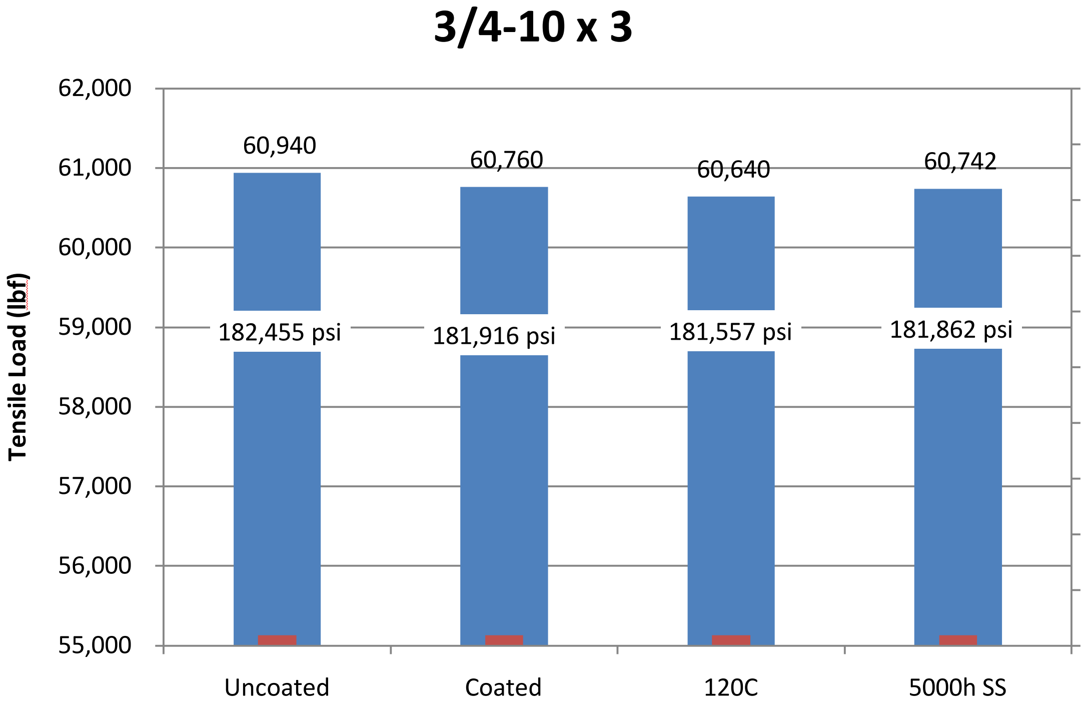


Figure 17 – Pre and post exposure tensile test results for 3/4-10 specimen bolts

5.9 Hydrogen embrittlement – process qualification

The incremental step load test method described in ASTM F1940 was used to quantify the risk of internal hydrogen embrittlement (IHE) posed by the Magni 565 coating process. In this test the coated notch bar is subjected to a sustained four-point bending load and slow strain rate under displacement control. [1] The test indirectly quantifies the amount of residual hydrogen introduced during the coating process by measuring the hydrogen embrittlement threshold strength of the notch bar using a standardized loading protocol in air as specified in ASTM F1940. The threshold, also called 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) SQB specimens were tested in the same manner to establish a baseline Notch Fracture Strength. The ratio of the threshold for each witness test specimen over the baseline represents the percent Notch Fracture Strength (%NFS).

$$NFS\% = \frac{NFS_{(W)F1940}}{NFS_{(B)F1940}} \times 100$$

Where:

NFS% = Percent Notch Fracture Strength
NFS(W)F1940 = Notch fracture load of coated SQB witness specimen
NFS(B)F1940 = Notch fracture load of bare SQB specimen

Two separate Magni 565 processing methods were tested. In the first included an acid pickling step (10% v/v sulfuric acid, 42 °C, 10 min) in the phosphate pre-treatment cycle. The second method did not include any acid pickling in the phosphate pre-treatment cycle. The sampling plan was designed to isolate the effect (if any) of acid pickling on notch fracture strength. Five (5) notch bars were exposed simultaneously for each of the two process conditions. The results showed no reduction of NFS% for both methods with average values of 99.5% and 99.1% respectively (Fig. 18). It should also be noted that that unlike the ASTM F1136 coating process, the lower curing temperature of the Magni 565 process (250 °C vs. 320 °C) did not alter the mechanical properties of the notch bars. These demonstrate that the Magni 565 coating process does not introduce any residual hydrogen, and therefore does not pose any risk of internal hydrogen embrittlement.

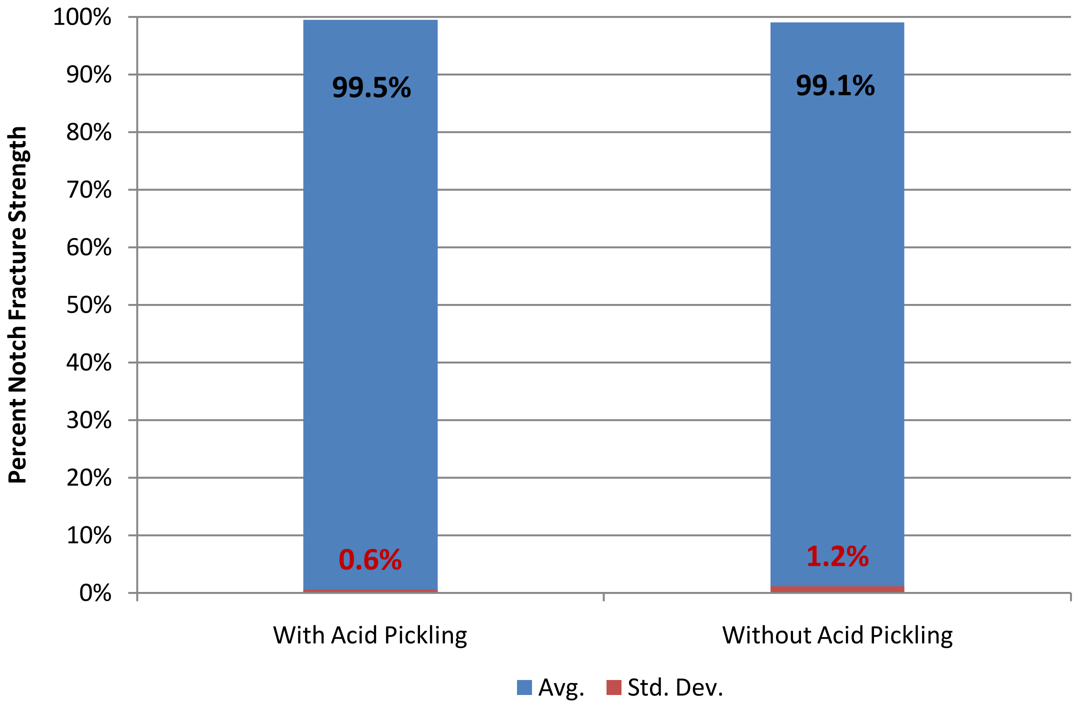


Figure 18 –NFS% values obtained for Magni 565, with and without acid pickling – the results clearly indicate that there is no risk of IHE for both conditions

Surface and core hardness of the notch bar specimens was measured before and after coating to verify if the curing temperatures had caused any decrease in strength. There was no measurable change in specimen hardness after the Magni 565 coating (Fig. 19). These results confirm that the ASTM F1940 process sampling did not deviate from the standard test conditions. In comparison, the higher curing temperatures of the ASTM F1136 (Dacromet) process did lower specimen hardness as was shown in a previous study, which required additional interpretation given the deviation from the ASTM F1940 standard test conditions.

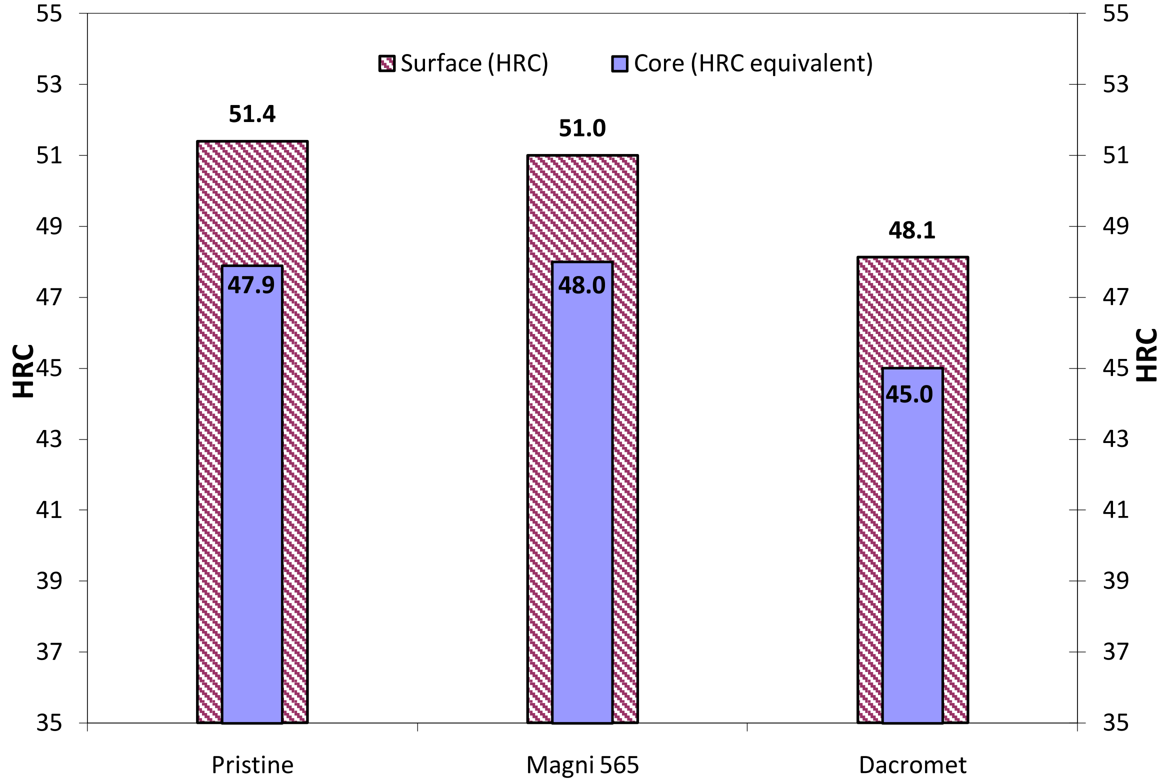


Figure 19 – Hardness of pristine and Magni 565 coated ASTM F1940 notch bar specimens showing no reduction in hardness from curing temperatures, in comparison to Dacromet coated specimens (Source for Dacromet: [2-3])

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 Magni 565 was measured and then compared to a measurement of the OCP for Dacromet. As can be seen from Figures 20 and 21, OCP values for both coatings, after 24h of stabilization, was in the order of -1.00 V. These results mean that Magni 565, similar to Dacromet is indeed sacrificial, but its corrosion potential is closer to that of steel than zinc. In other words, Magni 565 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

Closer scrutiny of the OCP test progression (Fig. 20) reveals that Magni 565 and Dacromet began at the same OCP value of roughly -0.993 V, but moved in opposite directions before stabilizing. This observation may be explained by the effects of the topcoats for each individual coating. Once the topcoat was consumed, the two coatings had nearly the same corrosion potentials. After 24 hours of stabilization the OCP for Magni 565 was -1.001 V, whereas the OCP for Dacromet was -0.991 V, a mere difference of 10 mV.

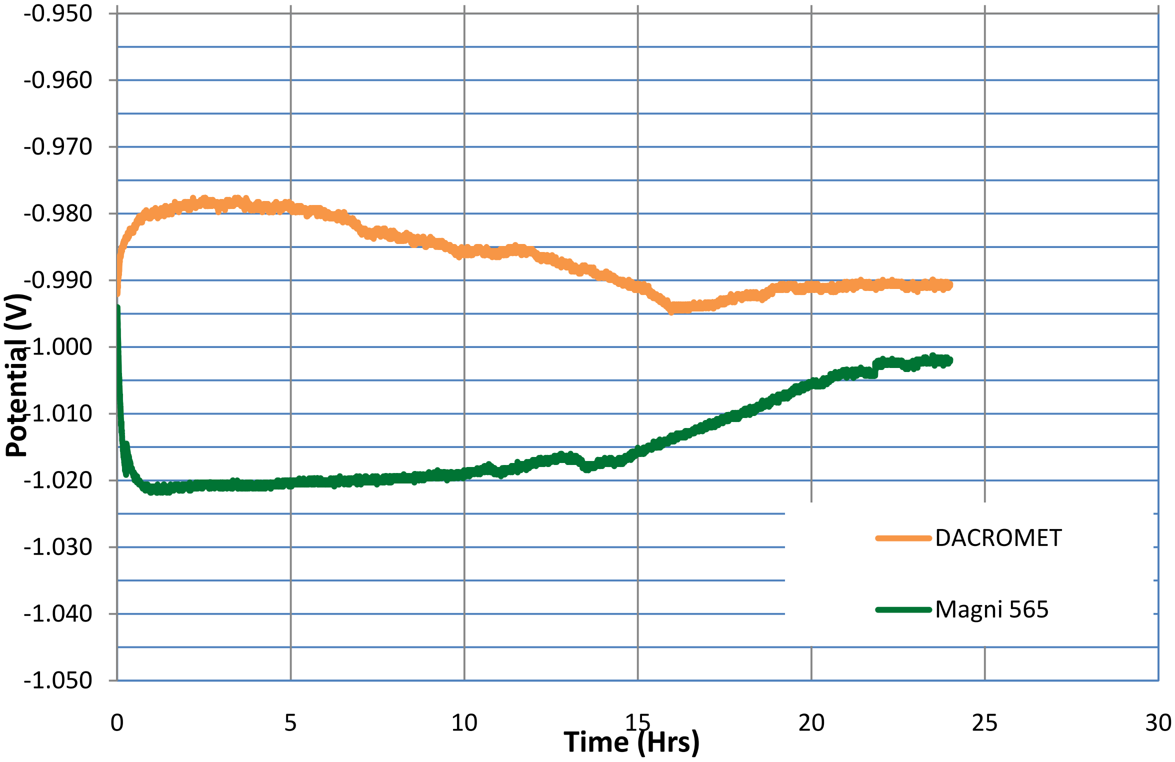


Figure 20 – OCP test progression for Magni 565 and Dacromet over 24 h period

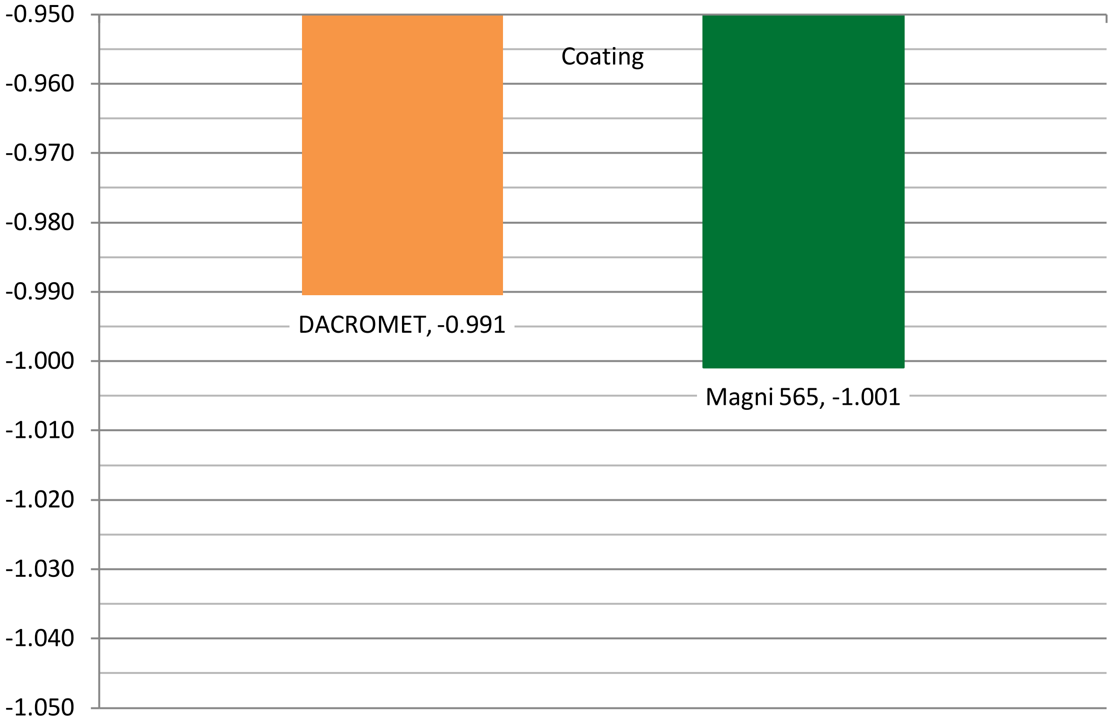


Figure 21 – OPC values for Magni 565 and Dacromet after 24 h of stabilization

Having established the corrosion potential for Magni 565, the methodology described in ASTM F1624 "***Standard Test Method for Measurement of Hydrogen Embrittlement in Steel by the Incremental Loading Technique***" was applied. More specifically ASTM F1624 was adapted to testing coated fasteners in a test method titled "***Standard Test Method to Qualify Coatings for Use on A490 Structural Bolts.***" This test method has been developed by an ASTM Subcommittee F16.01 task group under work item number WK13659, and has been submitted to Committee F16 for main committee balloting.

The scope of the test method in WK13659 states:

"***1.1 This test method defines the procedures and tests to evaluate the effect of a coating system on the susceptibility to hydrogen assisted failure of an ASTM A490 high strength structural bolt.***

1.2 This test method shall qualify a coating system for use with A490 high strength structural bolts, regardless of steel composition.

> ***1.3 The characteristic to be evaluated by this test method is the susceptibility to environmental hydrogen embrittlement (EHE) caused by hydrogen generated from corrosion protection of the steel bolt by sacrificial galvanic corrosion of the coating. Testing shall be performed on coated ASTM A490 bolts under the maximum susceptible hardness condition of the bolt (specimen bolt). The internal hydrogen embrittlement (IHE) susceptibility will also be inherently evaluated when the EHE is tested through this test method. There is no need for a separate IHE susceptibility test.***
>
> ***1.4 This test method shall qualify a coating system for use with any size of A490 bolts (e.g. 1/ 2 to 1-1/2 in).***"

The methodology applied in this study was more exhaustive in its scope, number of samples, and conditions tested than the requirements in WK13659. A detailed description of the methodology is as follows.

The test environmental hydrogen embrittlement test was conducted on 3/4-10 bolts. The specimen bolts were truncated by removal of the bolt head. The thread length (~1.4 in) was then inserted into the thread gripping fixtures to achieve the four-point bending, with a minimum of two threads between the gripping devices as shown in Figure 22. The loading method was four-point pure bending under displacement control. [1]

The first step in the testing sequence was a measurement of the *fast fracture* load of the specimen bolts in bending by performing a test in accordance with ASTM F1624, Section 8. Fifteen (15) uncoated specimen bolts and fifteen (15) coated specimen bolts were tested in using the fast fracture protocol. The average of these results was used to determine the fast fracture strength of each condition.

The average fast fracture strength in bending of coated bolts, *FFS(B) coated* had a standard deviation of 2% and was well within 2% of the average fast fracture strength for uncoated bolts, *FFS(B)uncoated*. Therefore, as was demonstrated with the F1940 test method, the Magni 565 coating process did not affect the strength of the specimen bolts.

To measure the EHE susceptibility of the fastener/coating system, bolts were tested in the three (3) separate environmental conditions using the incremental step load (ISL) methodology described in ASTM F 1624, to measure the hydrogen embrittlement threshold load P_{th}. All of the conditions tested are shown in Table 18 below.

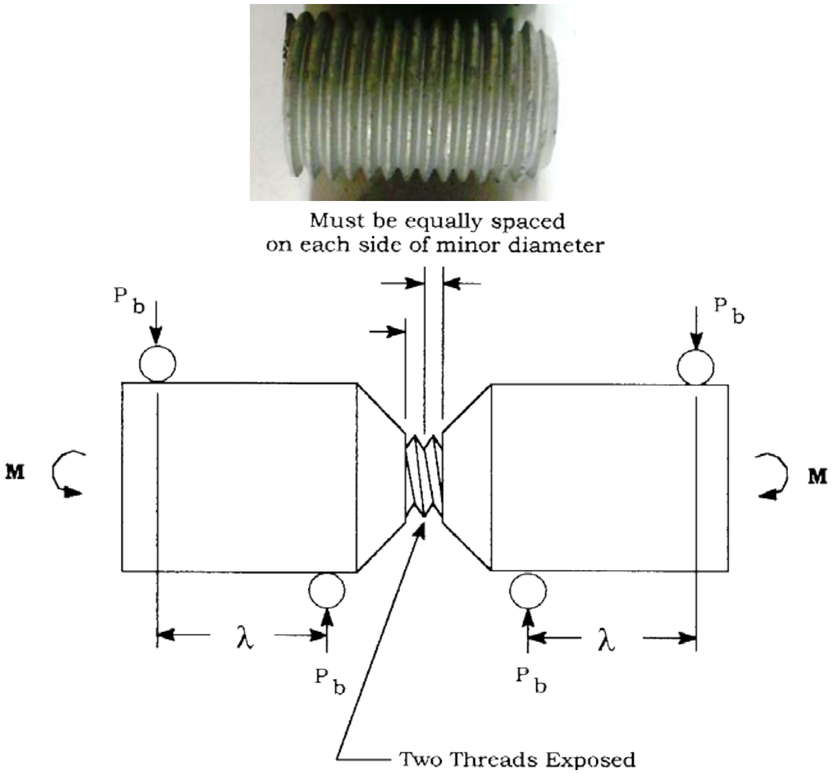


Figure 22 – Truncated bolt and schematic of bend test fixture (Source: ASTM WK 13659)

Table 18 – Environmental test condition

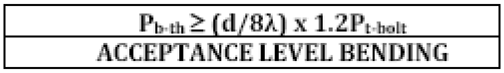
Test Method	Environment	Specimen Condition	Samples Size
FF	Air	Bare	15
		Coated	15
ISL	Air	Bare	5
		Coated	5
ISL	1.2V Imposed Potential in 3.5% NaCl solution	Bare	5
		Coated	5
ISL	**No imposed Potential in 3.5% NaCl solution (free corrosion)**	Bare	5
		Coated	5
		Scribed coating	**5**

The focal condition for determining the EHE susceptibility of the fastener/coating system is the simulation of a galvanic corrosion condition, highlighted in red in Table 18. 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 specimen were immersed in an environmental chamber filled with 3.5 % NaCl solution. The bolts and fixtures were each isolated from other metal contacts using teflon tape. A reference electrode was placed in close vicinity to the scribe mark. In the case of imposed potential, a platinum wire was used as anode.

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 used in the calculation described in the equation shown in Table 19.

Table 19 – Equation for minimum acceptance threshold load $P_{b\text{-}th}$
(Source: ASTM WK 13659)[13]



$P_{b\text{-}th} \geq (d/8\lambda) \times 1.2P_{t\text{-}bolt}$
ACCEPTANCE LEVEL BENDING

Bolt Size Equivalence: WK 13659 establishes that a smaller diameter bolt size can be tested to satisfy the acceptance criterion for a larger bolt size diameter if it attains a

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[13] ASTM F519 Type 1a specimens that require a threshold stress equal in value to that of 75% of the notch tensile strength (NTS = 1.6 UTS) of the Type 1a specimen, which is equal to or greater than 1.2 UTS. To obtain the equivalent acceptance criterion for the coated specimen colts, the net tensile stress at the root of the thread should also be equal to or greater than 1.2 UTS. Since d/D for fasteners is always greater than 0.8, that is greater than 0.7 used with ASTM F519 notched tensile specimen, the fastener must be tested in bending to attain the same stress level.

The acceptance criterion for EHE must be a threshold load in bending that produces the same stress or greater than the load in tension that produces a stress of 1.2 UTS. Since the limit load of a bolt in bending equals 2.3YS ≈ 2.0 UTS; the acceptance criterion for EHE is equivalent to ≥ 60% of the fast fracture load in bending (i.e., FFS(B) coated).

threshold load with a higher value than the minimum value of 60% of the fracture load in bending. This equivalence among the different sizes is summarized in Table 20.

Table 20 – Bolt size equivalence for acceptance (source: ASTM WK 13659)

Bolt Diameter	**1/2"D**	**3/4"D**	**1.0"D**	**1.5"D**
1/2"D	**≥ 60% FFS(B)**	≥ 72% FFS(B)	≥ 82.2% FFS(B)	≥ 98.3% FFS(B)
3/4"D	N/A	**≥ 60% FFS(B)**	≥ 68.5% FFS(B)	≥ 81.9 FFS(B)
1.0"D	N/A	N/A	**≥ 60% FFS(B)**	≥ 71.8% FFS(B)
1.5"D	N/A	N/A	N/A	**≥ 60% FFS(B)**

The results obtained in this study for both specimen bolt sizes are shown in Figures 23 and 24. **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 Table 20 are used. For 3/4 inch specimen bolts the threshold value must equal or exceed 81.9 % of the baseline (FFS(B)) in order to be applicable to 1-1/2 inch bolts. From figure 26, a threshold value of 83% was obtained for the scribed condition. Consequently, the result satisfies the acceptance criterion up to 1-1/2 inch. These test results demonstrate the Magni 565 coating satisfies the acceptance criteria for EHE on A490 bolts of all sizes.

Additional to the immediate objective of qualifying Magni 565 for use on ASTM A490 bolts, a great amount of information may be extracted from the test data. For example, the ISL tests performed in air exhibited threshold strengths at 91-93 % of baseline strength. This loss of strength can be attributed to the residual (diffusible) hydrogen that already existed in the steel. Also, valuable information can be had from the ISL tests performed at imposed potential of – 1.2 V. These data represent the hydrogen susceptibility of the steel under hydrogen saturation conditions. Threshold values were understandably lower than for tests performed under free corrosion conditions. The in-depth analysis of these data exceeds the scope of this report.

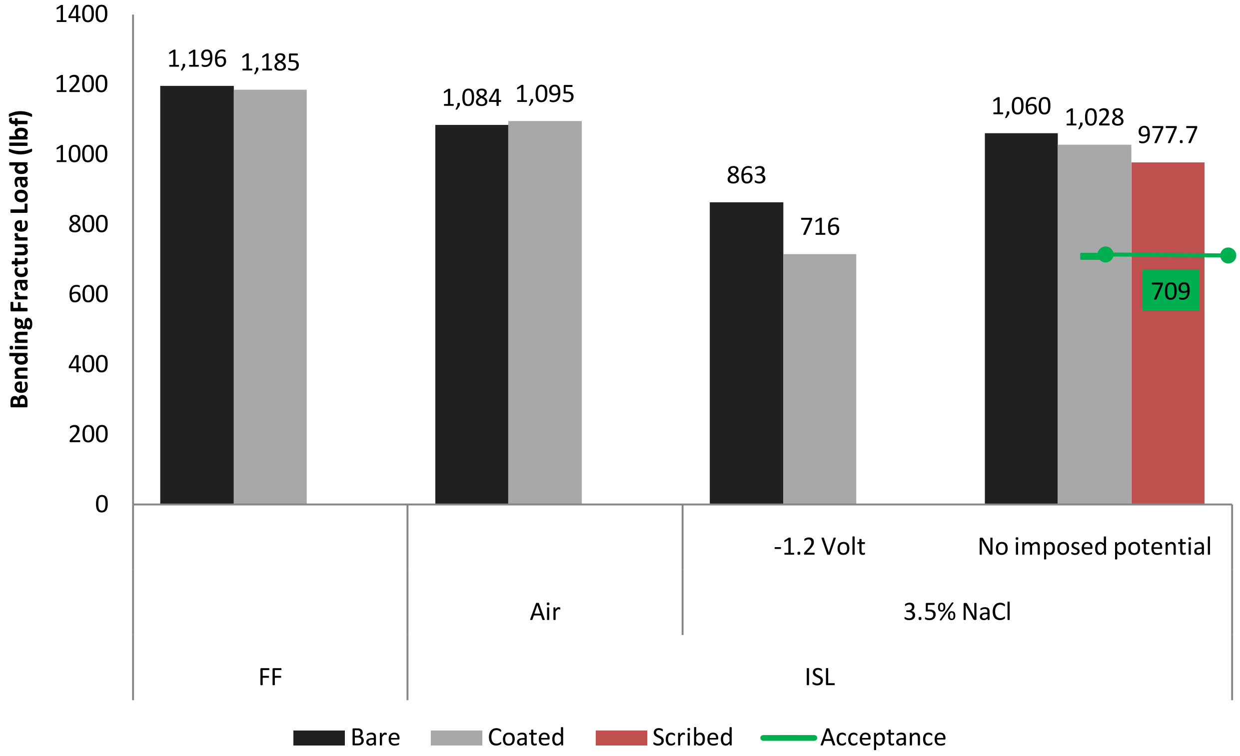

Figure 23 – 3/4-10 average threshold load values for all conditions tested

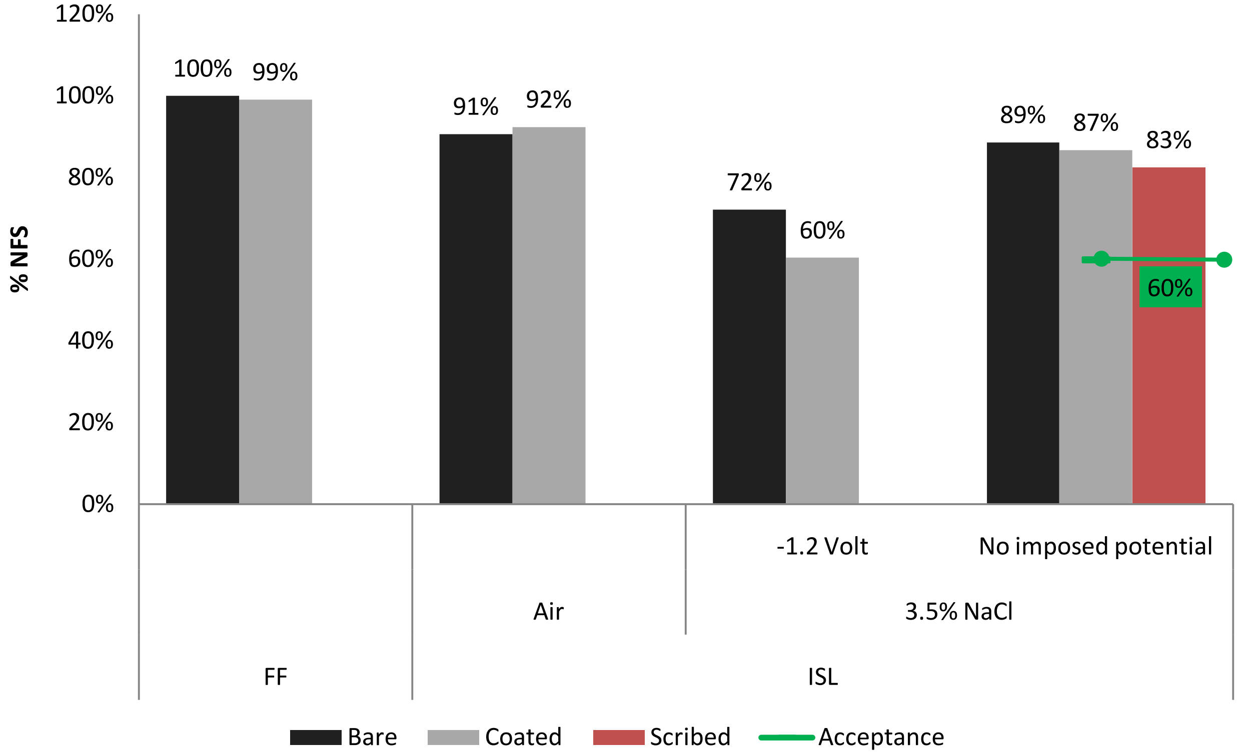

Figure 24 – 3/4-10 average threshold NFS% values (using FF Coated AIR as baseline strength to calculate NFS ratio)

6. SYNOPSIS AND CONCLUSIONS

The Magni 565 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 specimen bolts tightened by the turn on the nut method constituted a qualitative test that demonstrated that Magni 565 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 in accordance with ASTM F1940 demonstrated that the risk of IHE is nil. Product environmental testing of super high strength specimen bolts, performed in accordance with ASTM F1624, exceeded the acceptance criteria thresholds established in ATSM WK13659. These results demonstrated that Magni 565 does not promote environmental hydrogen embrittlement (EHE) on standard ASTM A490 high strength structural bolts, regardless of size.

Based on the findings of this study, which are consistent with previous findings related to Dacromet[14], it is strongly recommended that ASTM Committee F16 on Fasteners approve the use of ASTM F2833 on A490 high strength structural bolts.

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[14] Presented to ASTM Committee F16 in 2007 as ***IBECA Technologies Research Report 06-02***. A copy may be obtained from the ASTM Committee F16 homepage at www.astm.org

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

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 Draft Standard WK13659 *Standard Test Method to Qualify Coatings for Use on A490 Structural Bolts Relative to Hydrogen Assisted Failure*.

GM

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OTHERS

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APPENDICES

Appendix A: Salt Spray Images

See Attached PDF File