Research Report
TIR 10-119

QUALIFICATION OF ASTM F3019/F3019M COATINGS DELTA PROTEKT® KL 105 FOR USE ON 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 F3019/F3019M 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 F3019/F3019M, "***Standard Specification for Chromium Free Zinc-Flake Composite, with or without integral Lubricant, Corrosion Protective Coatings for Fasteners***" was adopted by Committee F16 on Fasteners, as a standard specification that is applicable to Delta Protekt® KL 105 Zinc Flake coating licensed by the Doerken Corporation headquartered in Grass Lake, MI. This work follows previous work and approvals of ASTM F1136/F1136M and ASTM F2833 coatings on A490 bolts in accordance with IFI-144 "***Test Evaluation Procedures for Coating Qualification Intended for Use on High-Strength Structural Bolts.***" This work adopts the same methodology and is being submitted to Committee F16 for approval of F3019/F3019M for use on A490 bolts.

The Delta Protekt® KL 105 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 was performed on *specimen bolts* produced strictly for this study at hardness exceeding the maximum limit for A490 (i.e., >38 HRC). The environmental testing was performed in accordance with ASTM F2660, and the results showed that the coating satisfied the acceptance criteria. In sum. the results demonstrated that Delta Protekt® KL 105 will not promote environmental hydrogen embrittlement (EHE) on standard A490 high strength structural bolts.

Based on the findings of this study, it is recommended that ASTM Committee F16 on Fasteners approve the use of ASTM F3019/F3019M (Delta Protekt® KL 105) on A490 high strength structural bolts.

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1. OBJECTIVE

The objective of this investigation is to qualify ASTM F3019/F3019M 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 F3019/F3019M, "***Standard Specification for Chromium Free Zinc-Flake Composite, with or without integral Lubricant, Corrosion Protective Coatings for Fasteners***" was adopted by Committee F16 on Fasteners, as a standard specification that is applicable to coatings licensed by Doerken Corporation headquartered in Grass Lake, MI, notably Delta Protekt® KL 120 and Delta Protekt® KL 105 Zinc Flake coatings. This study was performed on a coating system that is comprised of a zinc-flake basecoat containing integral lubricant, Delta Protekt® KL 105, and an inorganic topcoat, Delta-Protekt® VH 301.1 GZ.

Following decades during which coatings were not permitted on A490 bolts, an initiative to approve Zn-Al flake coatings resulted in approval of coatings per ASTM F1136/F1136M (2007 (chromium containing) [1,5], 2014 (non-chromium) [3,4]) and ASTM F2833 (2012) [2]. These approvals were explicitly included in ASTM A490 and now in the new High Strength Structural Bolt specification ASTM F3125. Although the three specifications, F1136/F1136M, F2833 and F3019/F3019M cover similar Zn/Al dispersion coatings, each specification is in fact adapted specifically to competing proprietary commercial coating systems. Consequently, they are treated as different coatings for qualification on A490 bolts, which is the reason why separate studies and approvals are required for each coating system.

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 (IHE) 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

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

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 for use on A490 bolts.

2. COATED SAMPLES

For 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 Delta Protekt® KL 105 process, in accordance with ASTM F3019/F3019M. 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 <u>*specimen*</u> bolts especially heat treated to achieve strength and hardness values that are slightly greater than the upper limit for A490 bolts (i.e., > 38 HRC). 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 test pieces are shown in Figure 1.

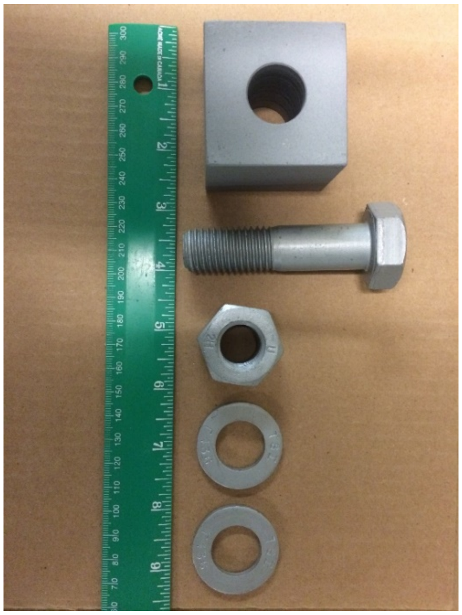

Figure 1 – Coated specimen bolt 3/4-10 x 3 with matching nut, washers and test block

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 specimen 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 4. 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. In either case, the hardness of the specimen bolts 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. Based on the measured hardness and tensile strength values, the specimen bolts significantly exceed the worst-case scenario in terms of susceptibility to environmental hydrogen embrittlement. Consequently, if they are found to pass the acceptance criteria for EHE, all A490 bolts would also pass the same acceptance criteria.

Finally, wedge tensile strength values for the specimen bolts were not statistically altered after being coated (Tables 3-4). These results confirmed that the Delta Protekt® KL 105 coating process did not alter the mechanical properties of the specimen bolts.

Table 2 – Specimen bolts hardness values (recorded after manufacturing)

	1/2-13 x 3		3/4-10 x 3	
Sample	Mid-radius (HRC)	Surface (HR 30N)	Mid-radius (HRC)	Surface (HR 30N)
1	40.4	58.8	41.0	61.0
2	39.9	60.5	40.8	60.5
3	40.5	58.9	40.7	60.9
4	40.6	57.8	40.8	59.6
5	40.7	58.6	41.1	59.8
Avg.	**40.4**	**58.9**	**40.9**	**60.4**

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

1/2-13 x 3 uncoated bolts			1/2-13 x 3 coated bolts	
Sample	Load (lbf)	Stress (psi)	Load (lbf)	Stress (psi)
1	27,530	194,010	27,500	193,798
2	26,960	189,993	27,150	191,332
3	27,245	192,001	27,250	192,037
4	27,300	192,389	27,275	192,213
5	26,950	189,922	26,950	189,922
Avg.	**27,197**	**191,663**	**27,225**	**191,860**

Tensile Stress Area 0.1419 in^2

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

3/4-10 x 3 uncoated bolts			3/4-10 x 3 coated bolts	
Sample	Load (lbf)	Stress (psi)	Load (lbf)	Stress (psi)
1	61,530	184,222	60,950	182,485
2	59,660	178,623	61,500	184,132
3	60,530	181,228	60,575	181,362
4	61,390	183,802	60,590	181,407
5	61,590	184,401	61,250	183,383
Avg.	**60,940**	**182,455**	**60,973**	**182,554**

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. The dimensions of the notch bar specimens are shown in Figure 2. The chemical composition of the lot of notch bars used in this study is given in Table 5. The average measured hardness of the lot of notch bars used in this study was 51.4 HRC.

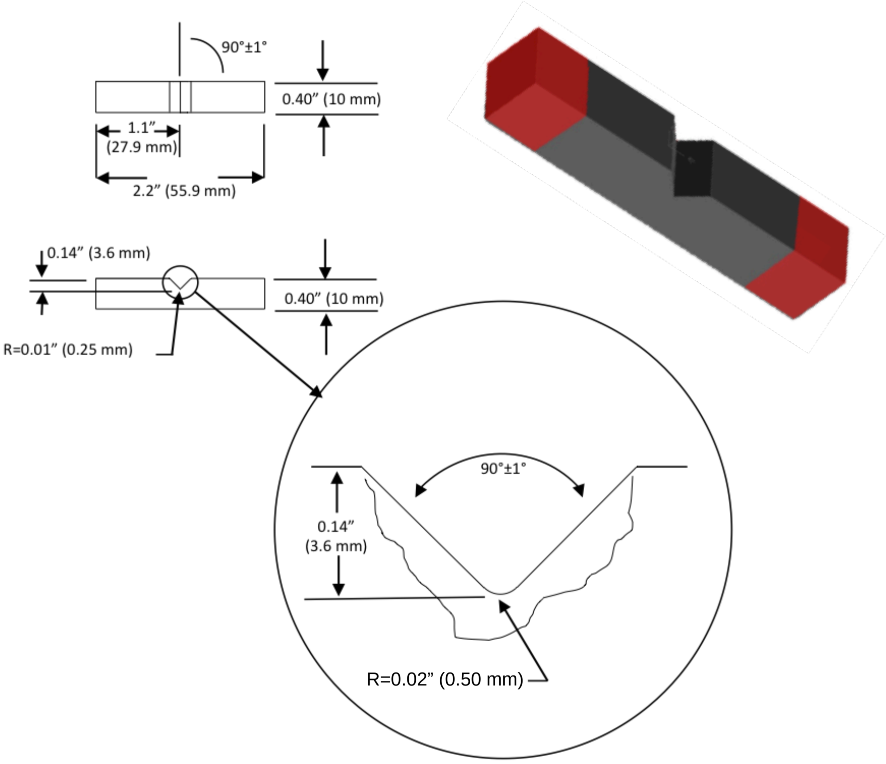

Figure 2 – ASTM F1940 notch bar specimen

Table 5 – 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. DELTA PROTEKT® KL 105 PROCESS

The Delta Protekt® KL 105 and Delta-Protekt® VH 301.1 GZ is a proprietary coating system licensed by Doerken Corporation headquartered in Grass Lake, MI. This coating system is comprised of a zinc-flake basecoat containing integral lubricant, Delta Protekt® KL 105, and an inorganic topcoat, Delta-Protekt® VH 301.1 GZ. The Delta Protekt® KL 105 zinc-flake basecoat provides cathodic corrosion protection of the underlying steel substrate through sacrificial loss of zinc. The Delta-Protekt® VH 301.1 GZ topcoat enhances the corrosion protection of the basecoat and contains lubricant to ensure consistent friction values. The Delta Protekt® KL 105 and Delta-Protekt® VH 301.1 GZ coating system is applied via a non-electrolytic process and has a typical coating thickness in the range of 7 to 15 microns.

The specimen bolts and ASTM F1940 notch bar specimens used in this study were coated at Elm Plating Company, which is located in Jackson, MI. The coating process was performed under normal operating conditions in accordance with the technical requirements established by the manufacturer as well as ASTM F3019/F3019M *Grade 4, L2*.

Surface preparation consisted of hot alkaline degreasing followed by mechanical descaling (i.e. shot blasting). Figure 3a illustrates the process flowchart for surface preparation.

The Delta Protekt® KL 105 and Delta-Protekt® VH 301.1 GZ coating system was applied to the parts via a dip-spin coating process. In this process, parts are loaded into a coating basket or barrel, immersed in the coating solution for a set duration, removed from the coating solution and subsequently centrifugated or tumbled to remove excess material. In addition to dip-spin processing, other application methods are available. These include application via spray (with or without electorstatics), dip-drain, and rack dip-spin. The method of application is generally dependent upon factors such as the size and geometry of the part to be coated.

After application of the Delta Protekt® KL 105 basecoat, parts were thermally cured for 20 minutes at approximately 230 °C part metal temperature. When the Delta-Protekt® VH 301.1 GZ topcoat was applied, the parts were thermally cured for 20 minutes at approximately 60 °C part metal temperature. Figure 3b illustrates the process flowchart for the Delta Protekt® KL 105 and Delta-Protekt® VH 301.1 GZ coating system.

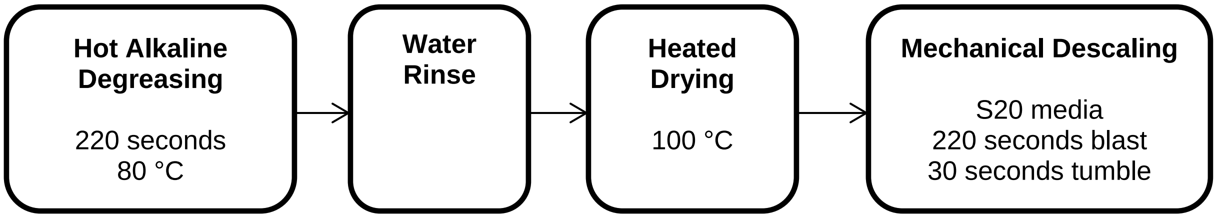

Figure 3a – Surface Preparation process flowchart

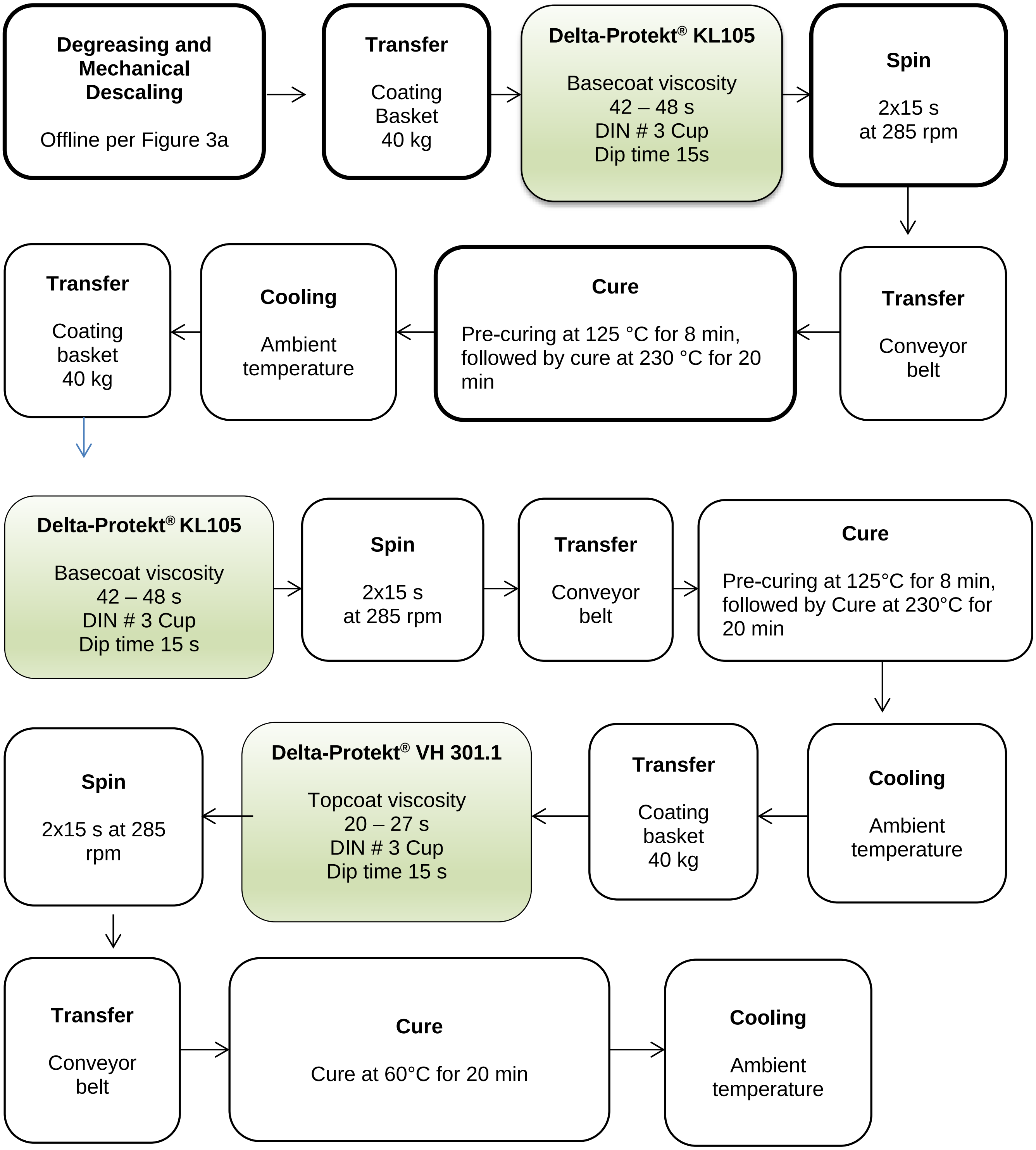

Figure 3b – Delta-Protekt® KL 105 and Delta-Protekt® VH 301.1 process flowchart

4. EXPERIMENTAL METHODS

Table 6 shown below 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 5).

Table 6 – IFI-144 qualification test methods

Sect.	Test	Specification	Condition
2	Hardness	ASTM F606	Specimens
2	Chemical Analysis	ASTM A751	Specimens
5.1	Microstructure	ASTM E3	Specimens
5.2	Coating Thickness	ASTM D1186	Coated Specimens
5.3	Adhesion	ASTM B571	Coated Specimens
5.4	Paintability	Visual	Coated Specimens
5.5	Rotational Capacity	FHWA/AASHTO	Standard A490 bolts
5.6	Salt Spray Exposure	ASTM B117 / ASTM D1654	Coated Specimens
5.7	Cyclic Exposure (conducted on bolts assembled in test blocks)	GM9540P (superseded by GMW 14872)	Coated Specimens
5.8	Tensile Pull	ASTM F606	Coated Specimens
5.9	Hydrogen Embrittlement (process)	ASTM F1940	F1940 notch square bars
5.10	Hydrogen Embrittlement (product)	ASTM F2660	Coated 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 4 and 5 respectively.

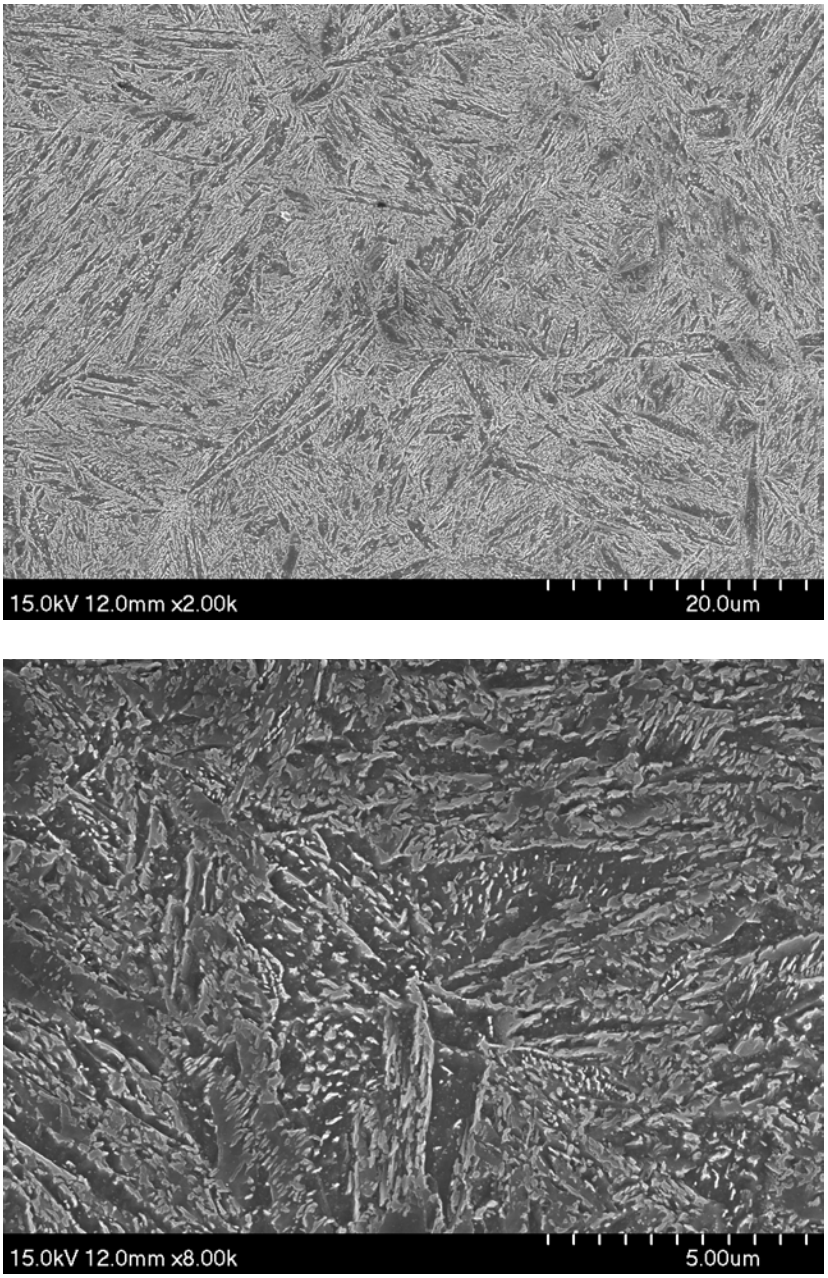

Figure 4 – Microstructure 1/2-13 x 3 specimen bolts (2,000 X and 8,000 X)

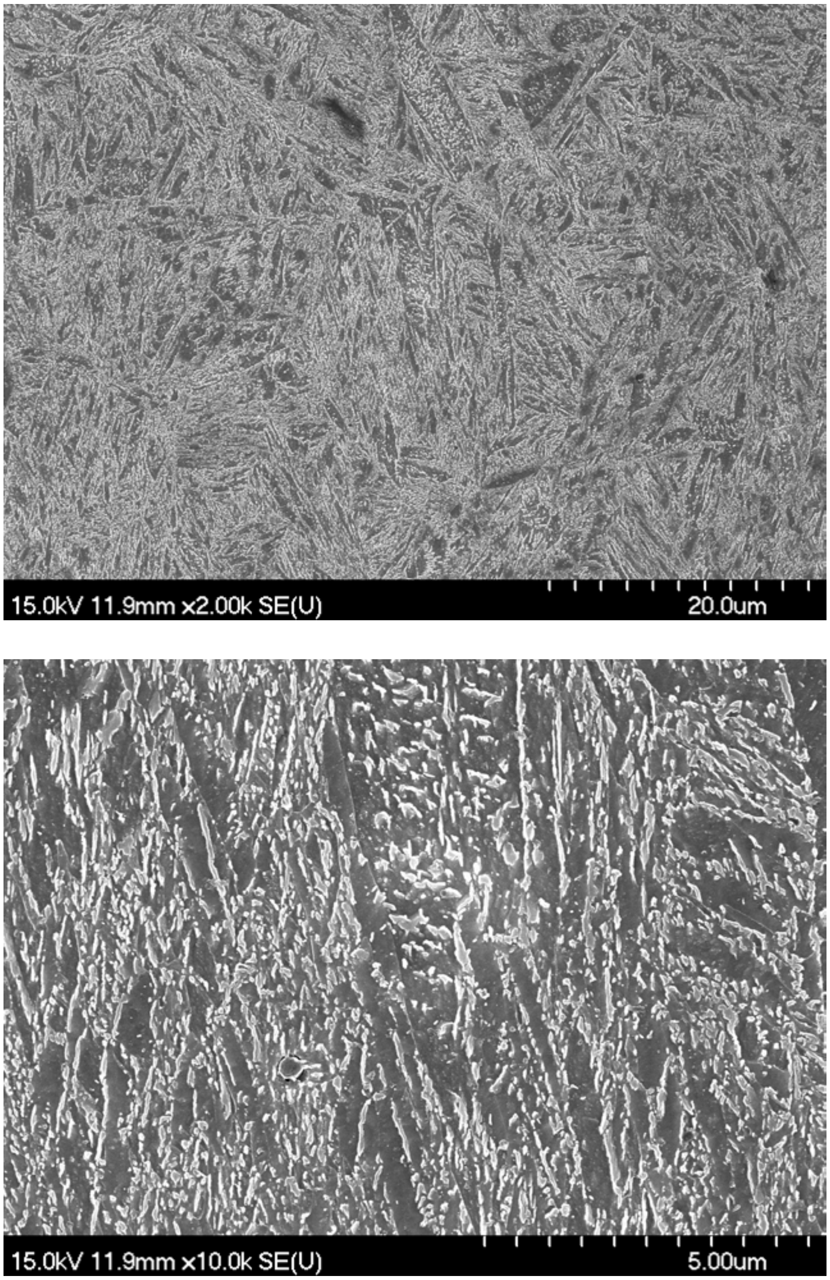

Figure 5 – Microstructure 3/4-10 x 3 specimen bolts (2,000 X and 10,000 X)

5.2 Coating thickness

Coating thickness was measured in accordance with ASTM D1186 using magnetic induction tester. The sampling size consisted of ten bolts, nuts and washers of each size. Surfaces measured were the top of the head and each hexagonal face. A minimum of 10 individual measurements were done per surface. For all parts, the average thickness was in the range of 0.51 mil (13 microns). Summary results are provided in Figure 6.

ASTM F3019/F3019M does not specify minimum coating thickness, only minimum neutral salt spray test requirements. 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. Recommended oversize allowances for nuts used with A490 bolts coated by Zn/Al dispersion coatings are given in ASTM F3125/F3125M, Table A1.1.

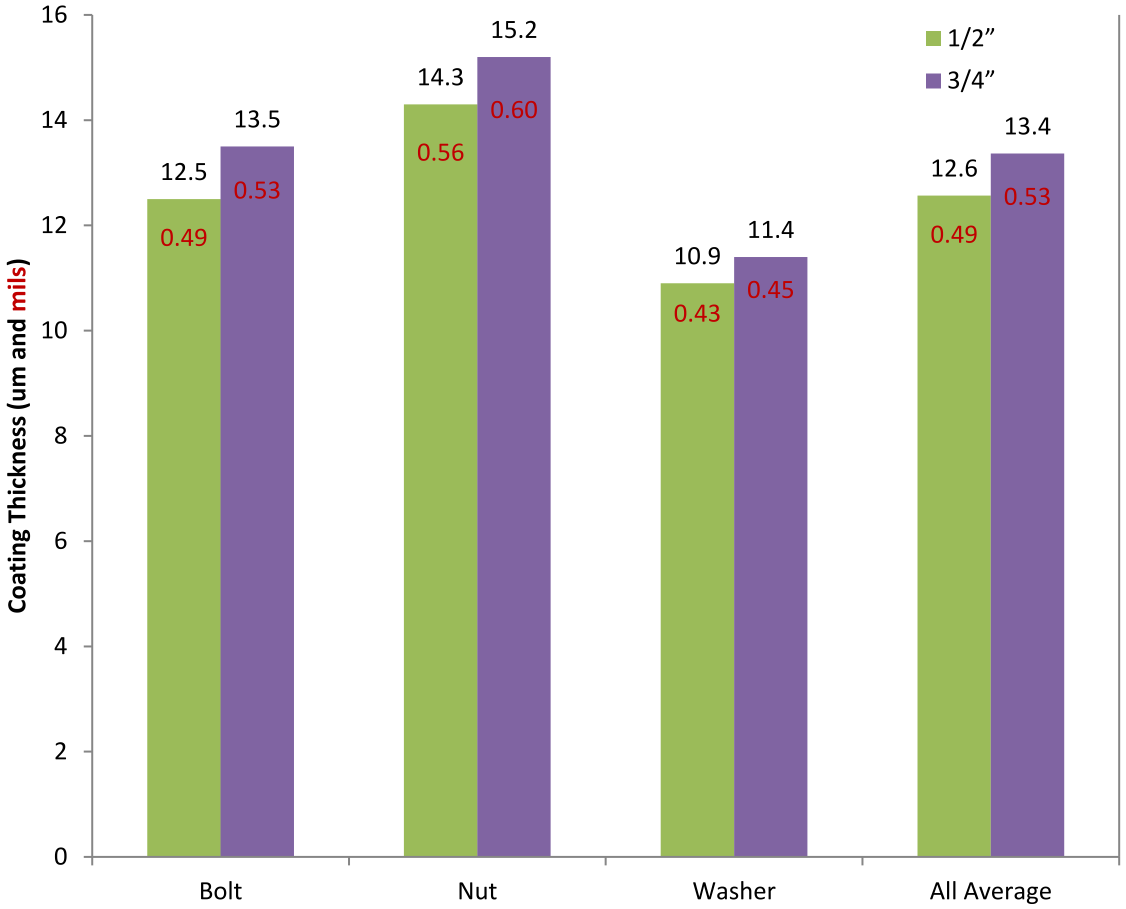

Figure 6 – Average coating thickness values – bolts, nuts, washers

5.3 Adhesion

IFI-144 stipulates adhesion testing in accordance with ASTM B571. Three coated bolts of each size (1/2 and 3/4 inch) were scribed in 3 parallel lines with sufficient pressure to penetrate the coating to the steel substrate, followed by application of 3M 610 clear tape with firm finger pressure and rapid removal at an angle of 180°. The *specified* bond strength of 3M 610 tape is 47 N/100mm width. No continuous portion of the coating between the parallel lines broke away from the substrate, indicating satisfactory adhesion (Figs. 7-8).

Figure 7 – 3M 610 tape samples showing satisfactory adhesion on 1/2-13 x 3 bolts

Figure 8 – 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. After 48 hours at room temperature, the coating was verified to be dry to the touch, fulfilling the requirement (Fig. 9).

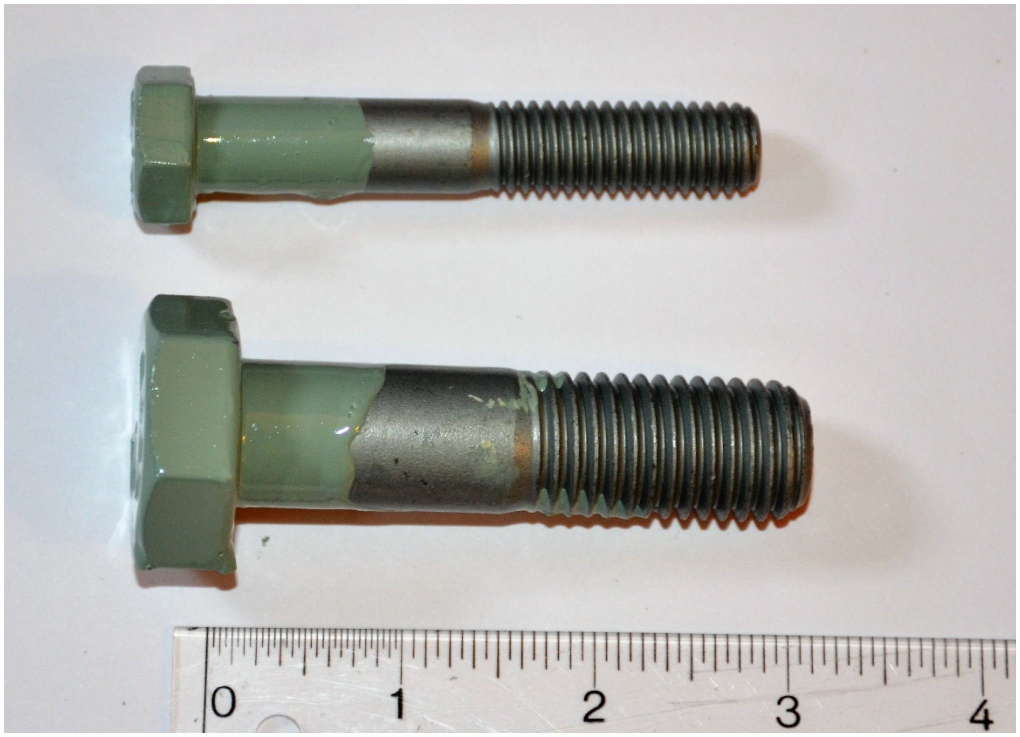

Figure 9 – Primed and painted bolt heads were dry to the touch after 24 h curing at room temperature, thus satisfying the 48 h requirement in IFI-144

5.5 Rotational capacity

IFI-144 stipulates that the coated bolts must pass the rotational capacity (RC) test. Torque tension testing was performed on ***standard A490 bolts*** coated with Delta Protekt® KL 105 to verify if rotational capacity (RC) test requirements as stipulated in ASTM F3125/F3125M would be met. the RC test requirements in ASTM F3125/F3125M also satisfy requirements by The American Association of State Highway and Transportation Officials (AASHTO) and by the Federal Highway Administration (FHWA).

Note 1: Standard A490 bolts were tested for RC testing because specimens have greater strength properties than what is allowed for A490 bolts, and would therefore have provided misleading torque/tension data. The sizes of standard A490 bolts used for the RC testing are 3/4-10 x 5 and 1/2-13 x 3. This is the only portion of the study that did not utilize the specimen bolts.

The results show the coated parts comfortably passed the RC test criteria, even if no allowance was made for coating thickness between the mating bolts and nuts. These results are consistent with previous results obtained for DACROMET, GEOMET and MAGNI 565 [1-5]. The results for both test sizes are shown in Figures 10 and 11 respectively. For each size, the data are presented in two charts: (i) angle vs. tension, and (ii) torque vs. tension.

A closer look at measured nut factor (K) values at installation tension illustrates the friction reducing effect of Delta Protekt® KL 105 (bolts-washers). The K value range at installation tension was 0.08 - 0.110 for both sizes. 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.

The test parameters for *pre-installation verification* and for *rotational capacity* testing are given in Tables 7-9 shown below, based on the length (L) to diameter (d) ratios for the standard A490 bolts.

Table 7 – length over diameter ratios for Standard A490 bolts

Bolt diameter d (in)	Length L (in)	L/d
0.50	3	6
0.75	5	6.7

Table 8 – Pre-installation verification testing and acceptance criteria

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

Bolt Length (L)	Nut rotation from snug tension
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 9 – Rotational capacity testing and acceptance criteria

Bolt size (in)	Snug tension[5] (kips)	Installation tension[6] (kips)	Max. torque at installation tension[7] (ft-lb)	Min. final tension[8] (kips)
1/2-13	1	15	156	17
3/4-10	4	35	546	40

Bolt length (L)	Nut rotation from snug tension
L ≤ 4d (not more than 4d)	240˚ (2/3 turn)
4d < L ≤ 8d (more than 4d but not more than 8d)	300˚ (1 turn)
8d < L ≤ 12d (more than 8d but not more than 12d)	360˚ (1 turn)

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

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

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

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

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

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

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

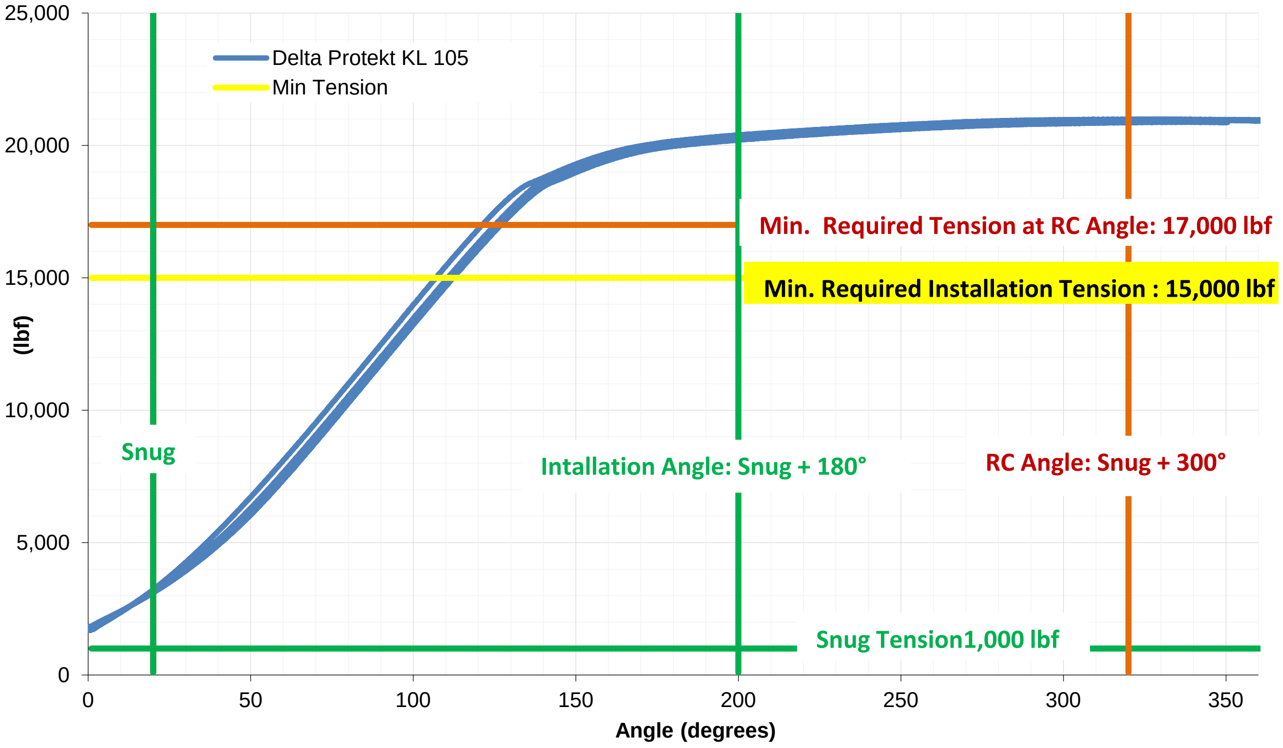


Figure 10a – RC test data for 1/2 inch parts: (i) angle vs. tension

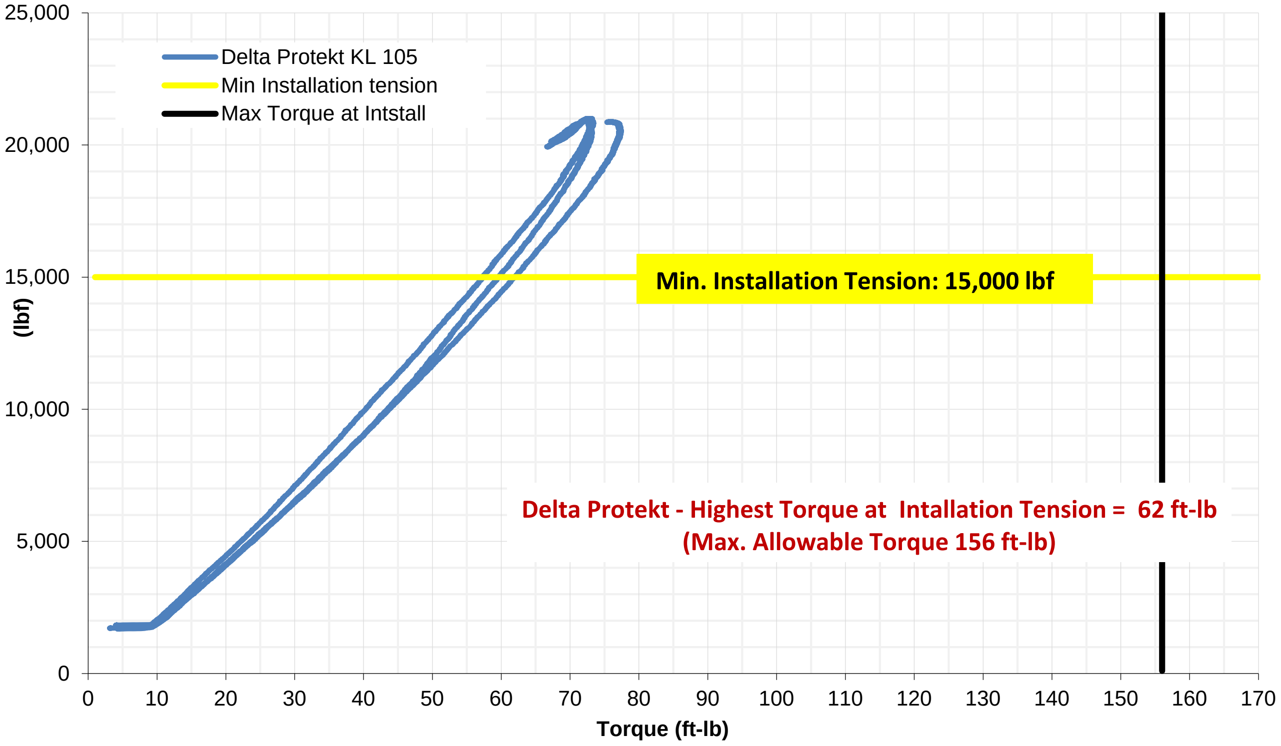


Figure 10b – RC test data for 1/2 inch parts: (ii) torque vs. tension

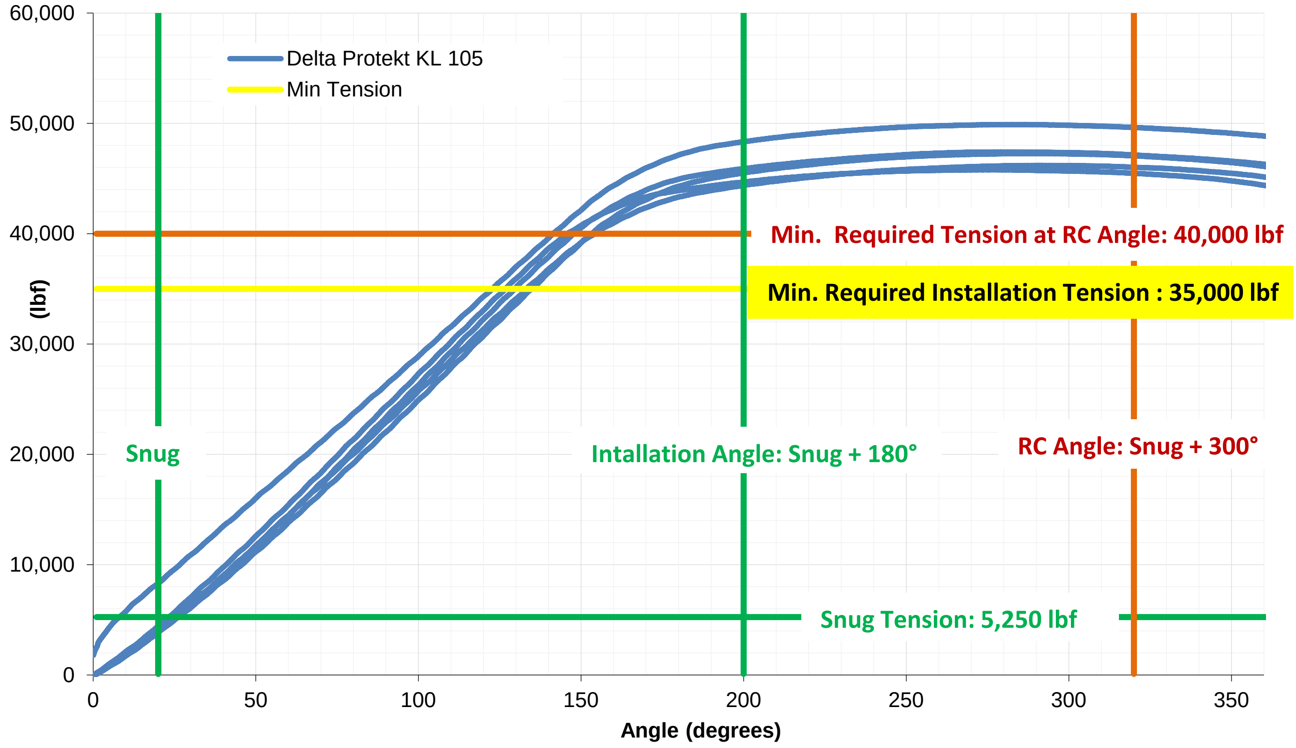


Figure 11a – RC test data for 3/4 inch parts: (i) angle vs. tension

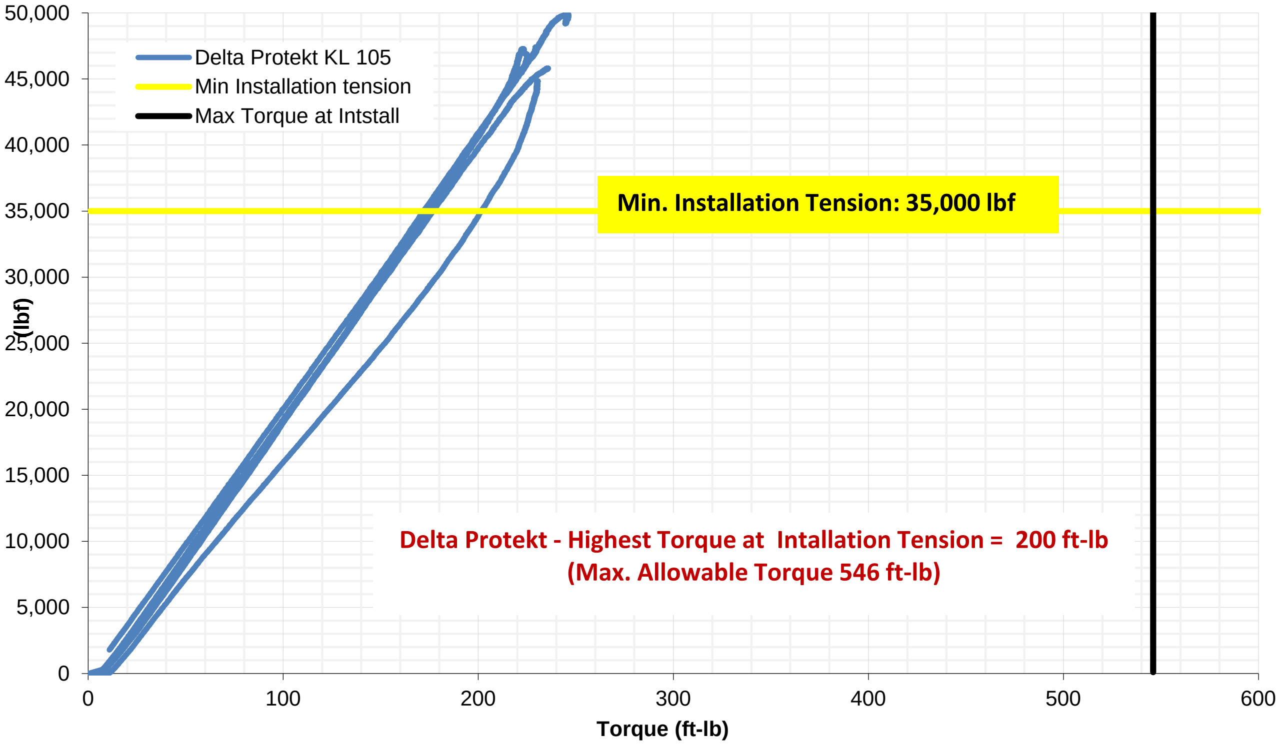


Figure 11b – RC test data for 3/4 inch parts: (ii) torque vs. tension

<u>Commentary</u>:

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. Simply stated, the basic concept for the test was to subject the bolts to a nut tightening rotation that is two times (2X) that which is needed for normal assembly. Some exceptions were later made to reduce the severity of the nut rotation for certain length/diameter ratios. If the bolt can be readily disassembled without any sign of damage or failure, then it passed the test. Later. 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 even though metallic coatings were not permitted on A490 bolts at the time. 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 been problematic from the outset, because A490 bolts do not exhibit the same ductility and elongation as A325 bolts. Consequently, ASTM Committee F16 on Fasteners, decided to introduce a comprehensive revision of the RC test procedure in the new combined structural bolt standard ASTM F3125/F3125M. The new procedure lowers the rotation requirements for A490 bolts. The revised procedure has been accepted by the Research Council on Structural Connections (RCSC) and the Federal Highway Administration (FHWA). As of this writing, RC test procedures and applicability continue to be subject of study and revision by ASTM Committee F16 on Fasteners.

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 3/4 inch specimen bolts were tested. The parts were examined after 504, 840, 1008, 1512, 2016, 3000 and 5000 hours of exposure. parts were rinsed and allowed to dry to be evaluated for red rust, and photographed. No red rust was observed on significant surfaces of any samples, even after 5000 hours of exposure (Figures 12 a-b).

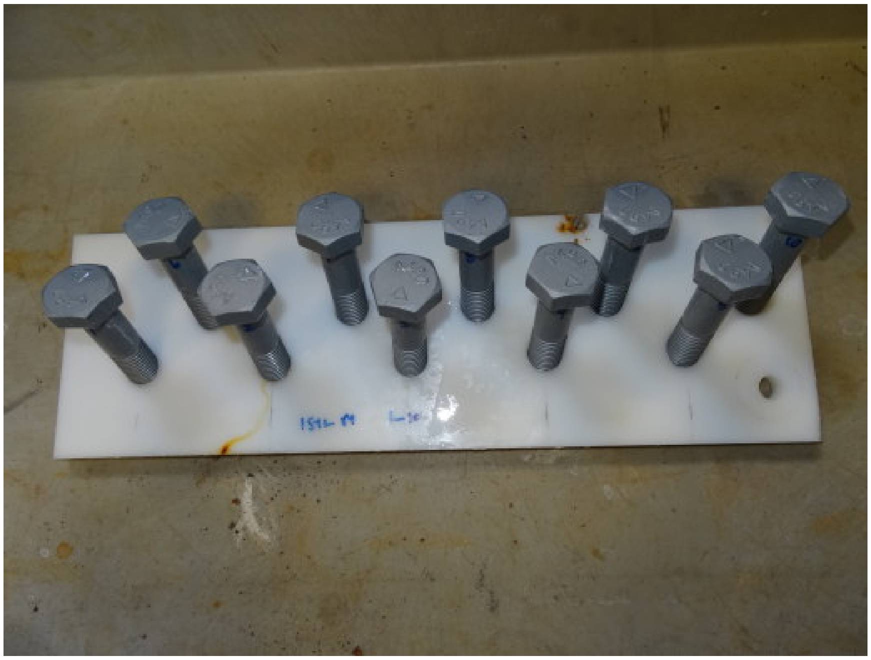


Figure 12a – 3/4 inch specimen bolts after 1000 h of salt spray exposure

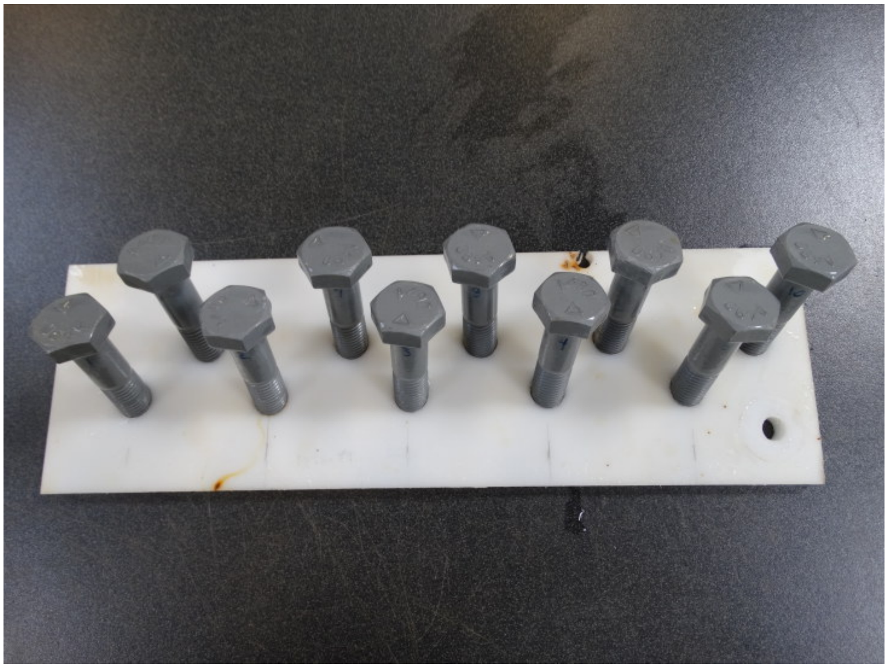


Figure 12b – 3/4 inch specimen bolts after 5000 h of salt spray exposure

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 to evaluate the potential for stress corrosion cracking (SCC) resulting from galvanic corrosion generated hydrogen. The cyclic corrosion testing was performed in accordance with GMW 14872, which unlike continuous salt spray, may be used to predict corrosion service life for automobiles in given conditions. Note, conditions prescribed in GMW 14872 (current) are more aggressive than those prescribed in GM 9540P (superseded). 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. Ten (10) coated 3/4 inch specimen bolts were tested, of which five (5) were finger tightened and (5) were turn-of nut tightened into yield. One bare (uncoated) bolt was used as a control sample. Sampling conditions are given in Table 10. Figure 13 shows the test components used for cyclic testing, each assembly comprising one bolt, one nut, two washers, all assembled in the test block. The test fixtures consisted of hardened rectangular steel blocks. The fixtures were coated with the same coating as the bolts to avoid any galvanic corrosion effects.

Table 10 – Cyclic test conditions on 3/4 -10 x 3 specimen bolts

Coating	Tightening Condition	Exposure	Sample Size
Bare	Finger tightened	120 Cycles	1
Delta Protekt® KL 105	Finger tightened	120 Cycles	5
Delta Protekt® KL 105	Turn-of-nut ½ turn	120 Cycles	5

Figure 13 – test components used for cyclic testing, each assembly comprising one bolt, one nut, two (2) washers, all assembled in the test block

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 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 1/3 turn + 30° (150° total) beyond snug tension for the 3/4-10 x 3 inch specimen bolts, as shown in Figure 14.

Figure 14 – illustration showing markings for rotation of nuts from snug tight starting position (1) to 1/3 turn + 30° (150° total) beyond snug (2).

The test pieces were exposed for a total of 120 cycles and evaluated for visual corrosion and weight loss. Periodic verification was performed during the exposure period, where the parts were rinsed with warm water and visually examined. None of the bolts showed any signs of cracking or failure. In addition, there were no indications of red rust on any of the coated bolts after 80 cycles, and even after 120 cycles, as shown in the summary results (Table 11) and photos shown in Figures 15-17. Minimal red rust was observed on the corners of the test blocks only. The bare bolt was completely corroded well before reaching 20 cycles. The corrosion weight loss of exposed parts was measured by weighing the parts prior to and following exposure. The results showed that Delta Protekt® KL 105 coated parts did not experience any significant weight loss. Similar to previous findings with DACROMET [1], GEOMET [3] and Magni 565 [2] coatings, a slight weight gain was observed which can be attributed to the presence of oxides and residues. The results of weight loss measurement are given in Table 12.

Table 11 – Visual estimation of red rust on 3/4 -10 x 3 specimen bolts during cyclic testing

	Turn-of-Nut Tightened Red rust on head (rating scale: 0-10)		
	40	**80**	**120**
Delta Protekt® KL 105	0	0	0
	0	0	0
	0	0	0
	0	0	0
	0	0	0
	No Load Red rust on head (rating scale: 0-10)		
	40	**80**	**120**
Delta Protekt® KL 105	0	0	0
	0	0	0
	0	0	0
	0	0	0
	0	0	0
Control (Bare)	10	10	10

Figure 15 – Bare sample after 20 cycles

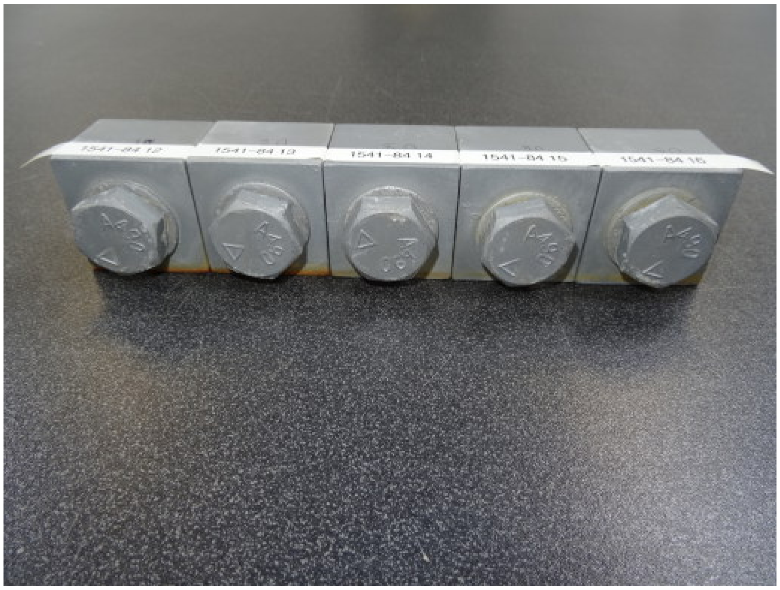


Figure 16 – Delta Protekt® KL 105 coated - Finger tightened - after 120 cycles

Figure 17 – Delta Protekt® KL 105 coated - Turn -of nut tightened - after 120 cycles

Table 12 – Weight change (g) following 120 cycles of exposure

Turn of the Nut				
Sample ID	Weight (g) - Start	Weight (g) - Final	Change (g)	Change %
Ton 1	1065.773	1065.910	0.137	0.013%
Ton 2	1066.390	1066.563	0.173	0.016%
Ton 3	1069.308	1069.583	0.275	0.026%
Ton 4	1064.339	1064.432	0.093	0.009%
Ton 5	1066.929	1067.072	0.143	0.013%
Finger Tightened				
Sample ID	Weight (g) - Start	Weight (g) - Final	Change (g)	Change %
U1	1065.317	1065.393	0.076	0.007%
U2	1066.994	1067.080	0.086	0.008%
U3	1063.695	1063.774	0.079	0.007%
U4	1066.008	1066.199	0.191	0.018%
U5	1067.003	1067.203	0.200	0.019%
Bare				
Sample ID	Weight (g) - Start	Weight (g) - Final	Change (g)	Change %
Bare	1064.064	1035.511	-28.553	-2.683%

Corrosion coupons: 120 Cycle Mass Loss: 15.39 g

5.8 Post exposure tensile tests

IFI-144 requires that bolts be axially tensile tested after cyclic exposure to ensure that no degradation in strength has occurred due to the corrosion of the coated bolts. In this study, the 3/4-10 specimen 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 the conditions are given in Table 13. The results demonstrate that Delta Protekt® KL 105 coating did not cause any measurable loss of strength following 120 cycles of cyclic exposure.

Table 13 – Post exposure wedge tensile test results for 3/4-10 specimen bolts after 120 cycles of exposure compared to pre-exposure

	Bolts tested after 120 cycles of exposure	Pristine bolts tested after coating (ref. Section 2.1, Table 4)
Sample	Load (lbf)	Load (lbf)
1	60,350	
2	59,975	
3	60,930	
4	61,550	
5	61,850	
Avg.	**60,931 (182,428 psi)**	**60,973 (182,554 psi)**

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 Delta Protekt® KL 105 coating process. In this test, five (5) Delta Protekt® KL 105 coated notch bars are 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 hydrogen embrittlement threshold strength of the notch bar using a standardized loading protocol in air as specified in ASTM F1940. The test 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) [6]. The results given in

Figure 18 showed no reduction of NFS% with average values of 99.6%, which is consistent with previous research findings relative to Zn-0Al flake coatings [7].

$$NFS_{\%\ F1940} = \frac{ISL_{air\ F1940-P}}{ISL_{air\ F1940\ -bare}}$$

Where:
$NFS_{\%\ F1940}$ = Percent Notch Fracture Strength
$ISL_{\ air\ F1940\text{-}bare}$ = Notch fracture load of bare SQB specimen
$ISL_{\ air\ F1940\text{-}P}$ = Notch fracture load of coated SQB witness specimen

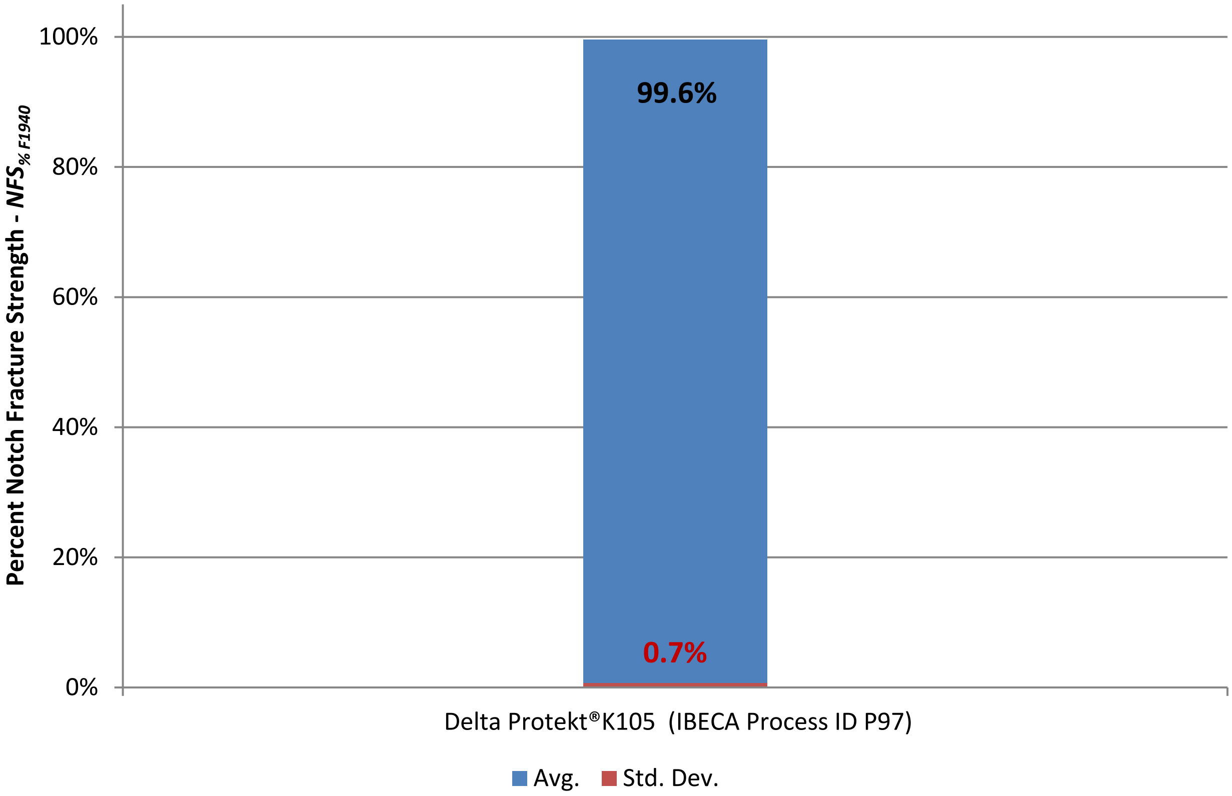


Figure 18 – NFS% values obtained for Delta Protekt® KL 105 – the results indicate no risk of Internal Hydrogen Embrittlement (IHE)

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 of Delta Protekt® KL 105 on a coated specimen bolt was measured and then compared to previous results obtained for GEOMET®, DACROMET® and Magni 565. As can be seen from figures 19 and 20, OCP value for Delta Protekt® KL 105, after 24h of stabilization was -1.013 V, which is both consistent with expectation and very similar to the previously studied coatings. The significance of this result is that Delta Protekt® KL 105 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.

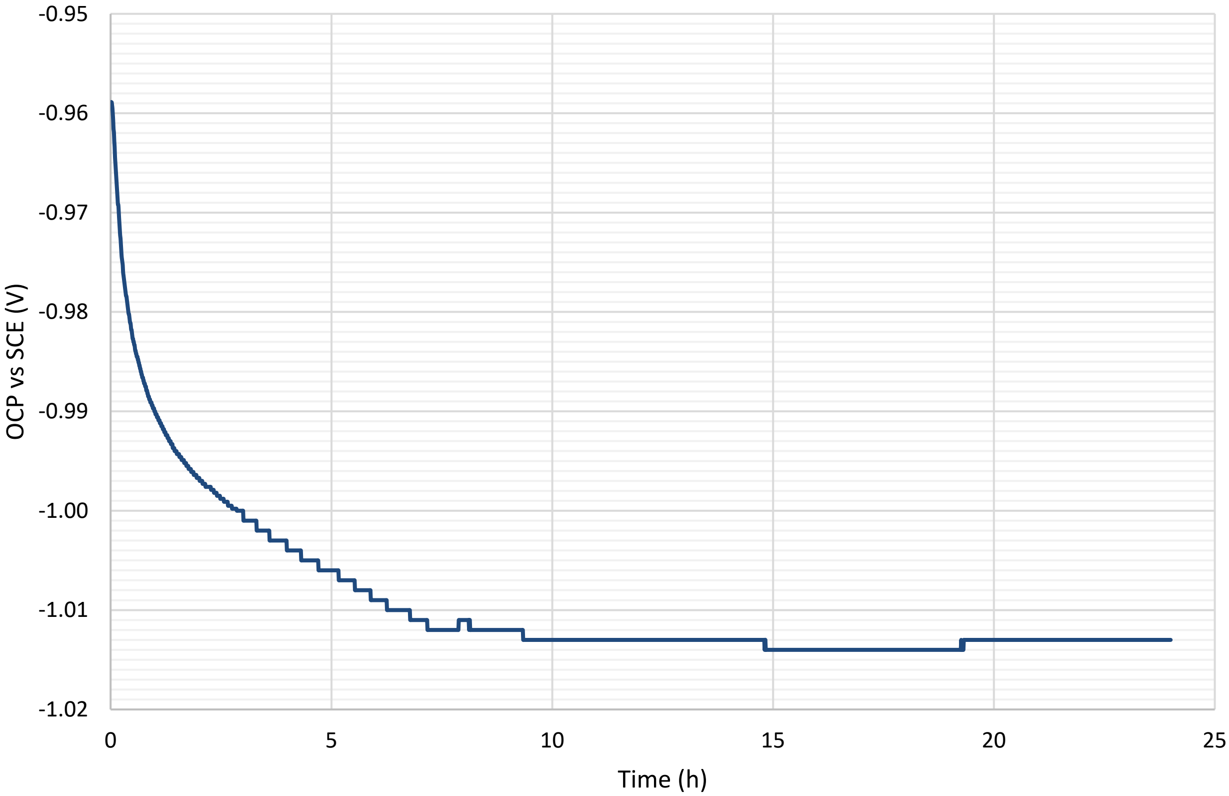
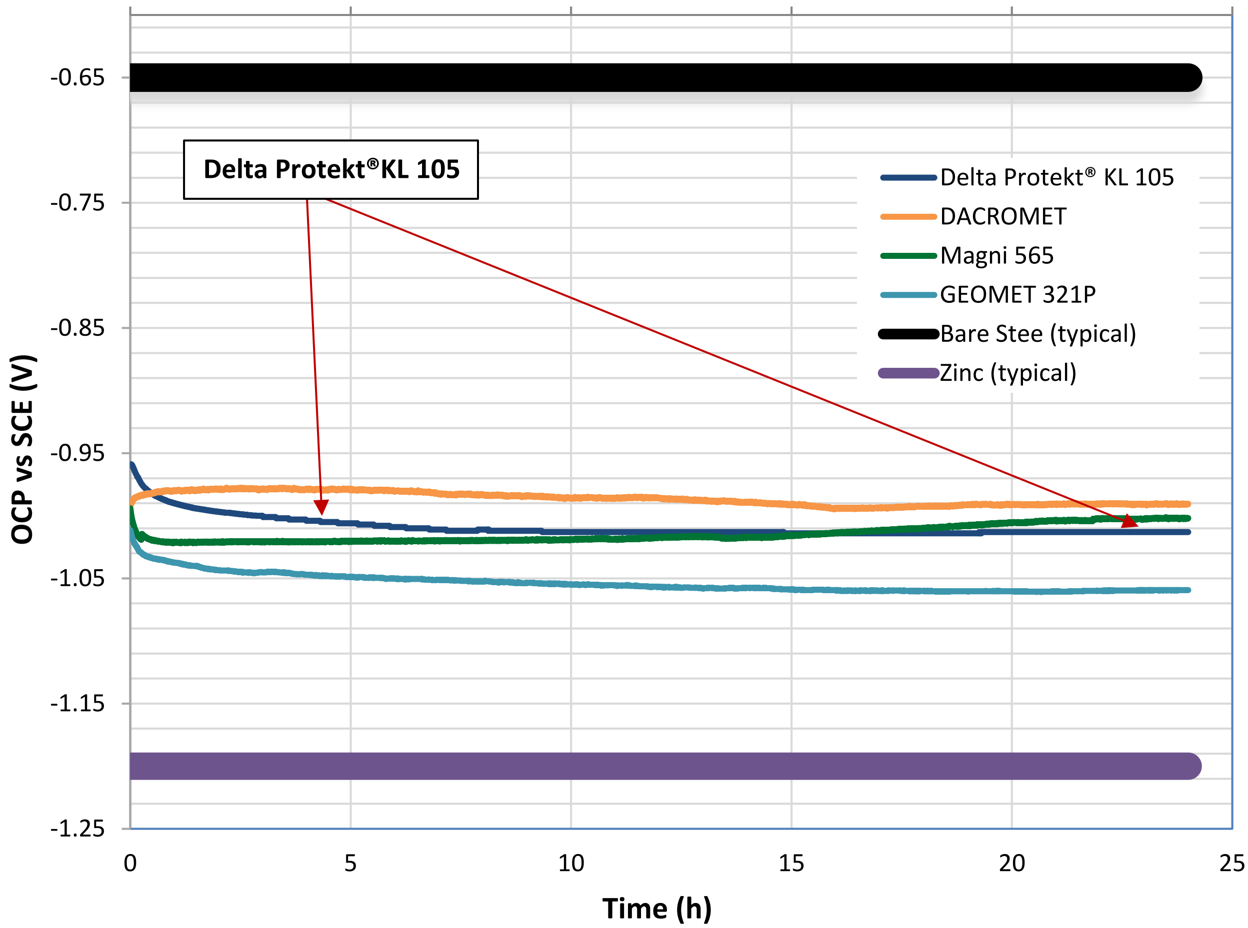


Figure 19 – OCP test progression for Delta Protekt® KL 105 over 24 h period



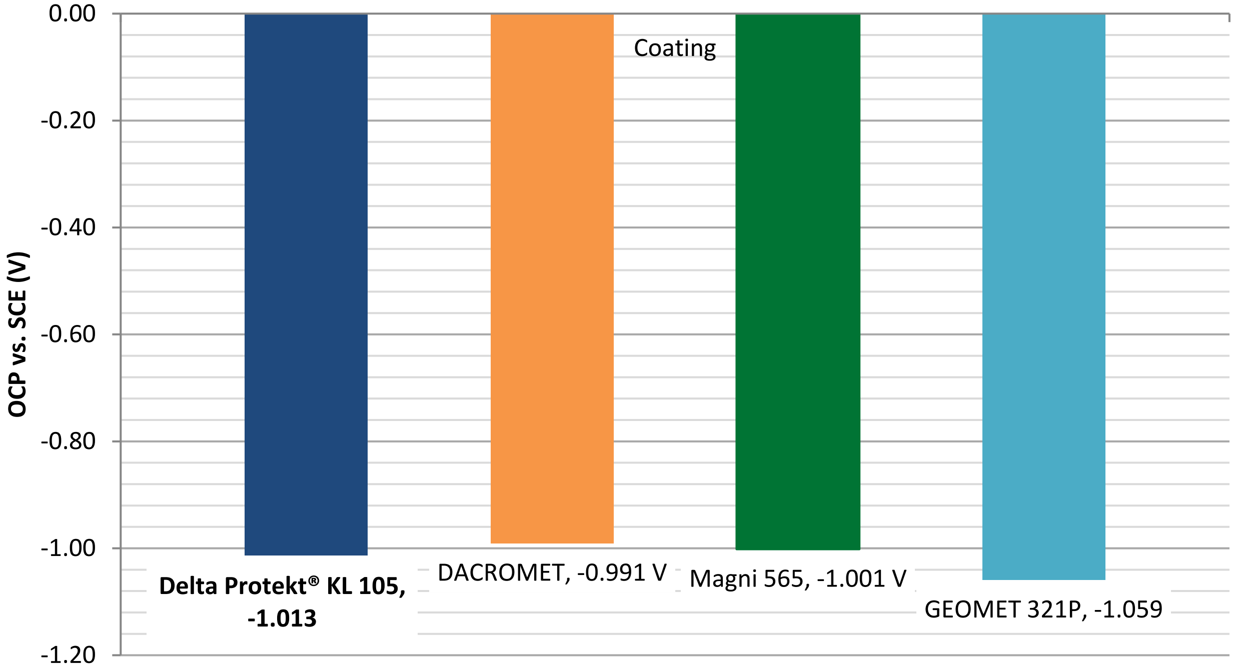


Figure 20 (a,b) – OCP test progression for Delta Protekt® KL 105 compared to previously approved coatings. (a) progression over 24 h, (b) stabilized value after 24 h

The first step in the testing sequence was a measurement of the *fast fracture* load of the specimen bolts in bending ($FFS(B)_{coated}$). The average value obtained from 5 tests was used as as baseline value. 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 and teflon tape. 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 10% 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, which exceed the maximum hardness allowed for A490 bolts, are shown in figures 21 and 22. The results show that the acceptance load level was comfortably exceeded in both cases**.** The acceptance level values which apply to the "coating holiday" or "scribed" condition (red bar) are illustrated in green on each chart are 81 and 83 % respectively for 1/2 inch and 3/4 inch bolts. These test results demonstrate the Delta Protekt® KL 105 coating satisfies the acceptance criteria for EHE on specimen bolts even though their hardness and therefore their susceptibility to HE is greater than for standard A490 bolts. Therefore, it is with categoric certainty that these test results demonstrate the Delta Protekt® KL 105 coating satisfies the acceptance criteria for EHE on A490 bolts.

Given that ASTM A490 bolts can be manufactured up to nominal size of 1-1/2 inch, ASTM F2660, Table 2 offers a calculation based approach to project equivalence values for smaller size specimen bolts. According to ASTM F2660, Table 2 For 3/4 inch specimen bolts the threshold value must equal or exceed 81.9 % of the baseline $FFS(B)_{coated}$ in order to be applicable to 1-1/2 inch bolts. From Figure 22, a threshold value of 83 % was obtained for the scribed condition. These test results demonstrate the Delta Protekt® KL 105 coating satisfies the acceptance criteria for EHE on A490 bolts of all sizes, given that the results were obtained using specimen bolts with hardness and therefore HE susceptibility greater than standard A490 bolts.

> **Note 2: for consideration of size equivalence, the 1/2-13 x 3 specimen bolts were disqualified for the following reasons. First, the large size difference between 1/2 and 1-1/2 significantly reduces the reliability of the projected threshold calculation. Second, the tensile strength of the 1/2-13 x 3 specimen bolts, nearly 192 ksi, is so much higher than the maximum allowable 173 ksi for A490 bolts that it would be incorrect to attempt to use them for in this calculation based approach.**
>
> **Note 3: the ISL tests performed in air exhibited threshold strengths at between 91 % and 95 % of baseline strength. This loss of strength, corroborated by previous studies [6,7], is attributed to the residual hydrogen that already existed in the steel prior to coating and environmental exposure.**

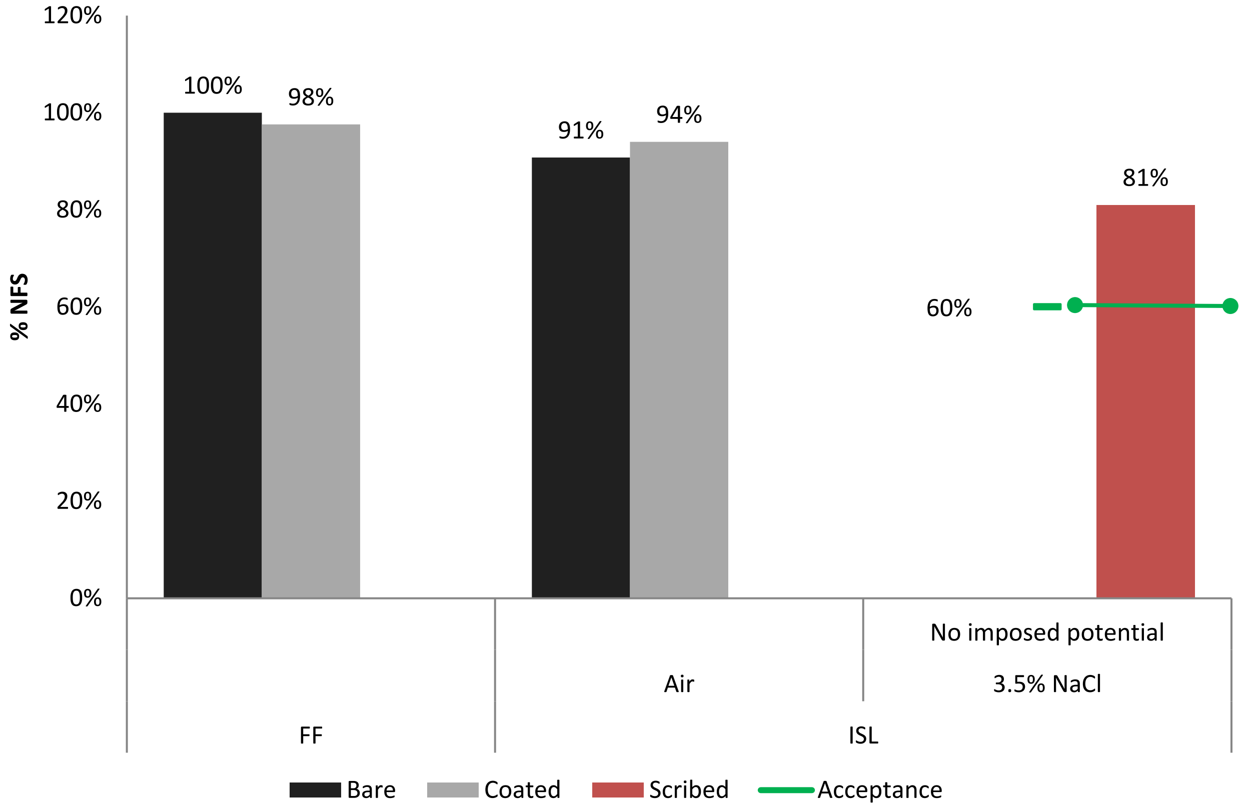


Figure 21 – 1/2-13 average threshold NFS% (using FF Coated AIR as baseline strength)

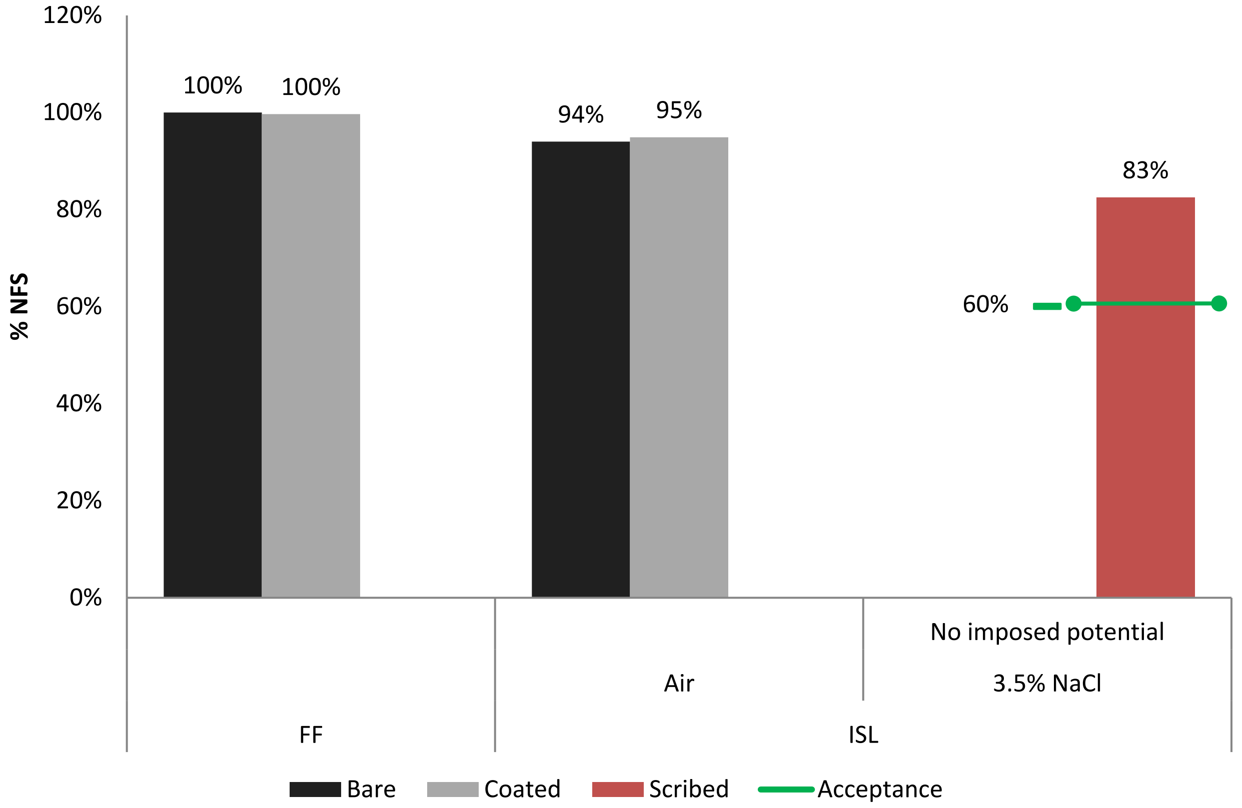


Figure 22 – 3/4-10 average threshold NFS% (using FF Coated AIR as baseline strength)

6. SYNOPSIS AND CONCLUSIONS

The Delta Protekt® KL 105 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 Delta Protekt® KL 105 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 Delta Protekt® KL 105 would not promote environmental hydrogen embrittlement (EHE) on standard ASTM A490 high strength structural bolts, regardless of size.

Based on the findings of this study performed on Delta Protekt® KL 105 (basecoat) and Delta-Protekt® VH 301.1 GZ (topcoat) coating system, it is recommended that ASTM Committee F16 on Fasteners approve the use of ASTM F3019/F3019M Grade 4 coating for use on A490 high strength structural bolts.

This report is presented to Committee F16 for review.

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IFI

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

ASTM

1. ASTM A490 *Standard Specification for Structural Bolts, Alloy Steel, Heat Treated, 150 ksi Minimum Tensile Strength*.
2. ASTM A490M *Standard Specification for High-Strength Steel Bolts, Classes 10.9 and 10.9.3, for Structural Steel Joints (Metric)*.
3. ASTM A751 *Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products*.
4. ASTM B117 *Standard Practice for Operating Salt Spray (Fog) Apparatus*.
5. ASTM B571 *Standard Practice for Qualitative Adhesion Testing of Metallic Coatings*.
6. ASTM B659 *Standard Guide for Measuring Thickness of Metallic and Inorganic Coatings*.
7. ASTM D1186 *Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base*.
8. ASTM D1654 *Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments*.
9. ASTM D3359 *Standard Test Methods for Measuring Adhesion by Tape Test*.
10. ASTM E3 *Practice of Preparation of Metallographic Specimens*.
11. ASTM E92 *Standard Test Method for Vickers Hardness of Metallic Materials*.
12. ASTM F606 *Test Methods for Determining the Mechanical Properties of Externally and Internally Threaded Fasteners, Washers, and Rivets*.
13. F1136/F1136M *Standard Specification for Zinc/Aluminum Corrosion Protective Coatings for Fasteners.*
14. ASTM *F1624 Standard Test Method for Measurement of Hydrogen Embrittlement in Steel by the incremental Loading Technique*.

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

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

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

18. ASTM F3019/F3019M *Standard Specification for Corrosion Protective Fastener Coatings with Zinc Rich Base Coat and Aluminum Organic/Inorganic Type*

19. ASTM F3125 *Standard Specification for Structural Bolts, High Strength Structural Bolts, Steel and Alloy Steel, Heat Treated, 120 ksi (830 MPa) and 150 ksi (1040 MPa) Minimum Tensile Strength, Inch and Metric Dimensions.*

GM

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