Research Report
TIR 18-390

QUALIFICATION OF DELTA-PROTEKT® KL 120 FOR USE ON GRADE A490 HIGH STRENGTH STRUCTURAL BOLTS

TEST METHODOLOGY PER IFI-144

By

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EXECUTIVE SUMMARY

This investigation is an addendum to a previous investigation of Doerken's DELTA-PROTEKT® KL 105 coating system conducted in accordance with IFI-144[1] (IBECA Research Report TIR 10-119, Aug. 6, 2017) [1]. Given that DELTA-PROTEKT® KL 105 satisfies the requirements of ASTM F3019/F3019M, Grade 4, the previous report served as the technical basis for ASTM Committee F16 on Fasteners to approve the use of ASTM F3019/F3019M[2] (Grade 4) zinc flake coating system on Grade A490[3] high strength structural bolts. The objective of this current investigation is to qualify DELTA-PROTEKT® KL **120** coating system, a variant within the family of DELTA-PROTEKT® coating systems that also fulfills the requirements of ASTM F3019/F3019M Grade 4. The previous investigation demonstrated that DELTA-PROTEKT® coatings satisfy performance criteria listed in IFI-144, notably, *corrosion performance*, *coating adhesion*, and *paintability*. Consequently, testing related to those performance criteria are not repeated in this current investigation. Rather, this investigation addresses the most significant aspect of IFI-144, i.e., the risk of hydrogen embrittlement (HE). Process qualification performed in accordance with ASTM F1940 demonstrated that the risk of *internal* hydrogen embrittlement (IHE) from the DELTA-PROTEKT® KL **120** coating *process* is nil. Qualification for *environmental* hydrogen embrittlement (EHE) was performed in accordance with ASTM F2660, on bolts coated with DELTA-PROTEKT® KL **120**. More precisely, testing was conducted with (i) "standard bolts," having hardness within the limits for Grade A490 (i.e., 33-38 HRC), and (ii) "specimen bolts," produced strictly for this study, having hardness slightly exceeding the maximum limit of 38 HRC to simulate "worst case conditions." The results confirmed that DELTA-PROTEKT® **KL 120**, same as DELTA-PROTEKT® KL 105, does not promote environmental hydrogen embrittlement (EHE) on Grade A490 structural bolts.

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

[2] **ASTM F3019/F3019M**, "Standard Specification for Chromium Free Zinc-Flake Composite, with or without Integral Lubricant, Corrosion Protective Coatings for Fasteners" was adopted by ASTM Committee F16 in 2014; it is applicable to **DELTA PROTEKT®** zinc flake coating systems licensed by Doerken Coatings North America, Grass Lake, MI.

[3] Any reference to Grade A490 implies all 150 ksi bolt grades listed in ASTM F3125/F3125M for which the coating permission has been granted by ASTM Coomittee F16: i.e., Grade A490, Grade A490M, and Grade F2280.

RECOMMENDATIONS TO ASTM COMMITTEE F16

Based on the findings of this investigation, it is recommended that ASTM Committee F16 on Fasteners formally approve the use of DELTA-PROTEKT® **KL 120** coating system for use on F3125/F3125M, Grade A490 high strength structural bolts.

Given that DELTA-PROTEKT® **KL 120** fulfills the requirements of F3019/F3019M Grade 4, L2, same as DELTA-PROTEKT® KL 105, and given that F3019/F3019M Grade 4 is already a permitted coating (see F3125/F3125M, Table A1.1), no revision of F3125/F3125M is required. Nevertheless, a formal motion put forward and approved in subcommittee F16.02 would be appropriate.

Additional Recommendations

Separately, the recent publication of a combined zinc flake coatings standard for fasteners, F3393, under the jurisdiction of subcommittee F16.03, has exposed the need to clearly distinguish which zinc flake coatings have been approved for coating Grade A490 structural bolts. It is therefore recommended that F3393 be revised to include an *appendix* on coating of structural bolts that:

i. lists which classification code(s) in F3393 are *permitted* by F3125/F3125M, with commentary on the correspondence between F3393 on one hand, and F1136/F1136M, F2833, and F3019, on the other hand,
ii. defines the required performance criteria specific to structural bolting (e.g., coating thickness, nut oversize allowance, Rotational Capacity testing, etc.),
iii. lists or references (directly or indirectly) which commercial coating systems under F1136/F1136M, F2833, and F3019, have undergone IFI-144 qualification and are approved specifically for use on Grade A490 bolts.

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1. OBJECTIVE

The objective of this investigation is to qualify DELTA-PROTEKT® KL 120 coating, a variant within the family of DELTA-PROTEKT® coating systems, for use with ASTM F3125/F3125M Grades A490, A490M and F2280[4] high strength structural bolts in accordance with IFI-144. These grades of high strength structural bolts are characterized by specified tensile strength of 150 to 173 ksi (1,040 to 1,210 MPa), and hardness of 33 to 38 HRC. DELTA-PROTEKT® KL 120 fulfills the requirements of ASTM F3019/F3019M Grade 4, which is a permitted coating per ASTM F3125/F3125M, Table A1.1.

ASTM F3019/F3019M, "*Standard Specification for Chromium Free Zinc-Flake Composite, with or without Integral Lubricant, Corrosion Protective Coatings for Fasteners*" was adopted by ASTM Committee F16 in 2014 and is applicable to DELTA PROTEKT® zinc flake coating systems licensed by Doerken Coatings North America headquartered in Grass Lake, MI, notably, DELTA-PROTEKT® KL 120 and DELTA-PROTEKT® KL 105 Zinc Flake coatings.

This investigation is an addendum to a previous investigation of Doerken's DELTA-PROTEKT® KL 105 coating system conducted in accordance with IFI-144 (IBECA Research Report TIR 10-119, Aug. 6, 2017) that served as the technical basis for ASTM Committee F16 on Fasteners to approve the use of ASTM F3019/F3019M (Grade 4) zinc flake coating system on Grade A490 high strength structural bolts. The previous investigation demonstrated that DELTA-PROTEKT® coatings satisfy performance criteria listed in IFI-144, *notably, corrosion performance, coating adhesion*, and *paintability*. Consequently, testing related to those performance criteria are not repeated in this current investigation. Rather, this investigation addresses the most significant aspect of IFI-144, i.e., the risk of hydrogen embrittlement (HE).

DELTA-PROTEKT® KL 120 is a coating system that is comprised of a zinc-flake basecoat containing integral lubricant, and an inorganic topcoat, DELTA-PROTEKT® VH 301.1 GZ.

[4] Any reference to Grade A490 in this document implies the inclusion of Grades A490M and F2280.

2. BACKGROUND

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) [5,6], 2014 (non-chromium) [2,4]) and ASTM F2833 (2012) [3] and DELTA-PROTEKT® KL 105 (IBECA Research Report - TIR 10-119) [1]. These approvals were explicitly included in ASTM A490 and now in the new combined high strength structural bolt specification ASTM F3125/F3125M. 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 were treated as different coatings for qualification on Grade A490 bolts, which is the reason why separate studies and approvals were required for each coating system. The recent publication of a combined zinc flake coatings standard for fasteners, ASTM F3393, under the jurisdiction of subcommittee F16.03, is intended to eliminate the need for separate, product specific standards. It is intended that F3393 will eventually replace F1136/F1136M, F2833 and F3019/F3019M. However, before those standards can be withdrawn, both F3393 and F3125/F3125M must be amended to clearly distinguish which zinc flake coatings have been approved for coating Grade A490 structural bolts.

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 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.

3. COATED SAMPLES

Three separate lots of bolts, combined with nuts, and washers in two nominal inch sizes were tested: 1/2-13 UNC, and 7/8-9 UNC. The test pieces were coated by the DELTA-PROTEKT® KL 120 process, in accordance with ASTM F3019/F3019M (Grade 4, L2). In addition to testing 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.

3.1 Test bolts

The first set of tests were carried out on standard bolts with hardness within the limits for A490 bolts (i.e., 33-38 HRC). Another set of tests were carried out using *specimen* 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.

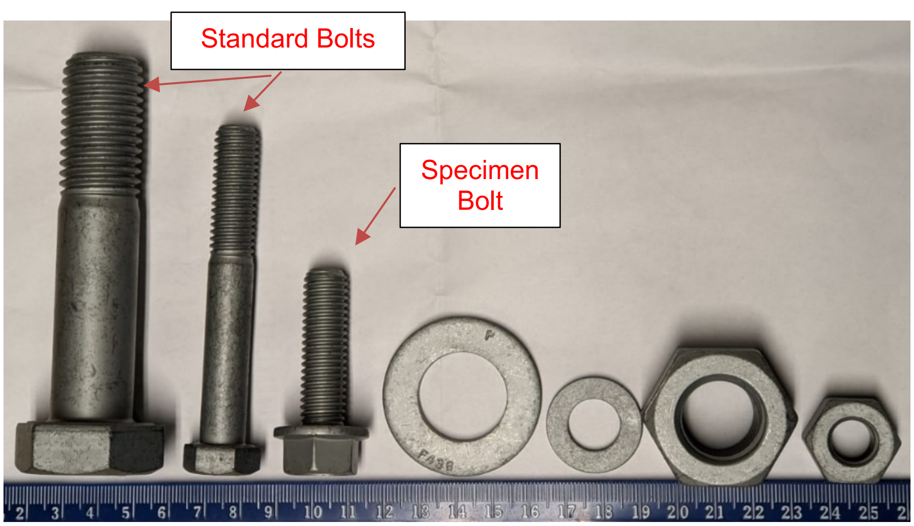


Figure 1 – Coated standard and specimen bolts with matching nut, washers

Standard bolts were hex head 1/2-13 x 3 1/2, and 7/8-9 x 4, and *specimen* bolts were hex flange head 1/2-13 x 1-1/2. All three sets of bolts were from homogeneous lots traceable to mill heats of alloy steel. Chemical composition for the test bolts is given in Table 1. The alloy steel grades for the 1/2-13 x 3 1/2, and 7/8-9 x 4 *standard* bolts were AISI 1330 and AISI 1335 respectively (i.e., manganese alloy). The alloy steel grade for the 1/2-13 x 1-1/2 *specimen* bolts was AISI 4037 (molybdenum alloy).

Table 1 – Test bolts chemical composition

	Standard Bolts		**Specimen Bolts**
Wt % Conc.	**1/2-13 x 3 1/2**	**7/8-9 x 4**	**1/2-13 x 1-1/2**
Carbon	0.31	0.36	0.36
Manganese	1.63	1.72	0.77
Phosphorus	0.016	0.010	0.012
Sulfur	0.015	0.010	0.010
Silicon	0.22	0.29	0.21
Vanadium	0.030	0.030	0.025
Nickel	0.05	0.06	0.06
Chromium	0.10	0.09	0.10
Molybdenum	0.017	0.009	0.233
Copper	0.17	0.12	0.13
Aluminum	0.002	0.001	0.002
Vanadium	0.027	0.027	0.025
AISI Designation	**1330M**	**1335V**	**4037V**

Mechanical properties for the standard and specimen bolts, coated and uncoated, are given in Tables 2 to 6. The average measured mid-radius hardness values for the standard 1/2 inch and 7/8 inch bolts were 36.6 and 36.4 HRC, respectively. Hardness of specimen bolts was measured to be 38.2 HRC. The specified hardness range for A490 bolts is 33-38, therefore, hardness of the specimen bolts slightly exceeds the maximum hardness of standard Grade A490 bolts.

Average wedge tensile strength values for the ½ - 13 X 1-1/2 specimen bolts was roughly 175 ksi. 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 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 standard and specimen bolts were not statistically altered after being coated (Tables 4-6). These results confirmed that the DELTA PROTEKT® KL 120 coating process did not alter the mechanical properties of the standard and specimen bolts.

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

	1/2-13 x 3 1/2		7/8-9 x 4	
Sample	**Mid-radius (HRC)**	**Surface (HR 30N)**	**Mid-radius (HRC)**	**Surface (HR 30N)**
1	36.6	52.9	36.2	57.1
2	36.3	55.3	37.0	57.3
3	36.9	55.9	36.2	56.1
4	36.0	55.9	36.3	56.5
5	37.3	55.7		
Avg.	**36.6**	**55.1**	**36.4**	**56.8**

Table 3 – Specimen bolts hardness values (recorded after manufacturing)

1/2-13 x 1 1/2		
Sample	**Mid-radius (HRC)**	**Surface (HR 30N)**
1	38.0	56.2
2	38.3	54.7
3	38.3	55
Avg.	**38.2**	**55.3**

Table 4 – Standard bolts full sized wedge tensile test results 1/2-13 x 3 1/2

1/2-13 x 3 1/2 uncoated bolts			1/2-13 x 3 1/2 coated bolts	
Sample	Load (lbf)	Stress (psi)	Load (lbf)	Stress (psi)
1	23,839	168,000	23,820	167,865
2	23,555	166,000	23,780	167,583
3	23,839	168,000	23,940	168,710
Avg.	**23,744**	**167,333**	**23,847**	**168,053**

Tensile Stress Area 0.1419 in^2

Table 5 – Standard bolts full sized wedge tensile test results 7/8-9 x 4

7/8-9 x 4 uncoated bolts			7/8-9 x 4 coated bolts	
Sample	Load (lbf)	Stress (psi)	Load (lbf)	Stress (psi)
1	79,926	173,000	79,830	172,792
2	79,002	171,000	79,970	173,095
3	79,926	173,000	80,300	173,810
4			79,530	172,143
5			79,880	172,900
Avg.	**79,618**	**172,333**	**79,902**	**172,948**

Tensile Stress Area 0.4620 in^2

Table 6 – Specimen full sized wedge tensile test results 1/2-13 x 1-1/2

1/2-13 x 1-1/2 uncoated bolts			1/2-13 x 1-1/2 coated bolts	
Sample	Load (lbf)	Stress (psi)	Load (lbf)	Stress (psi)
1	24,780	174,630	24,790	174,700
2	24,940	175,758	24,740	174,348
3	24,860	175,194	24,700	174,066
Avg.	**24,860**	**175,194**	**24,743**	**174,371**

Tensile Stress Area 0.1419 in^2

3.2 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*." By this approach, standardized notch square bars are coated as "witness" samples along with production parts and are tested using the incremental step load protocol prescribed in ASTM F1940.

In this investigation, process qualification for DELTA PROTEKT® KL 120 was conducted using ASTM F519 Type 1e square notch bars were used instead of ASTM F1940 square notch bars. ASTM F519 Type 1e notch bars have a smaller notch radius (R=0.01" instead of 0.02"), making them more sensitive than F1940 notch bars. ASTM F519 standardized notch bars are made of AISI 4340 steel, heat treated to 51-53 HRC. The dimensions of the notch bars are shown in Figure 2. The chemical composition of the notch bars used in this study is given in Table 7. The average measured hardness of the lot of notch bars used in this investigation was 52 HRC.

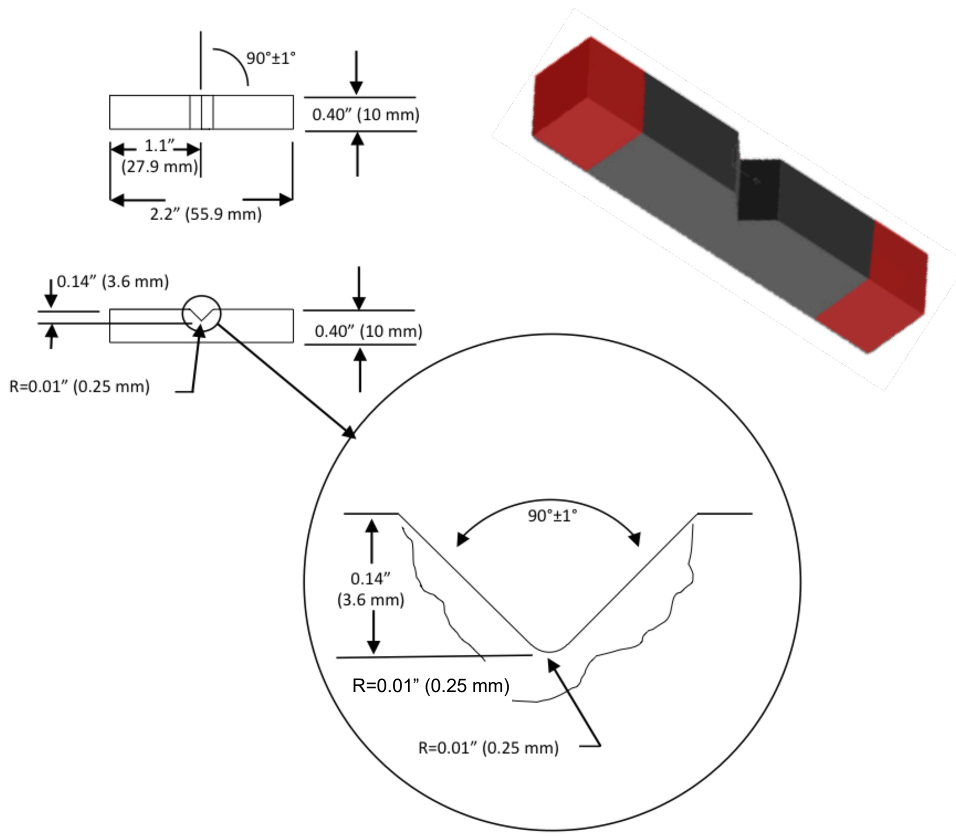


Figure 2 – ASTM F519 1e notch bar specimen

Table 7 – Chemical composition of ASTM F519 Type 1e 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	-

4. DELTA PROTEKT® KL 120 PROCESS

The DELTA-PROTEKT® KL 120 and DELTA-PROTEKT® VH 301.1 GZ is a proprietary coating system licensed by Doerken Coatings North America headquartered in Grass Lake, MI. This coating system is comprised of a zinc-flake basecoat, DELTA-PROTEKT® KL 120, and an inorganic topcoat, DELTA-PROTEKT® VH 301.1 GZ. The DELTA-PROTEKT® KL 120 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 120 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 8 to 16 microns.

The specimen bolts and ASTM F1940 notch bar specimens used in this study were coated at Elm Plating Company, 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 120 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 120 basecoat, parts were thermally cured for 30 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 110 °C part metal temperature. Figure 3b illustrates the process flowchart for the DELTA-PROTEKT® KL 120 and DELTA-PROTEKT® VH 301.1 GZ coating system.

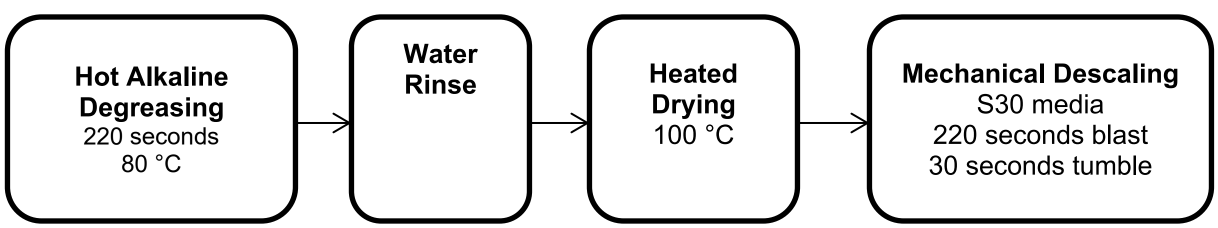


Figure 3a – Surface preparation process flowchart

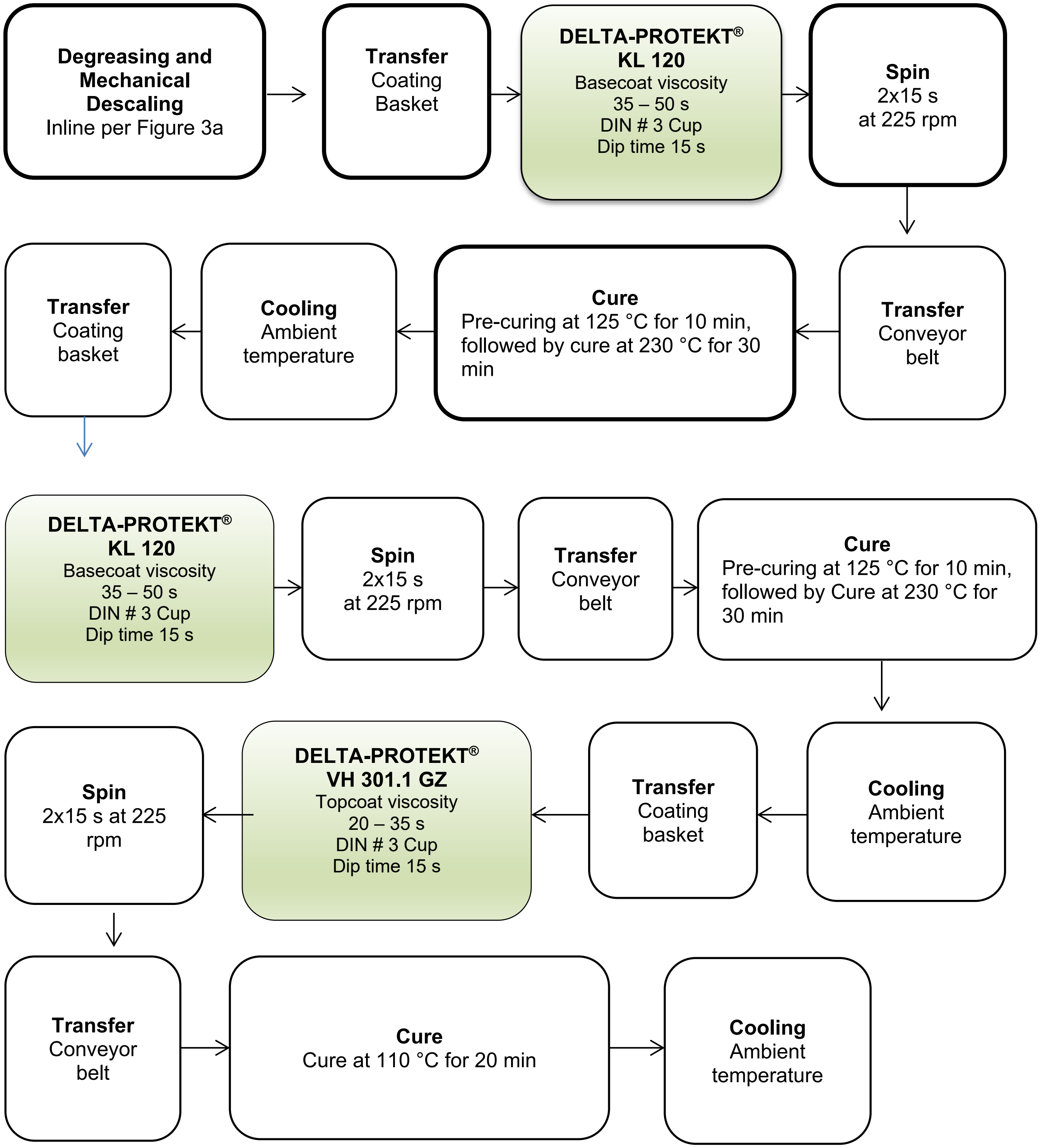


Figure 3b – DELTA-PROTEKT® KL 120 and DELTA-PROTEKT® VH 301.1 GZ process flowchart

5. EXPERIMENTAL METHODS

Table 8 shown below lists the test methodologies required by IFI-144 and applied in this investigation. This investigation addresses the most significant aspect of IFI-144, i.e., the risk of hydrogen embrittlement (HE). Methodology description for each test method is given in the Results (Section 6). In addition, torque-tension testing was performed to verify the rotational capacity criteria per ASTM F3125/F3125M.

Table 8 – IFI-144 qualification test methods performed in the current investigation

Sect.	Test	Specification	Condition
3.1	Hardness	ASTM F606	Standard Bolts / Specimens
3.1	Chemical Analysis	ASTM A751	Standard Bolts / Specimens
3.1	Tensile Pull	ASTM F606	Standard Bolts / Specimens
6.1	Microstructure	ASTM E3	Standard Bolts
6.2	Coating Thickness	Optical	Coated Specimen Bolts
6.3	Torque -Tension- Measurements	ASTM F3125	Standard bolts
6.4	Hydrogen Embrittlement (process)	ASTM F1940	F1940 notch square bars
6.5	Hydrogen Embrittlement (product)	ASTM F2660	Specimen Bolts

6. RESULTS & DISCUSSION

6.1 Microstructure

The metallurgical structure of standard bolts was examined using a Keyence Digital Microscope, VHS-5000. The findings were satisfactory and comprised normal fully transformed tempered martensite. Optical images of the microstructures are given in Figure 4 and 5, respectively. The discolorations observed in the images are etching artifacts.

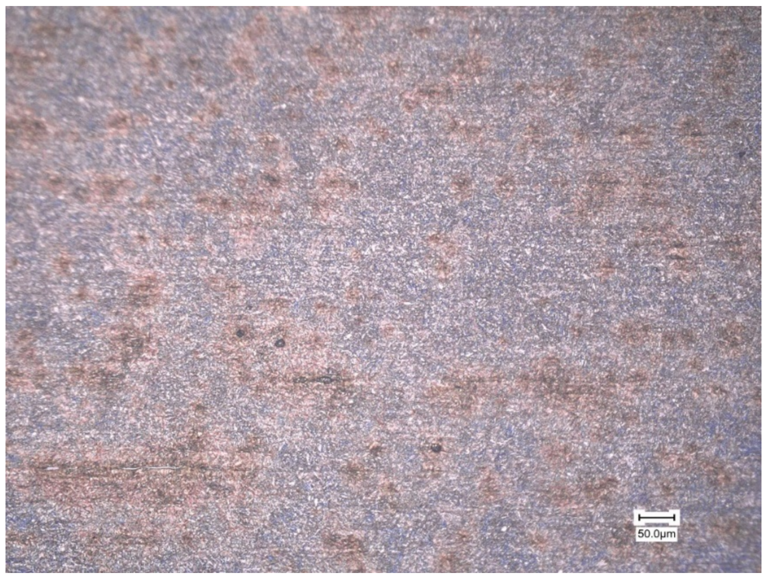


Figure 4 – Microstructure 1/2-13 x 3 1/2 standard bolt

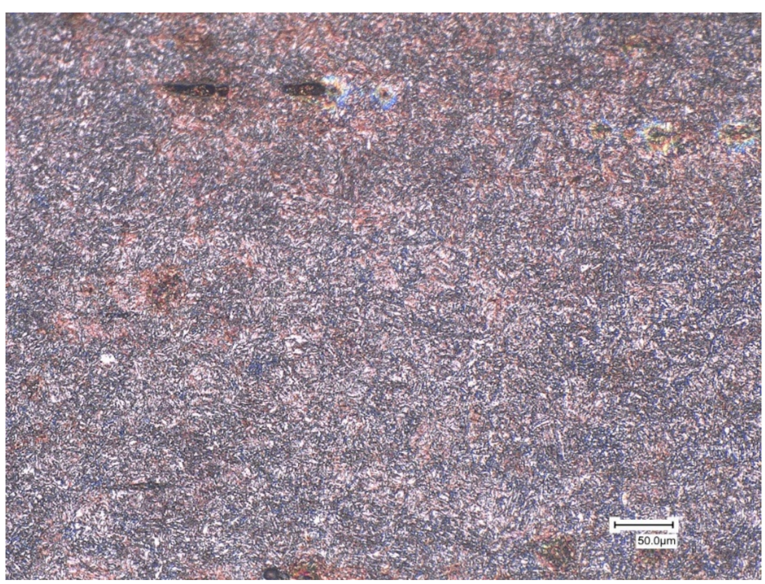


Figure 5 – Microstructure 7/8-9 x 4 standard bolt

6.2 Coating thickness

Coating thickness was measured using a Keyence Digital Microscope, VHS-5000 optical microscope. The bolts were sectioned, and mirror polished to measure coating thickness near the threaded sections. Coating thickness at the outer surfaces were measured for nuts and washers. For all parts, the average thickness was in the range of 0.83 mil (21 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 zinc flake coatings are given in ASTM F3125/F3125M, Table A1.2.

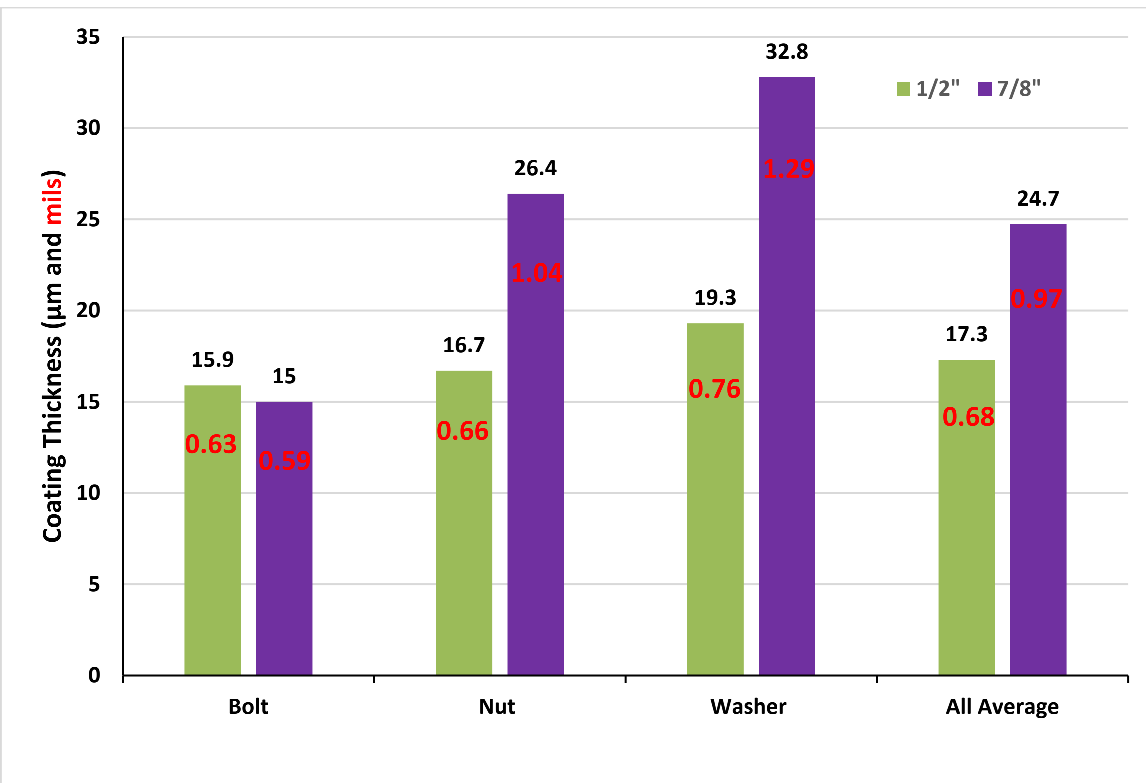


Figure 6 – Average coating thickness values (standard bolts) – bolts, nuts, washers

6.3 Torque Tension Measurement

IFI-144 stipulates that the coated bolts must pass the rotational capacity (RC) test. The acceptance criteria for RC testing are given ASTM F3125/F3125M. For this purpose, torque-tension testing was performed using equipment manufactured by Microcontrol inc. Torque-tension testing was performed on *standard A490 bolts* coated with DELTA-PROTEKT® KL 120. Standard Grade A490 bolts were used because specimens have greater strength properties than what is allowed for Grade A490 bolts and would therefore have provided misleading torque/tension data. The sizes of standard Grade A490 bolts used for the torque-tension testing are 1/2-13 x 3 1/2 and 7/8-9 x 4.

The results show the DELTA-PROTEKT® KL 120 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 DELTA-PROTEKT® KL 105 [1], and previously for DACROMET, GEOMET and MAGNI 565 [2-6]. The results for both test sizes are shown in Figures 7 and 8, 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 120 (bolts-washers). The K value range at installation tension was **0.14 - 0.16** for the ½ inch bolts and **0.12 - 0.14** for 7/8 inch bolts. These consistent 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 9-11 shown below, based on the length (L) to diameter (d) ratios for the standard A490 bolts.

Table 9 – length over diameter ratios for Standard A490 bolts

Bolt diameter d (in)	Length L (in)	L/d
0.50	3 1/2	7
0.875	4	4.6

Table 10 – Pre-installation verification testing and acceptance criteria

Bolt size (in)	Snug tension[5] (kips)	Min. installation tension[6] (kips)	Min. pre-installation verification tension[7] (kips)
1/2-13	1	15	16
7/8-9	5	49	51

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 11 – Rotational capacity testing and acceptance criteria

Bolt size (in)	Snug tension[8] (kips)	Installation tension[9] (kips)	Max. torque at installation tension[10] (ft-lb)	Min. final tension[11] (kips)
1/2-13	1	15	156	17
7/8-9	5	49	893	56
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)	

[5] Equal to 10% of minimum installation tension, rounded off to nearest kip.
[6] Equal to 70% of minimum specified tensile strength of bolts, rounded off to nearest kip. Used for design, actual installation and inspection.
[7] Equal to 1.05 times the minimum installation tension values, rounded to the nearest kip.
[8] Equal to 10% of installation tension, rounded off to nearest kip.
[9] Equal to 70% of minimum tensile strength of bolts, rounded off to nearest kip.
[10] T= 0.25 PD, where T= max. torque (ft-lb) P= tension (lbf) D= bolt diameter (ft).
[11] Equal to 1.15 times the minimum installation tension values, rounded to the nearest kip.

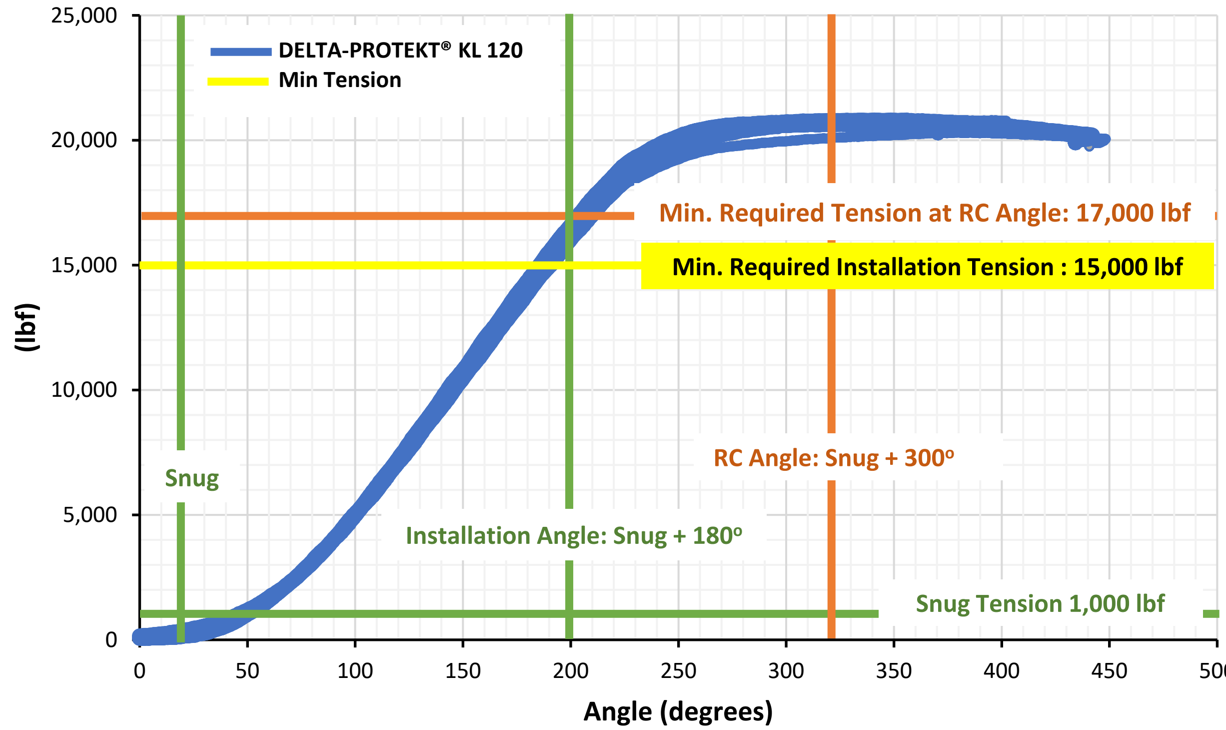


Figure 7a – RC test data for 1/2 inch parts: (i) angle vs. tension

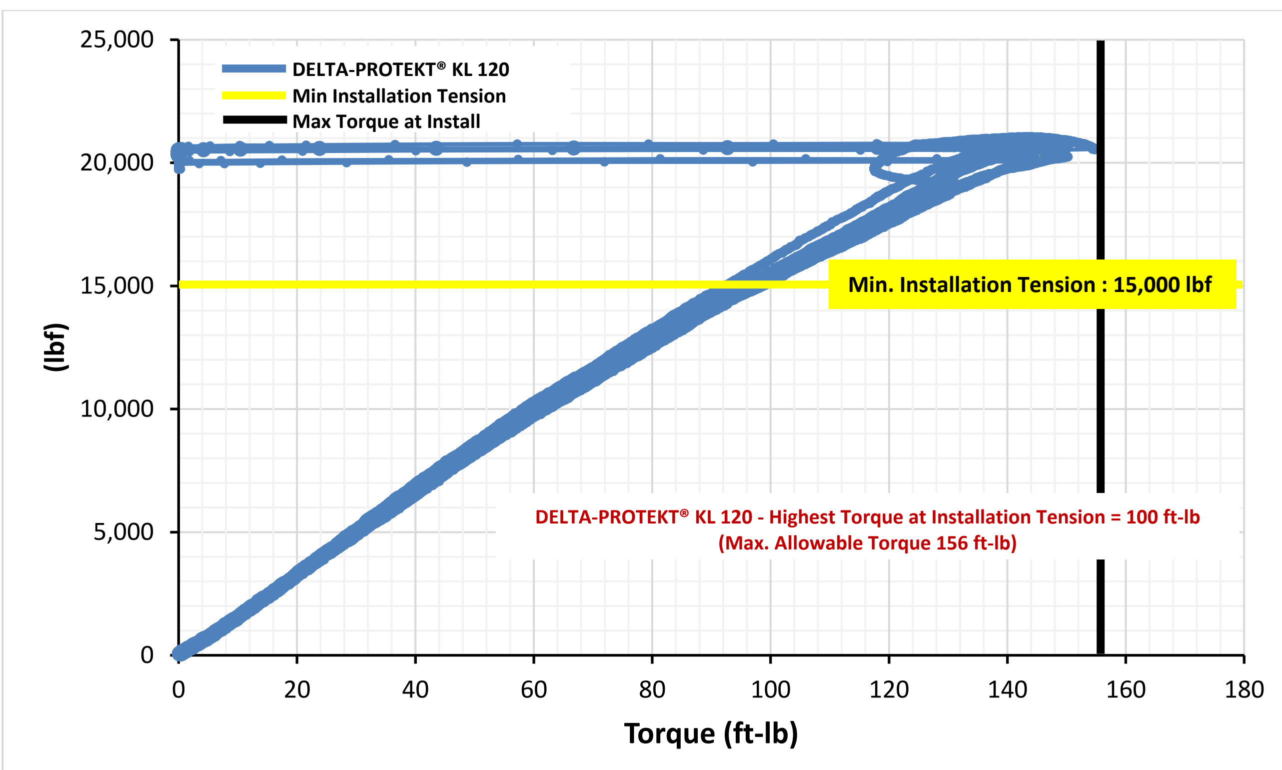


Figure 7b – RC test data for 1/2 inch parts: (ii) torque vs. tension (K = 0.14-0.16)

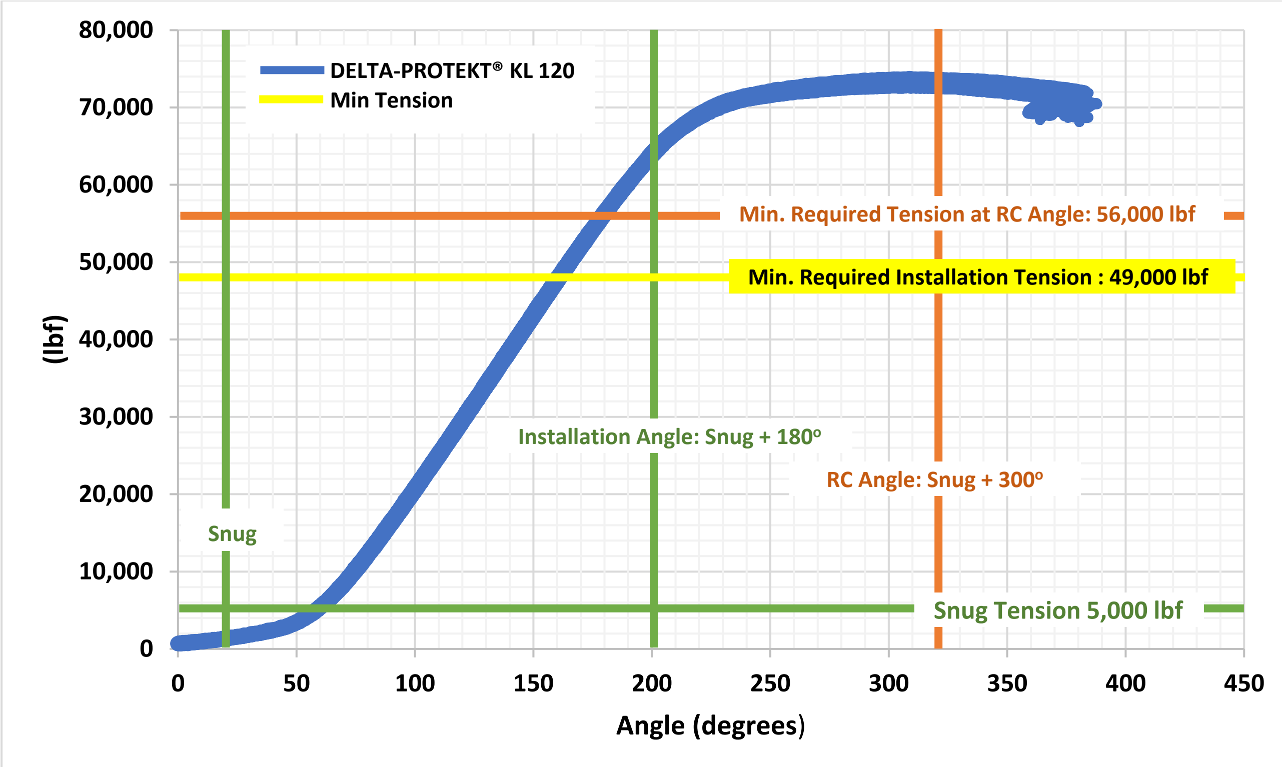


Figure 8a – RC test data for 7/8 inch parts: (i) angle vs. tension

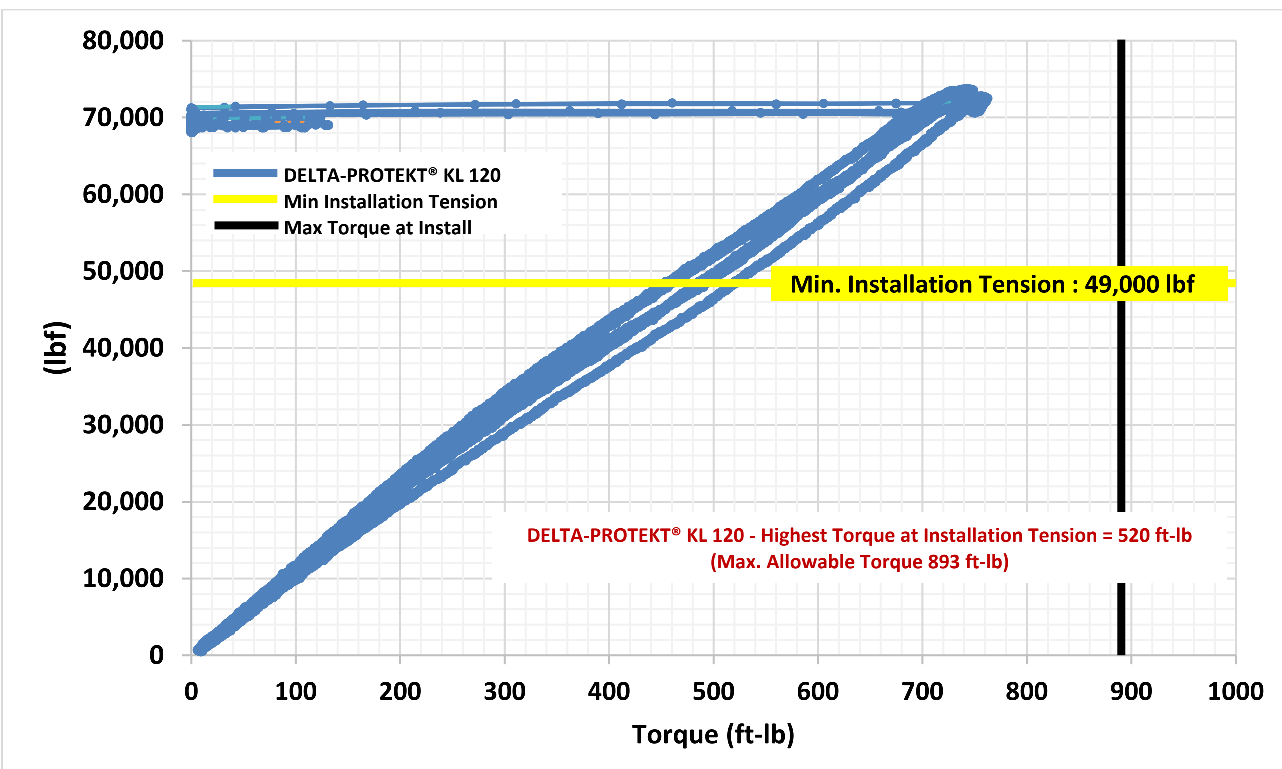


Figure 8b – RC test data for 7/8 inch parts: (ii) torque vs. tension (K = 0.12-0.14)

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 120 coating process. In this test, five (5) DELTA-PROTEKT® KL 120 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 (F1940_Mod) 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. The ratio of the threshold for each witness test specimen over the fast fracture strength represents the percent Notch Fracture Strength ($NFS_{\%}$) [7]. The results given in Figure 18 showed no reduction of $NFS_{\%}$ with average values of 94.7%, which is consistent with previous research findings relative to Zn flake coatings [8].

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

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

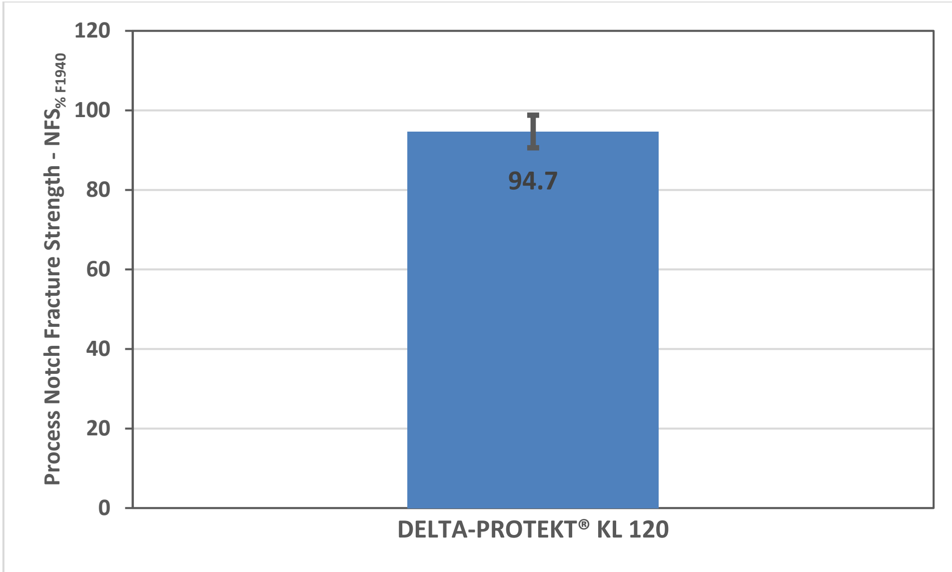


Figure 18 – NFS% values obtained for DELTA PROTEKT® KL 120 – the results indicate no risk of Internal Hydrogen Embrittlement (IHE)

6.4 Hydrogen embrittlement – product qualification

The primary concern that is intended to be addressed in qualifying coatings for use on Grade 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 Grade A490 bolt material to EHE. Susceptibility increases with increasing strength. By using *specimen* bolts heat treated to strengths slightly 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 investigation, OCP of DELTA-PROTEKT® KL 120 on a coated specimen bolt was measured and then compared to previous results obtained for DELTA-PROTEKT® KL 105, GEOMET®, DACROMET® and Magni 565. As can be seen from figures 19 and 20, OCP value for DELTA-PROTEKT® KL 120, after 24h of stabilization was -0.977 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 120 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.

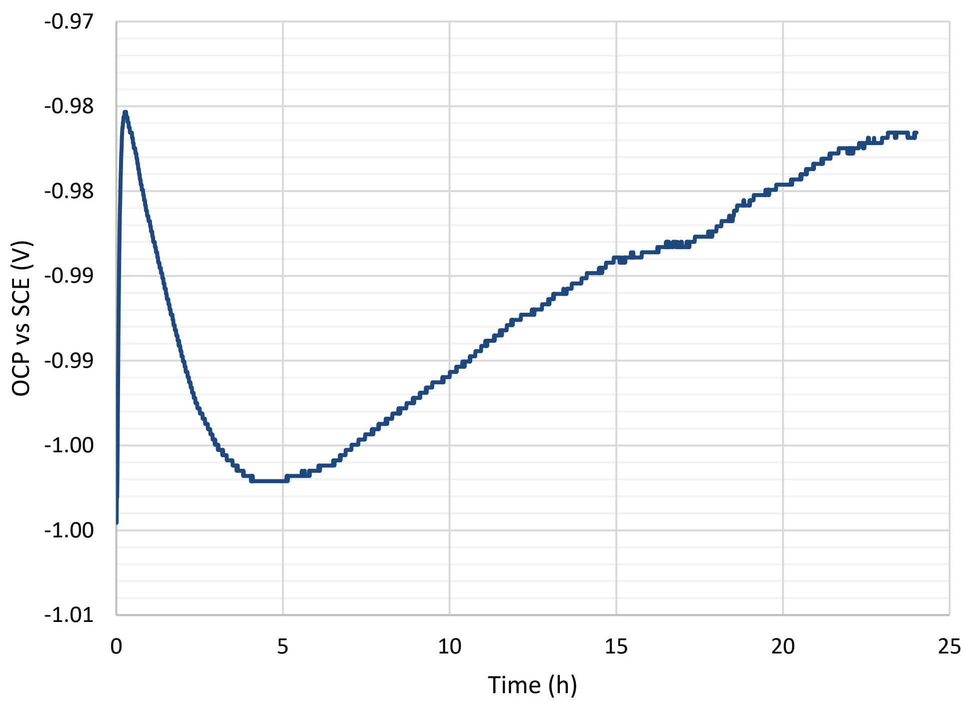
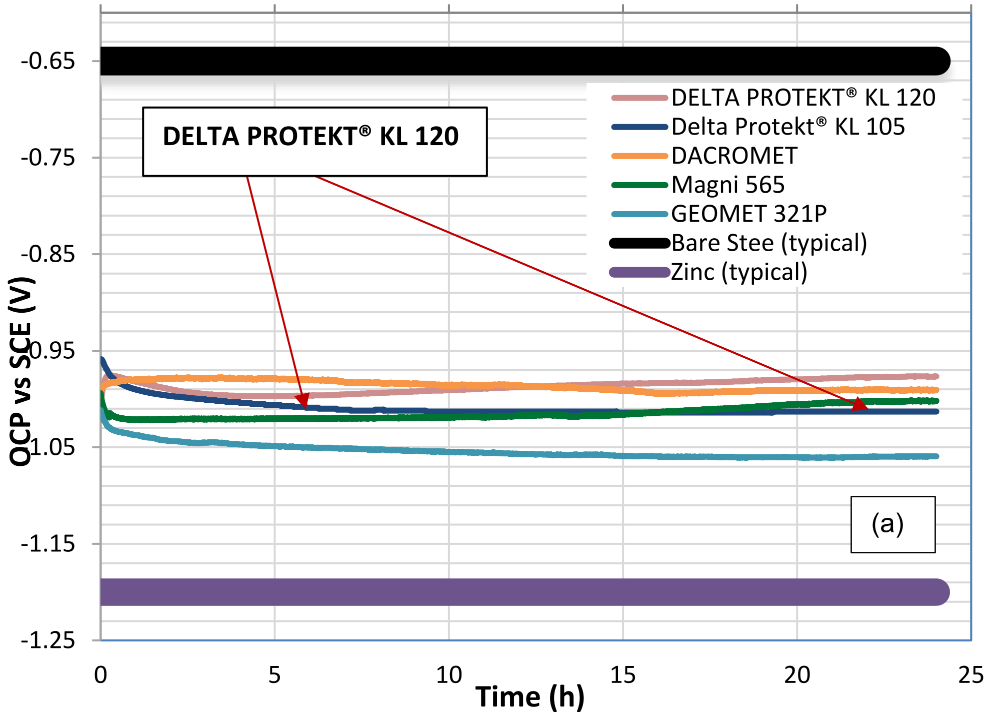


Figure 19 – OCP test progression for DELTA-PROTEKT® KL 120 over 24 h period



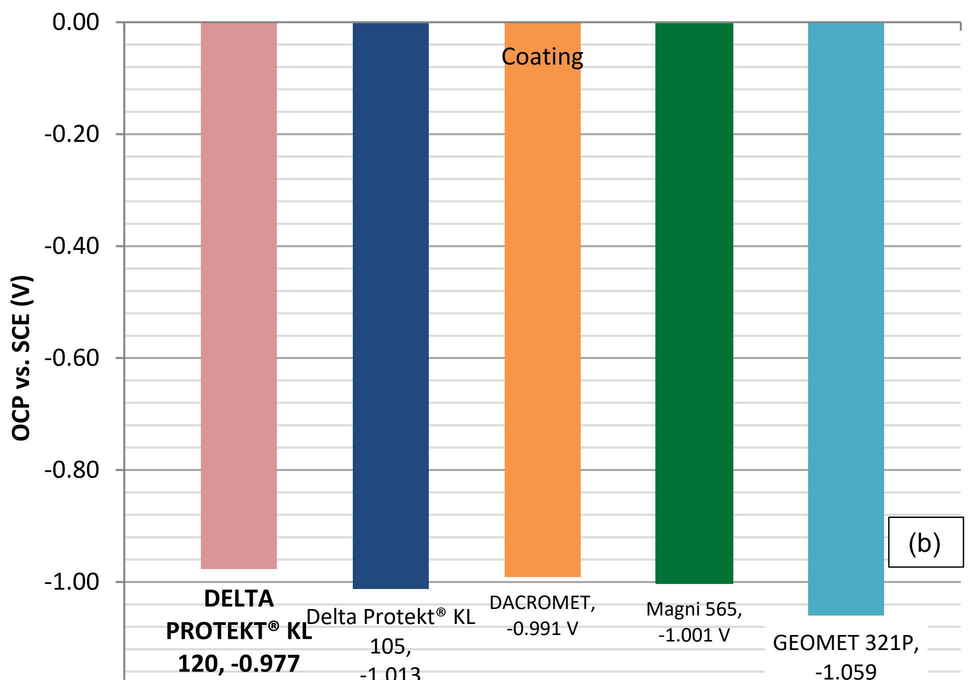


Figure 20 (a,b) – OCP test progression for DELTA-PROTEKT® KL 120 compared to previously approved coatings. (a) progression over 24 h, (b) stabilized value after 24 h

The first step in the testing sequence was a measurement of the *fast fracture* load of the standard and specimen bolts in bending ($FFS(B)_{coated}$). The average value for each lot was used as baseline value. The EHE susceptibility of the fastener/coating system was tested in environmental conditions using the incremental step load (ISL) methodology described in ASTM F2660 to obtain 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 made in the exposed threads between the gripping fixtures and had a length of one diameter. For the environmental tests, the standard and specimen bolts 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. The average 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 standard and specimen 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 97 % and 93 % for *standard* bolts and *specimen* bolts respectively. These test results demonstrate the DELTA-PROTEKT® KL 120 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 Grade A490 bolts. Therefore, the results demonstrate the DELTA-PROTEKT® KL 120 coating satisfies the acceptance criteria for EHE on Grade A490 bolts. Furthermore, the results also confirm that

Delta Protekt® KL 120 coating satisfies the acceptance criteria for EHE on Grade A490 bolts of *all sizes*, ranging from ½ inch to 1-½ inch.[12]

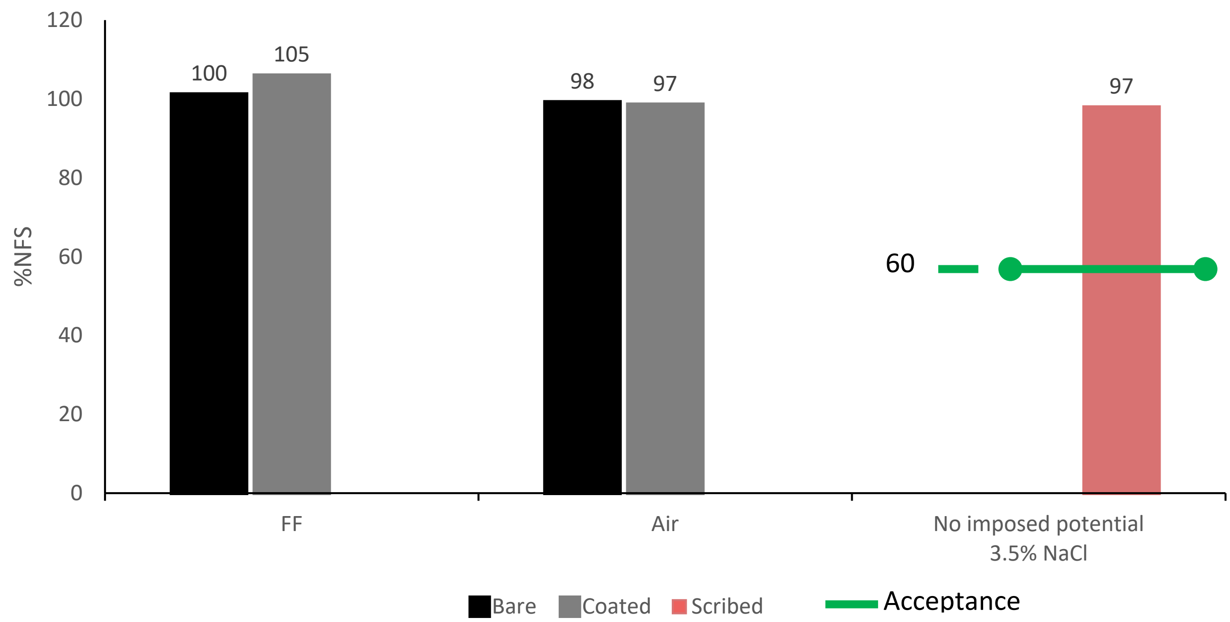


Figure 21 – 1/2-13 x 3 ½ STANDARD BOLTS average threshold NFS%
(using FF coated as baseline strength for coated samples)

[12] Given that the nominal size range for Grade A490 bolts is ½ inch to 1-½ inch, ASTM F2660 offers a calculation-based approach to project "equivalent" acceptance threshold values when bolts smaller than 1-½ inch are tested. For ½ inch *test bolts*, ASTM F2660, Table 2 proposes an acceptance threshold value of 82.2% to be applicable to 1 inch *production bolts*, and 98.3 % to be applicable to 1-½ inch *production bolts*. From Figures 21 and 22, threshold values of 97% (*standard* bolts) and 93% (*specimen* bolts) were obtained using ½ inch test bolts. These results exceed the proposed threshold of 82.2 % for 1 inch production bolts and are very close to the 98.3% threshold for 1-½ inch production bolts. The size difference between ½ inch and 1-½ inch bolts is so great that the reliability of the proposed "equivalent" acceptance threshold between those two limiting sizes is lowest. This same objection was made in the previous report, IBECA Research Report TIR 10-119, Aug. 6, 2017, regarding the reliability of the "equivalent" acceptance threshold for 1-½ inch bolts when using ½ inch test bolts.

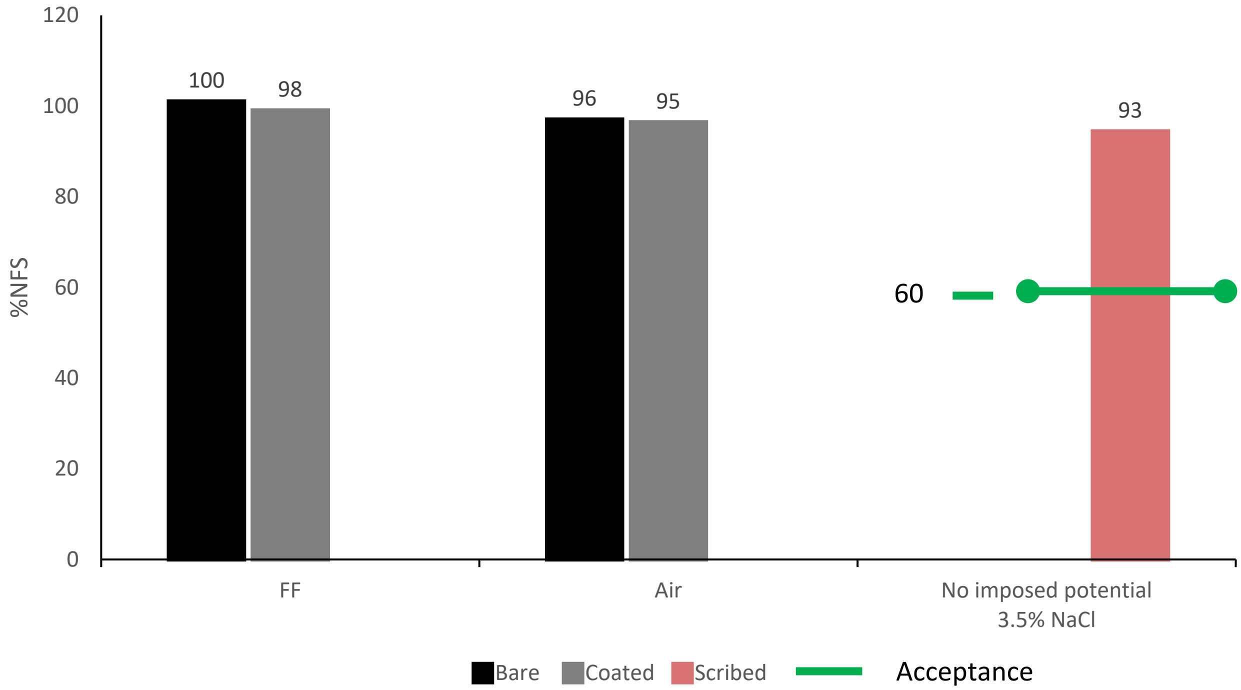


Figure 22 – 1/2-13 x 1-1/2 SPECIMEN BOLTS average threshold $NFS_{\%}$ (using FF coated as baseline strength for coated samples)

7. CONCLUSIONS

The objective of this investigation, conducted in accordance with IFI-144, is to serve as the technical basis for ASTM Committee F16 on Fasteners to approve the use of DELTA-PROTEKT® KL 120 coating system for use on Grade A490 high strength structural bolts. The DELTA-PROTEKT® KL 120 coating is a variant within the family of DELTA-PROTEKT® coating systems that fulfills the requirements of ASTM F3019/F3019M Grade 4. The investigation is principally intended to address the risk of delayed hydrogen induced failure, more specifically either due to internal hydrogen embrittlement (IHE) or environmental hydrogen embrittlement (EHE).

- DELTA-PROTEKT® KL 120 coating system satisfied the Rotational Capacity test criteria specified in ASTM F3125/F3125M.

- Process qualification results performed using ASTM F1940 methodology demonstrated that the risk of internal hydrogen embrittlement (IHE) from the DELTA-PROTEKT® KL 120 coating process is nil.
- Product environmental testing of high strength *specimen* bolts, performed in accordance with ASTM F2660, confirmed that DELTA-PROTEKT® KL 120, same as DELTA-PROTEKT® KL 105, does not promote environmental hydrogen embrittlement (EHE) on Grade A490 structural bolts, regardless of size.

Based on the findings of this investigation, it is recommended that ASTM Committee F16 on Fasteners formally approve the use of DELTA-PROTEKT® KL 120 coating system for use on F3125/F3125M, Grade A490 high strength structural bolts.

Respectfully submitted,

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2. *Qualification of ASTM F1136 Non-Chrome Coating (GEOMET® 321) for Use on ASTM A490 High Strength Structural Bolts*, IBECA Research Report 12-194*,* ASTM Committee F16 Documents, April 2014.

3. *Qualification of ASTM F2833 Coatings for use on ASTM A490 High-Strength Structural Bolts.* IBECA Research Report 08-12, ASTM Committee F16 Documents, January 2012.

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5. S. Brahimi, *Qualification of Dacromet coated ASTM A490M bolts for use on the San Francisco-Oakland Bay Bridge*, IBECA Technologies Research Report 09-64*,* December 2009.

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

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

<u>Other Relevant Documents</u>

1. AASHTO, LRFD Bridge construction specifications. 2009, The American Association of State Highway and Transportation Officials. p. 11.32-11.40.

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

3. RCSC, Specification for Structural Joints Using High-Strength Bolts, June 11, 2020.

REFERENCED SPECIFICATIONS

IFI

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

ASTM

1. ASTM F606/F606M *Standard Test Methods for Determining the Mechanical Properties of Externally and Internally Threaded Fasteners, Washers, Direct Tension Indicators, and Rivets*.

2. F1136/F1136M *Standard Specification for Zinc/Aluminum Corrosion Protective Coatings for Fasteners.*

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

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

5. ASTM F2660 *Standard Test Method for Qualifying Coatings for Use on F3125 Grade A490 Structural Bolts Relative to Environmental Hydrogen Embrittlement*.

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

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

8. ASTM F3125/F3125M *Standard Specification for High Strength Structural Bolts and Assemblies, Steel and Alloy Steel, Heat Treated, Inch Dimensions 120 ksi and 150 ksi Minimum Tensile Strength, and Metric Dimensions 830 MPa and 1040 MPa Minimum Tensile Strength.*

9. ASTM F3393 *Zinc-Flake Coating Systems for Fasteners*