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Evaluation of Alternatives to Electrodeposited Cadmium for Threaded Fasteners Applications

--Phase II and III Test Results

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1. Background

Cadmium (Cd) electroplating is coming under increasing pressure due to both environmental and worker safety issues. Cd plating is used for corrosion inhibition on diverse items such as aircraft components, locks, and fasteners. The problems with Cd are that it is an inherently toxic heavy metal poison, and therefore poses environmental and health problems throughout its life cycle. Not only does Cd pose ESH issues in the plating plant, but it can be leached (for example, in aircraft engine wash-downs), and thus can contaminate ground water simply by being exposed to cleaning solutions during normal use. At end of life it can also be leached into ground water. The European Commission has proposed requiring substitution of almost all Cd, hexavalent chrome, and even lead in electrical and electronic equipment. Since then various Cd alternative options have emerged.

Since 2005, Alcoa Fastening Systems (AFS) and Lockheed Martin have been conducting a collaborative research program to identify the most appropriate fastener coating materials for a Cd plating replacement. There are three primary uses for Cd plating. First, providing corrosion resistance for components; second, providing proper torque-tension (i.e. proper lubricity) for threaded fasteners; and last, providing oxidation-corrosion resistance and reliable conductivity for electrical connectors. As with Cd plating, it is unlikely that a single alternative will supplant all applications of Cd. This is consistent with AFS published initial screening results. The observation made during the Phase I study was that the Elisha Zinc-Nickel (Zn-Ni) and the AlumiPlate coatings were the best of the four candidates for Cd replacement. However, it is hard to conduct direct comparisons with different coating thicknesses, surface treatments, and lubrication among various Cd alternatives. Thus, further evaluation with more careful control of these parameters would be necessary. Therefore, the Phase II and III evaluations focused on more detailed characteristics of the Elisha Zinc-Nickel and AlumiPlate platings, along with a reference Cd plating.

Similar to the Phase I study, all testing was designed to simulate the typical use of threaded fasteners. Detailed, carefully controlled experiments and microscopy studies were used, including coating thickness, mechanical properties, stress corrosion, salt spray (fog) tests, torque-tension relationship, locking and breakaway torque measurements, and push-in and interference tests. The work presented here, which was carried out under Lockheed Martin Aeronautics Purchase Order numbers 7122826 and 7157361, summarizes our findings during the Phase II and III stages.

2. Test Program

2.1 Fasteners

As in Phase I, NAS1580A3T14 alloy steel fasteners were used to evaluate the properties of three different coatings. The base material was a low strength 8740 alloy with an Ultimate Tensile Strength (UTS) of 160 ksi. The purchased NAS1580A3T14 fasteners were stripped of the Cd and chromate conversion coating, followed by a baking cycle. They were then sent out for the application of the Cd alternative coatings. It is important to note that the nuts and bolts tested were all barrel plated and secondarily coated with some sort of dry film lubrication. Initial fastener observations revealed an off-set drive concentricity issue, meaning the center line of off-set drive does not coincide with that of fastener. This would impact mechanical performance of all Cd alternatives, especially fatigue properties. However, relative properties difference among all Cd alternatives is more important than absolute values. Thus, the Phase II and III evaluations continued.

2.2 Elisha Zn-Ni and AlumiPlate coatings

In this investigation, two candidate coatings were downselected from Phase I: Electrodeposited Zinc-Nickel (Zn-Ni) and Electrodeposited Aluminum (AlumiPlate). Detailed information on how the various coatings were applied to the fasteners was provided in the Phase I report. Table 1 lists specifications for each of the coatings tested.

Table 1. Summary of Coating Information.

Coatings	Specification	Conversion coating	Base coating target thickness (inch)	Lubrication
Cd	QQ-P-416 Type II Class 2	Chromate	0.0003-0.00045	Cetyl alcohol lube
Elisha Zn-Ni	AMS 2417 Type II	Surface mineralization	0.0003-0.00045	Sharperize 0121
AlumiPlate	MIL-DTL-83488D Type II, Class 3	None	0.0003-0.00045	Dry film lube

2.3 Test Requirements

Table 2 lists the engineering and testing requirements for evaluating alternatives to Cd coatings used for corrosion protection and lubricity on threaded parts. The listings also include acceptance criteria and references used for developing the tests. Each test consisted of ten specimens for each of the selected candidate coatings Zn-Ni and AlumiPlate, in addition to Cd plated fasteners.

Cross-sectional optical microscopy was performed on sample fasteners using a Nikon optical microscope (Model EPIPHOT 200, Japan). A portion of each sectioned specimen was mounted in diallyl phthalate powder (LECO Corporation, MI). The specimens then were mechanically polished to a 0.05 μm (alumina powder) surface finish using standard metallographic polishing techniques. During the evaluations, all testing machines and load cells were well calibrated.

Table 2. Critical test and performance requirements for corrosion protection and lubricity on threaded parts NAS1580A3T14. Each test consists of 10 threaded fasteners for each selected coating: Electrodeposited Elisha Zinc-Nickel and AlumiPlate, in addition to Cd plating.

Engineering Requirement	**Test Type**	**Reference Specification**	**Evaluation Criteria**
General properties	Appearance	AMS-QQ-P-416, NAS4002 &, NAS4444	Coating is continuous, smooth, uniform in appearance, free from blisters, pits, nodules, and other apparent defects.
General properties	Quick Dimensional and metallurgical check	AMS-QQ-P-416, NAS4002 &, NAS4444	1. General requirement, including thread geometry, microstructure, untempered martensite, grain flow, stays within the process specification. 2. Only desired fasteners shall be used for further evaluations.
General properties	Thickness and Composition Uniformity via X-ray	AMS-QQ-P-416, NAS4002, NAS4444 & MIL-STD-1312-12	1. Composition of the coating must stay within the process range when measured using the X-ray Alloy Composition Uniformity Test. 2. Only desired thickness of 0.0003 inch Cd alternatives shall be used for further evaluations.
General properties	Hydrogen content	AMS-QQ-P-416, NAS4002 &, NAS4444	Hydrogen content stays within the process specification.
Lubricity	Torque-Tension	NASM1312-15 NASM25027	Torque-tension for candidate material is within the range for Cd-plated fasteners. Fastener does not yield or fracture, threads do not strip.
Vibration resistance	Multi-cycle Run-on and Breakaway Torque	NASM1312-15 NASM25027	During installation, the maximum locking torque shall not exceed requirements. During removal, the minimum breakaway torque shall not be less than requirements in-lb. After 5 cycles of the locking torque test, nut and bolt threads shall remain in serviceable condition; when examined at high magnification, thread peel, cracks, galling, or splits are unacceptable.
Tensile property	Tensile strength	MIL-STD-1312-8	Tensile strength values to be comparable to cadmium plated fasteners.
Double shear property	Double shear strength	MIL-STD-1312-13	Double shear strength values to be comparable to cadmium plated fasteners.
Fatigue Resistance	Standard Fastener Fatigue Tests	MIL-STD-1312-11 and	1. Fatigue values to be comparable to cadmium plated fasteners. 2. Based on MIL-STD-1312-11, Fastener fatigue properties, including the

		ASTM- E466	S-N curves, a plot of stress against the number of cycles to failure N, will be evaluated.
Resistance to Embrittlement	Stress durability test	MIL-STD-1312-105 and ASTM F 519-93	No test fasteners fracture within the 1000 hour exposure time.
General properties	Tribological properties	AMS-QQ-P-416, NAS4002 &, NAS4444	Tribological properties, including friction and roughness, to be comparable to cadmium plated fasteners.
Resistance to insertion & Interference	Adhesion and push-in test	ASTM B 571-91	1. Fastener surface separation (weight loss, flaking, or peeling) for candidate coating is within the range for cadmium plated fasteners. 2. No separation (flaking, or peeling) from the basis metal or from any underplating at the edge. 3. Evaluate force required to insert fasteners into Aluminum structure with interference.
Corrosion Protection	Salt Spray (Fog)	NASM1312-1 and/or ASTM B117-03	1. A 1000 hours exposure until corrosion resistance ranking can be determined. 2. In the mean time, corrosion potential shall be closely monitored in real time at Alcoa technical Center.
Resistance to Stress Corrosion Cracking	Stress Corrosion	NASM1312-9	1. Evaluation based on the time to failure for the benchmark Cadmium-plated test fasteners. Determine if failure for fasteners with candidate coating is significantly different than equivalent Cadmium-plated fasteners. Development of cracks or failure by fracture constitutes failure of the specimen. In the mean time, corrosion potential shall be closely monitored in real time at Alcoa technical Center.

2.3.1 Salt Fog Testing

During salt-spray test, corrosion potentials were measured periodically among Zn-Ni, AlumiPlate, Cd and bare 8740 fasteners, to evaluate the relative long term galvanic sacrificial protection capability up to 1000 hours. The corrosion potential measurements were conducted via manual intervention to hook up the wires and electrometer. The overall intervention time was less than 30 minutes.

2.3.2 Stress Corrosion Cracking (SCC) Testing

In addition, stress corrosion tests were conducted to determine susceptibility of structural materials/coating to stress corrosion cracking up to 1000 hours. Two slightly different stress corrosion tests were conducted.

1. Stress corrosion testing per NASM specification while measuring corrosion potential.
2. Stress corrosion testing via Al structure while measuring corrosion potential. Instead of using specification allowed stainless steel, Al 7075 fixture and nuts were used and installed. This would allow the measurement of galvanic compatibility with Al.

The applied loads were 1300 and 1500 pounds for aluminum alloy 7075-T6 and stainless steel 303, respectively, due to limited offset torques. Five replicate fasteners for each coating system were stressed into each of these collar materials. At the same time, fastener potentials were measured to assess galvanic compatibility of the given fastener/coating/joining fixture systems. Each fastener/collar set had a lead-wire affixed to it, and were properly coated so as not to be a part of the galvanic potential. On a weekly basis, the potential of each fastener/collar set was then measured via this lead-wire, using a high-impedance electrometer, and referenced to a commercially-available saturated calomel electrode (SCE).

3. Test Results and discussion

Test results for the two potential Cd alternatives and the baseline Cd process are presented in this section. It appears that no one coating offers the same broad range of properties as Cd plating. Table 3 presents a summary of the pass/fail test results for the two Cd alternatives and the baseline Cd process for each test. Green and red shading indicate passing and failing results, respectively, compared to Cd plated bolts. Yellow shading indicates that interpretation of the test results is not straightforward and the discussion in the applicable results section should be reviewed to fully understand the rating. The following sections give detailed results of the individual tests and compare the performance of the candidate coatings to that of Cd plating.

Table 3. Test Results Pass/Fail Summary

Test Description	Elisha Zn-Ni	AlumiPlate
Appearance	🟩	🟩
Mass	🟩	🟩
Coating thickness	🟩	🟩
Microstructure	🟩	🟩

Hydrogen level	🟩	🟩
Torque Tension	🟩	🟩
Multi-cycle Run-on Torque	🟩	🟩
Multi-cycle Breakaway Torque	🟩	🟪
Tensile strength	🟩	🟩
Double shear strength	🟩	🟩
Fatigue	🟪	🟩
Sustained Tensile Load	🟩	🟩
Roughness	🟩	🟩
Friction	🟪	🟩
Push-in test	🟩	🟪
Salt Fog test	🟨	🟩
Salt Fog corrosion potential	🟩	🟩
Stress corrosion test (Stainless steel fixture)	🟩	🟩
Stress corrosion potential (Stainless steel fixture)	🟩	🟩

3.1 General properties

3.1.1 Appearance, mass and coating thickness

The appearance characteristics of each of the plating technologies are acceptable. Representative coupons of each plating type are shown in Figure 1. In addition, the average weight of each group was measured. The Cd coated fasteners showed the highest weight of 3.23 gram, slightly higher than AlumiPlate (3.20 gram) and Elisha ZnNi (3.21 gram).

All coatings were applied commercially and attempts were made to obtain a uniform coating thickness among all Cd alternatives. The target thickness was 0.3-0.45 mil, excluding any lubrication. However, accurately measuring the average thickness of these coatings is problematic. A few methods were used in this study, including electronic magnetic gages, weight change before and after each operation, and metallurgical cross-section to the average fastener's diameter subtraction before and after plating stripping. Figure 2 shows the various Cd replacement materials' thicknesses. In general the coatings meet the desired thickness. Please note the huge difference among different lubrications. This could potential affect fatigue and fastener torque performances. Additionally, a variation in plating thickness at different sections of the fasteners was observed. It is believed to be due to the presence of a voltage potential along the fasteners' length during the electro-plating operation. Figure 3 shows the metallurgical cross-section determined total thickness at different fastener locations for various coatings.

3.2 Microstructures and Hydrogen

Metallographic cross-sections were prepared through two fasteners from each group. The microstructure of the substrate materials from AlumiPlate and Elisha groups consisted of fine-grained tempered martensite, which is typical for quenched and tempered 8740 steel (Figure 4). No microstructural anomalies were observed, indicating no microstructural changes occurred during plating. All coatings were adherent to the substrate and relatively uniform in thickness. The micrographs taken from the cross-sections of different coatings at the fastener shank and the thread root can be seen in Figure 5. In addition, all hydrogen tests revealed zero or minimum hydrogen pick-up during plating (Figure 6). For comparison, bare fasteners after Cd stripping were measured as the control. Due to tiny amounts of coating elements, X-ray Fluorescence failed to quantitatively identify the detailed coating elements' compositions.

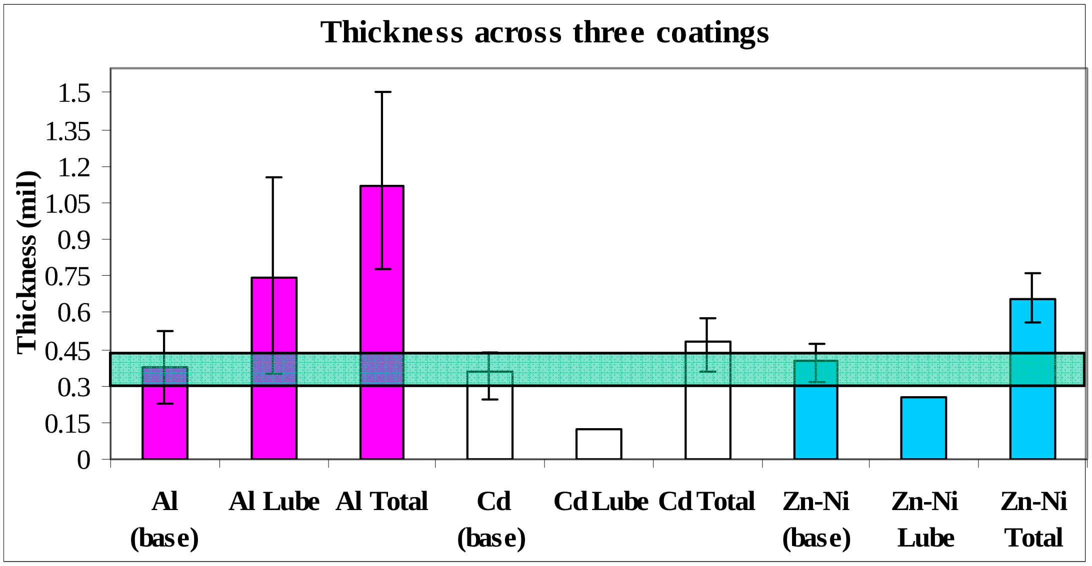


Figure 2: Mean plating thickness of three different coatings with target thickness of 0.3-0.45 mil, excluding any lubrication. The green box shows the target base coating thickness. The vertical bar shows the range of measured thickness. Same as below.

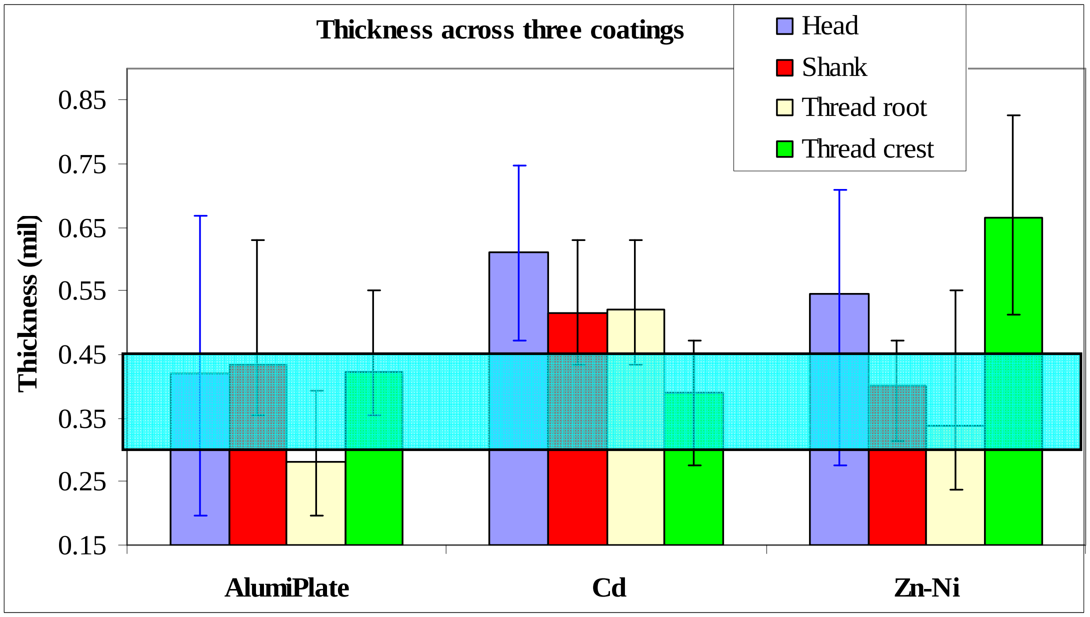
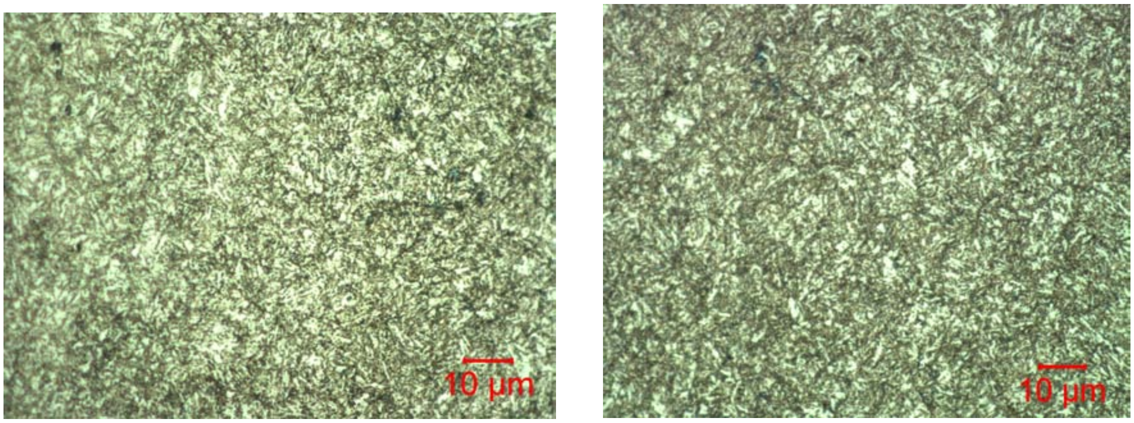
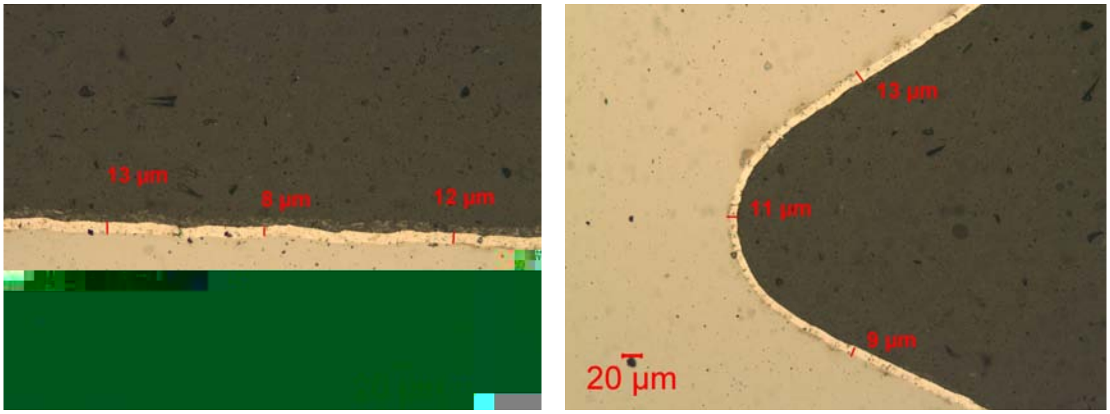


Figure 3: Variation in plating thickness at different locations of the fasteners via optical microscope approach with target thickness 0.3-0.45 mil, excluding any lubrication. The blue box shows the target base coating thickness.



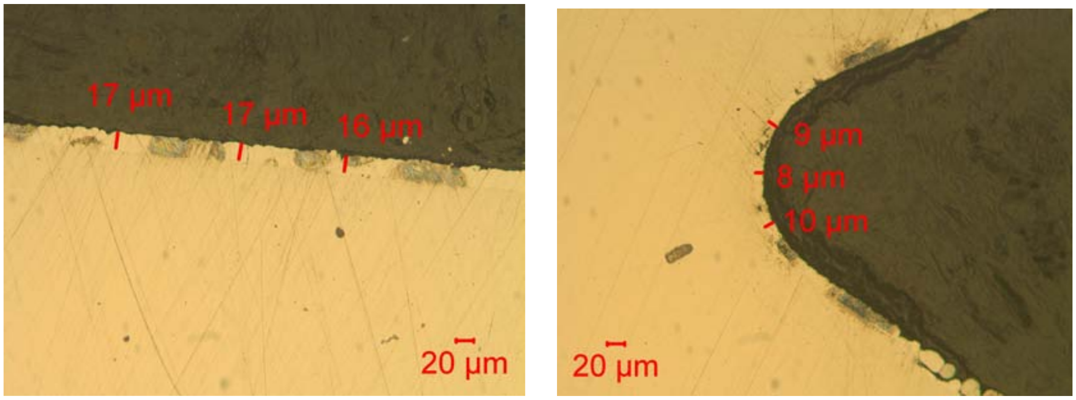
a) AlumiPlate b) Cd

Figure 4: Typical microstructures of the base metal 8740 after various coatings.



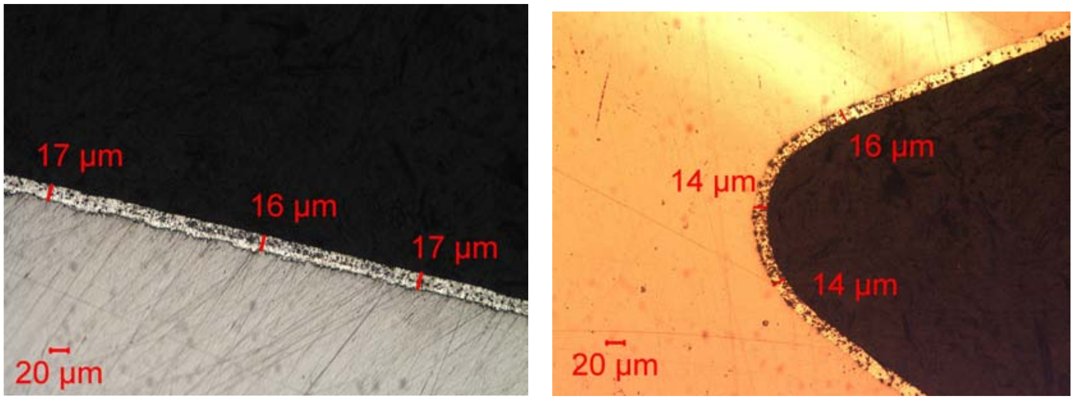
a) AlumiPlate; Shank

b) AlumiPlate; Thread root



c) Elisha Zn-Ni; Shank

d) Elisha Zn-Ni; Thread root



e) Cadmium; Shank f) Cadmium; Thread root

Figure 5: Micrographs showing the cross sections of coated fasteners.

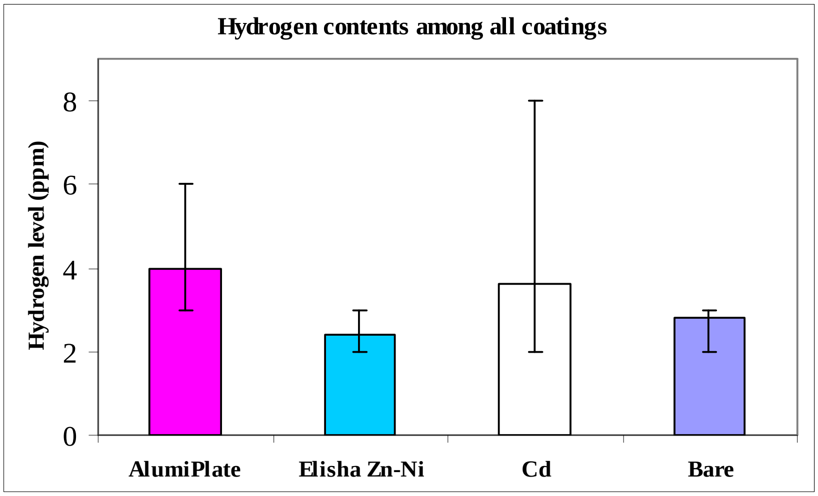


Figure 6. Measured Hydrogen level after various coatings.

3.3 Lubricity

3.3.1 Torque-Tension Properties

The tension as a result of a given nut torque was determined using the Torque-Tension stage. An incremental tightening of Aerospace grade K-nuts (KFN541L-3F nuts with dry film lubrication) method was used to measure tension load as a function of torque. Figure 7 shows the typical torque-tension results. It is desired that the candidate coatings mimic the torque-tension characteristics of bolts coated with cadmium. Please note that additional supplemental lubrications were used on all coatings. As expected, the lubricants had a significant effect on torque-tension characteristics, making the alternative coatings similar in performance to Cd plating. Due to surface lubrication, Zn-Ni and AlumiPlate have similar torque to achieve the required tension.

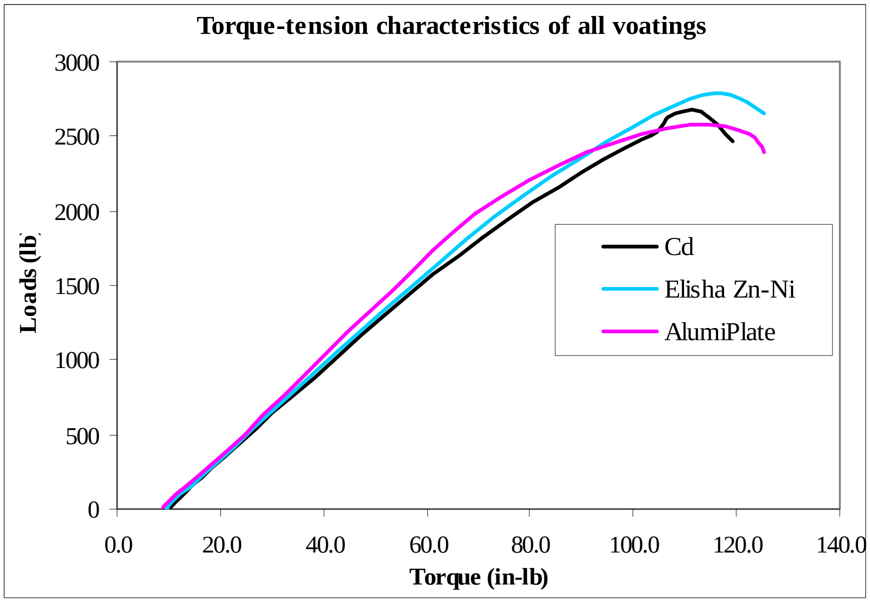


Figure 7: Typical torque-tension curves of various coatings.

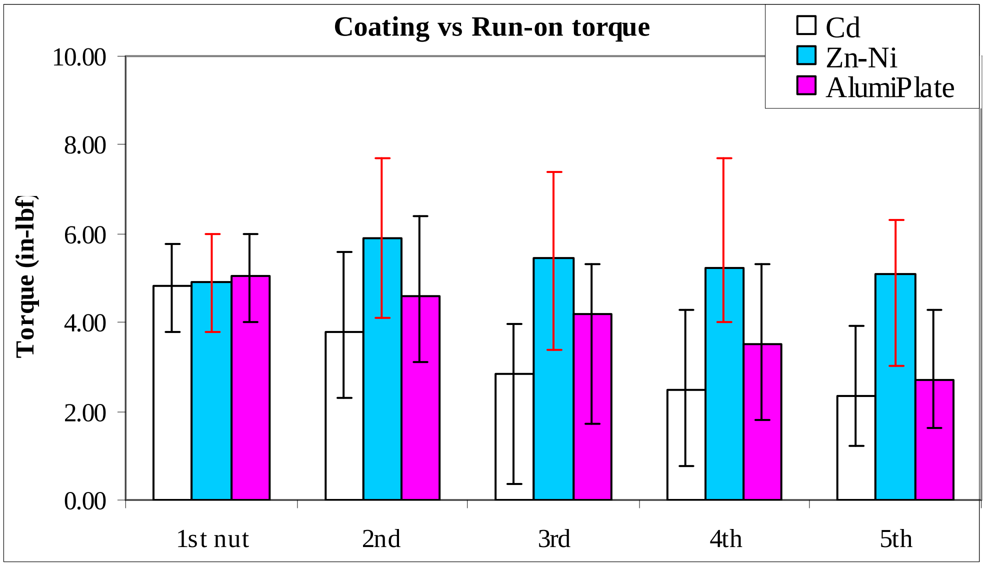
3.3.2 Run-on and Breakaway Torque

To evaluate the effects of coating and lubrication on maximum run-on and minimum break-away torque values, multi-cycle run-on and break-away tests were conducted. The same bolt was used to measure run-on and break-away torque values five times using five different new Aerospace grade K-nuts (KFN541L-3F nuts with dry film lubrication). These represent the performance at original manufacture or first use and for a reuse condition that might be encountered in field maintenance. The requirements for the KFN541L-3F nuts are a maximum of 18 in-pounds torque during locking and a minimum 2 in-pounds torque to start the nut rotating for removal. No axial load is allowed during the run-on and breakaway testing. An incremental tightening method was used that measured torque as a function of degrees of rotation. Nuts were engaged on the bolt threads and then driven an appropriate number of turns to expose 1 to 2 threads beyond the nut. The results are shown in Figure 7. The data shown are averages of 10 bolts per coating. It is desired that the candidate coatings have consistent and sustainable torque-tension characteristics.

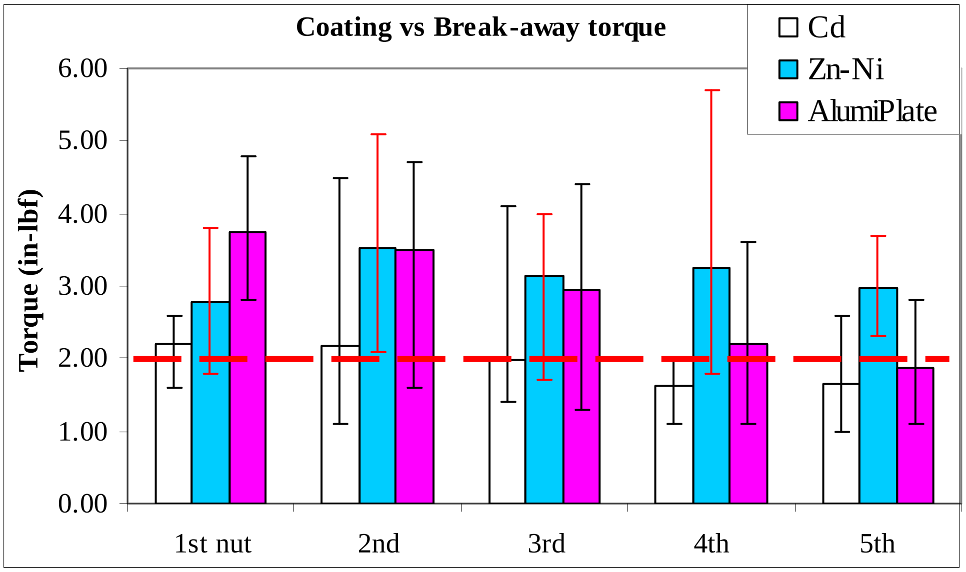
All run-on torque values meet the requirements of 18 inch-pounds. Data for AlumiPlate show a mean break-away torque of 3.9 in-pounds dropping to about 1.9 inch-pounds on 5th cycles. The Elisha Zn-Ni nut/bolt sets meet the specification requirements. The first cycle run-on torque is less than 18 inch-pounds. The breakaway torque mirrors the run-on torque and in all cases is greater than 2.0 inch-pounds. Subsequent cycles show a small *increase* in both run-on and break-away torque values. Further multi-cycle torque-tension tests need to be conducted to evaluate friction coefficients.

The first cycle of Cd and AlumiPlate showed elevated torque values. The locking feature of the nut is deformed during the first run-on. Also, the dry film lubricant on the nut and the plating on the nut and bolt become displaced and compressed during the first cycle. Subsequent cycles show a steady decrease in maximum values as the cycle number increases. The data for the Cd plated nut/bolt samples show more consistent behavior after the first cycle than AlumiPlate. The greater spread in the torque values by cycle for the candidate coatings may indicate that the candidates have overly thick coatings and lubricants and/or a lower inherent lubricity than Cd. The coatings and lubricants are progressively deformed as the cycle number increases, effectively reducing the interference between nut and bolt.

Micrographs taken after run-on and break-away testing are shown in Figure 9. For Cd plated bolts, the coatings are clearly removed from the bearing surfaces where load is applied after the first cycle. The tooth profiles show little or no wear with plating metal particles in the threads. The subsequent cycles result in little change (Figure 9(a) and (b)). However, for AlumiPlate and Zn-Ni plated bolts, the test coatings were glazed and scored after the first cycle. Thread galling also occurs. Further testing leads to more thread galling and particles left between threads (Figure 9(c)-(f)). Thus it is possible that Zn-Ni demonstrated higher run-on and break-away torque values as the cycle number increases due to base coating galling. The higher strength metal or silica particles leftover between threads could increase the interference between nut and bolt, resulting in higher torque values. However, the AlumiPlate coating is very soft and could be compressed even after thread galling, leading to reduced run-on and break-away torque values as the cycle number increases.

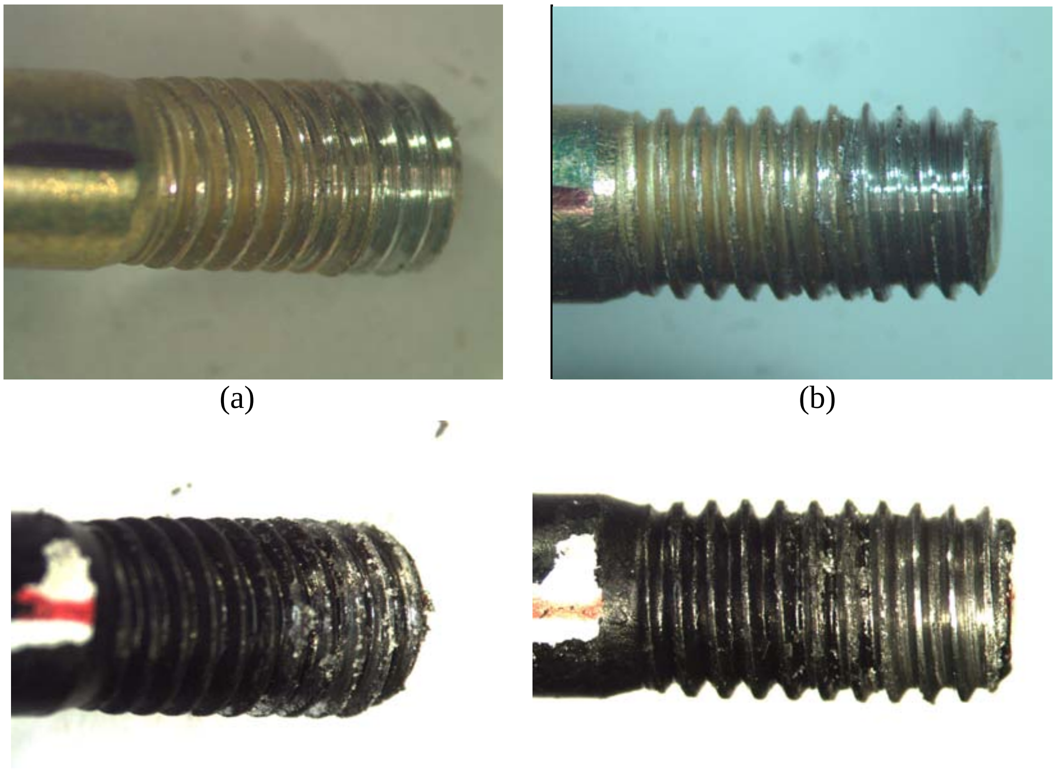


(a)



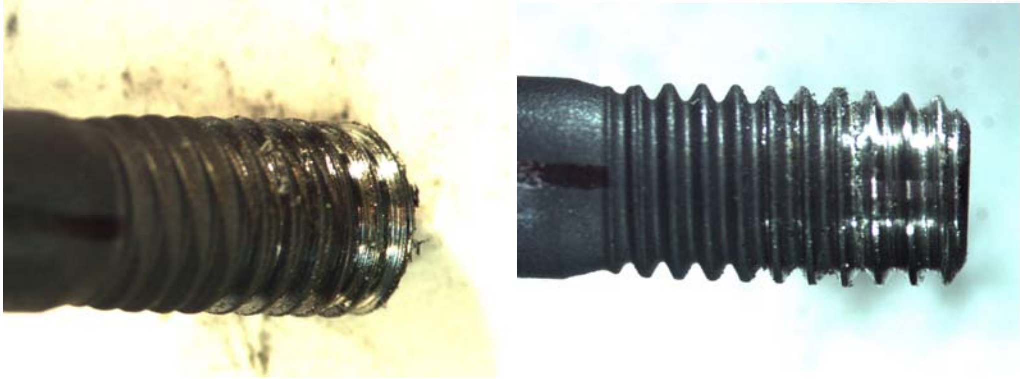
(b)

Figure 8 shows the 5-cycle maximum run-on and minimum break-away values for all Cd alternatives.



(a) (b)

(c) (d)



(e) (f)

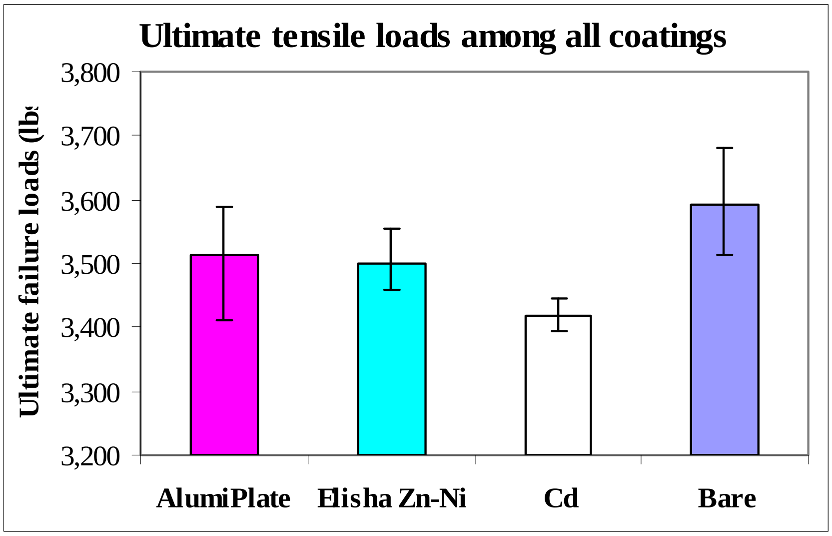
Figure 9. Micrographs after multi-cycle run-on and break-away experiments showing Cd plated bolt after 1st (a) and (b) 5th cycles, Zn-Ni plated bolt after 1st (c) and (d) 5th cycles, and AlumiPlate bolt after 1st (e) and (f) 5th cycle's tests.

3.4 Mechanical properties

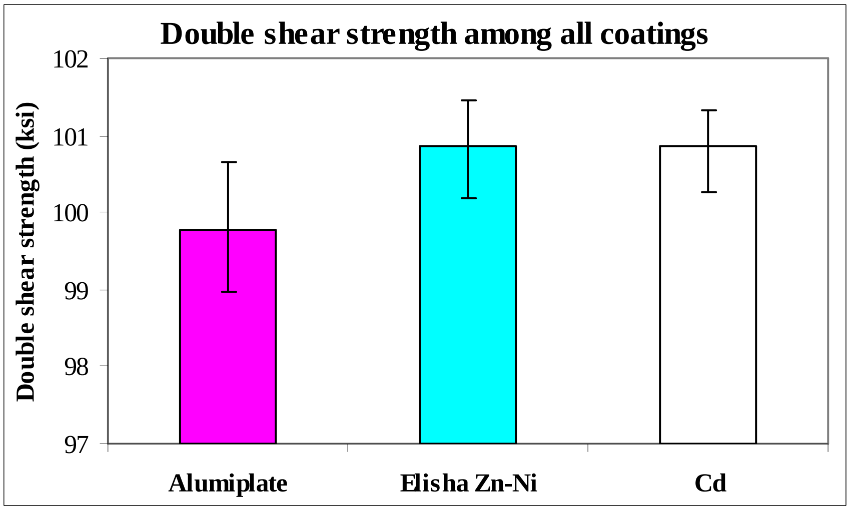
3.4.1 Ultimate tensile load and double shear strength

Ten specimens of each coating were tensile tested in accordance with NASM 1312-8 using a Universal Testing machine. The ultimate tensile tests were measured by applying load until fastener failure. The failure mode of all parts tested was head tension failure at the fillet radius. For comparison, bare NAS1580A3T14 fasteners were tested under the exact same conditions (Figure 10). After various coatings, fastener tensile strength typically drops due to the elevated temperature coating and baking processes. As expected, AlumiPlate and Zn-Ni coated bolts demonstrated higher failure loads. However, they all meet the specification requirement.

In addition, 10 specimens from each coating groups were tested for the double shear strength per NASM 1312-13 using a Universal Testing machine. Double shear strength results are shown in Figures 10. All coated bolts met the specification requirement. As known, shear testing inherently involves a number of variables. The test parts surface tribology, including friction coefficient, roughness, coating thickness, etc. could have a significant effect on the final shear strength value. Thus, it was expected to see different double shear strength values among various coatings due to different surface tribology.



(a)



(b)

Figure 10. Effects of various coatings on (a) tensile properties and (b) double shear strength.

3.4.2 Fatigue properties

It is well-known that fatigue life of a component could be affected by surface quality features, such as plating and lubrication thickness, nature and structure of coating, oxidation resistance, surface roughness, scratches, etc. These can cause stress concentrations or provide crack nucleation sites which can lower fatigue life depending on how the stress is applied. It is expected that discrepant plating and lubrication on the test fasteners could influence the fasteners' fatigue life, especially high cycle fatigue life. The fatigue testing was accomplished on 9 kip Static Testing machine. The testing produces a stress ratio of R=0.1 at a nominal frequency of 30 Hz. Fatigue test loads were specified as 45%, 60% and 80% of the minimum ultimate tensile failure load. The 45% ultimate tensile failure load corresponds to the NAS4002 specified load. In addition, un-coated or bare fasteners were tested under the same conditions for comparison. As mentioned before, due to off-set drive concentricity, actual fatigue life is much lower than specification limits. However, current fatigue data could still determine relative fatigue performances among all coatings.

The fatigue test data are presented graphically in Figure 11. Typical the S-N curves were generated to determine if there was a fatigue penalty associated with any of the coatings relative to Cd. The fatigue life of AlumiPlate coated fasteners was comparable to the Cd, indicating no apparent fatigue penalty is associated with these coatings. However, the Elisha Zn-Ni coated fasteners exhibited a lower fatigue life relative to Cd. This is consistent with a Boeing test report that the alkaline zinc-nickel fatigue coupons exhibited a significantly lower (11.5%) endurance limit relative to Cd. In addition, all coatings seem to improve fatigue life, compared to un-coated bolts (Figure 11). All of the failed fasteners had a Head Tension Failure at Off-Set Drive Cross Section to Fillet Radius. Each fracture was transgranular and exhibited typical fatigue characteristics.

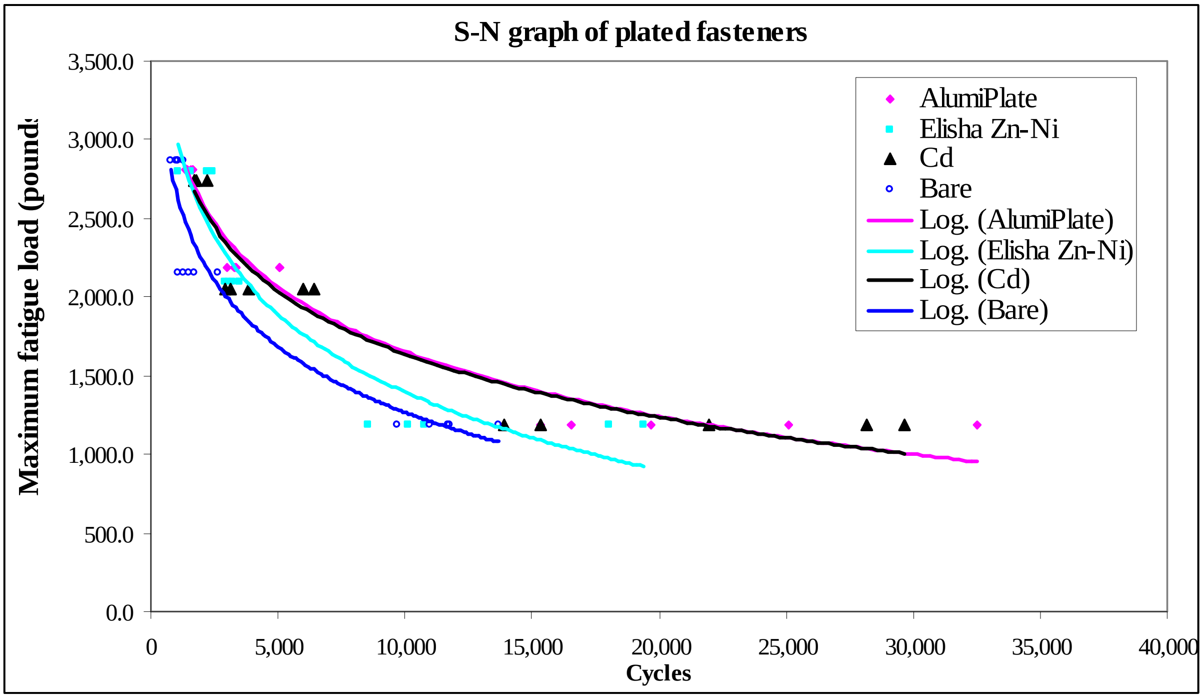


Figure 11. S-N plots for the various coated specimens

3.4.3 Stress-durability test

To determine the capability of externally threaded fasteners to withstand high stress load conditions, stress durability tests were conducted. Based on the fastener torque-tension relationship (Figure 7), a torque method was used. Test fasteners were assembled in a test block (Figure 11). Per NASM 1312-5, a minimum of two full threads of bolt were extended above the face of nut. Fasteners were tightened with a torque wrench to a torque equivalent of 70% of the minimum ultimate tensile failure load specified in specification NAS4002, which is 3180 pounds. Due to low recess torque values, we were unable to further increase loading torque to a value equivalent above 70% of the minimum ultimate tensile failure load. All of the fasteners' elongation data was recorded. All fasteners assemblies were to remain in a torqued condition at room temperature for more than 1000 hours.

Periodical and final examination revealed no cracking or fracture of any bolts. All fasteners passed 1000 hours at a tension of 70% of ultimate strength. In addition, no yielding or further elongation occurred, indicating load of 70% of ultimate strength is still below fastener yielding point.

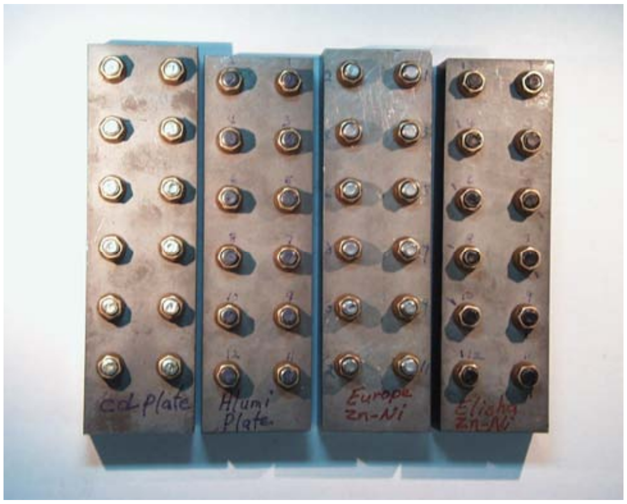
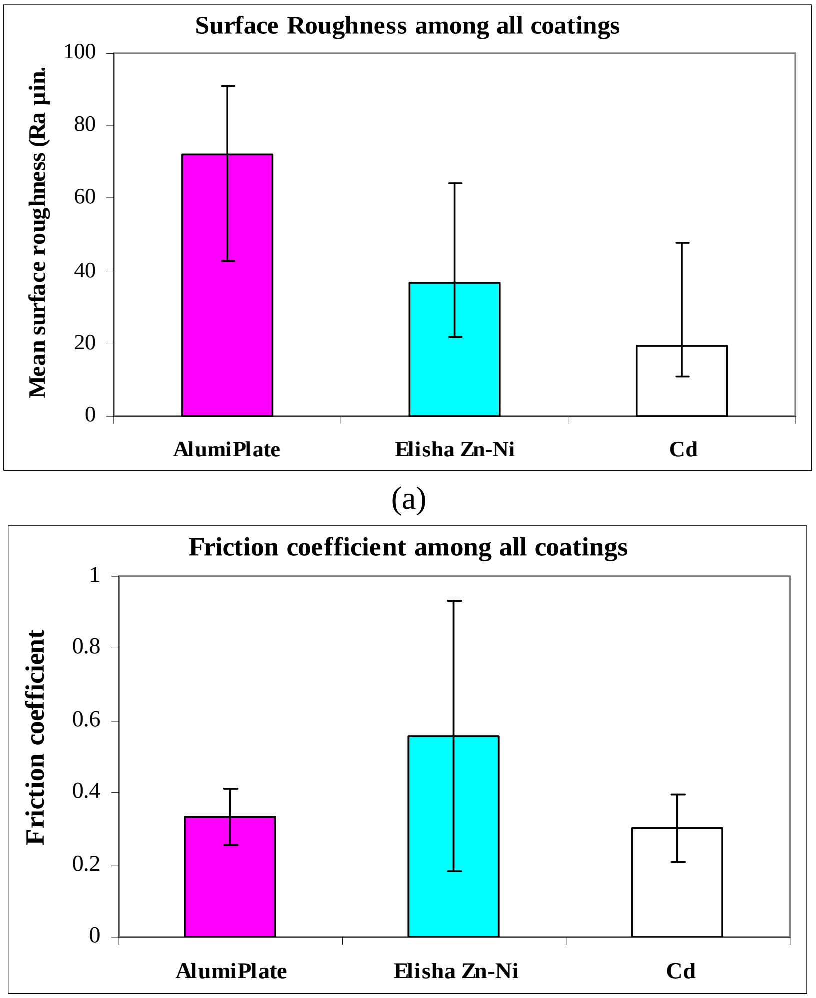


Figure 11. Stress-durability test blocks for various coatings.

3.5 Tribological properties

3.5.1 Surface Roughness and Friction coefficient

Ten specimens of each coating group were randomly selected to measure surface roughness (Ra values) using a Mitutoyo Surftest surface roughness tester. As can be seen in Figure 12, a higher surface roughness up to 90 micro-inches was recorded for AlumiPlate coatings while Zn-Ni coated parts had a very similar surface roughness to that of Cd plating. In addition, coefficient of static zero-load friction of all three coating materials has been determined (Figure 12). It is concluded that the AlumiPlate coatings have a coefficient of friction comparable to cadmium plating while Elisha Zn-Ni coating had significantly higher coefficient. This could explain why Zn-Ni coated bolts showed lower tensile strength than Cd plated bolts, but higher double shear strength and fracture load. It is widely known that shear testing inherently involves a number of variables such as component surface friction coefficient, roughness, and coating thickness. All these variables could have a significant effect on the final shear strength value.



(a)

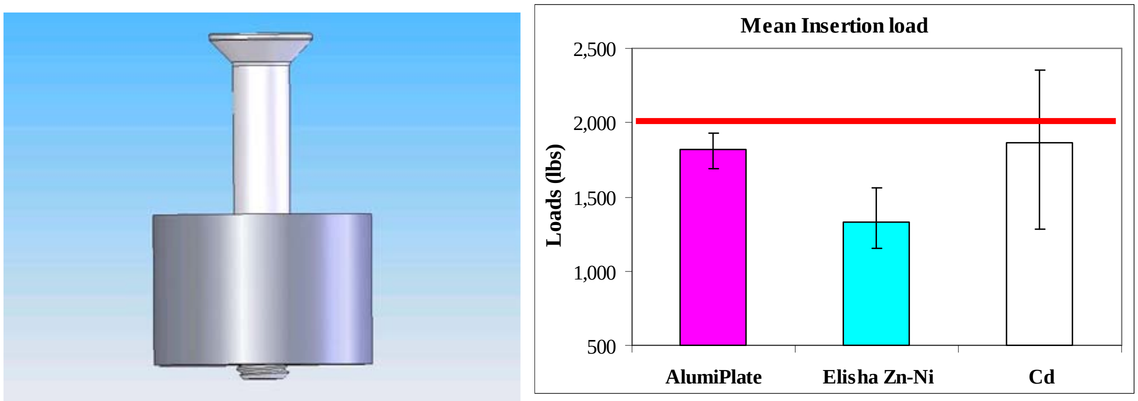
(b)

Figure 12: Mean (a) surface roughness and (b) static zero-load Friction coefficient for various coatings.

3.6 Insertion Testing

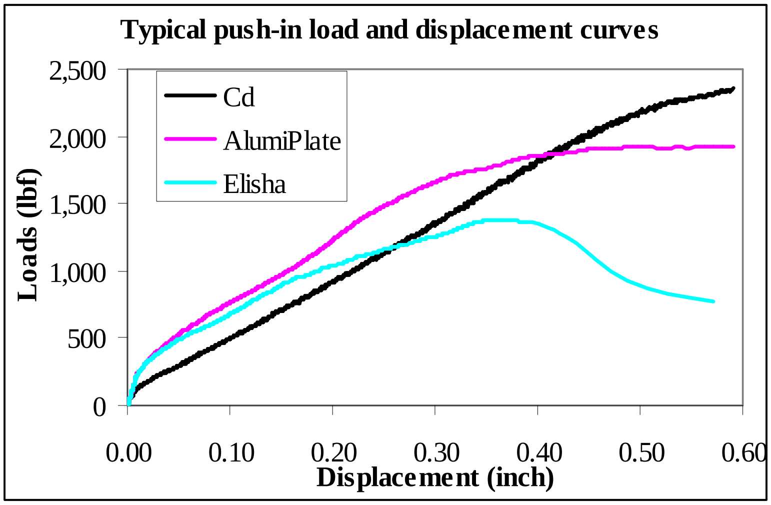
Fasteners were pushed into well-defined interference fit aluminum 2024-T351 holes with height 0.5 inch at a constant rate of 4000 pounds/min, and the required load and load–displacement curves measured (Figure 13). The interference level is 0.003-0.005 inch for all groups. The tests were monitored and recorded by a calibrated Digital Nicolet Oscilloscope Pro 40 model. The desired maximum installation force was less than 2000 pounds. All the coatings examined resulted in average insertion load less than the maximum load required (Figure 13).

Cd plated bolts demonstrated the highest push-in load among all coatings. However, only a few bolts have the overall push-in load slightly higher than 2000 pounds. Elisha Zn-Ni was the best overall performer. It had the least overall load to push plated fasteners into the interference hole and had the least variability from part to part. All Zn-Ni and AlumiPlate bolts were able to achieve full push in with less than 2000 pounds of load applied. In addition, weight measurements revealed that Zn-Ni had the minimum average weight change (Figure 14). Interestingly, a few Zn-Ni coated fasteners showed weight gain after the push-in test, indicating transfer of aluminum to fasteners. This is contrary to the Cd and AlumiPlate coated groups, showing consistent weight loss after the insertion tests (Figure 14(b)). In addition, the push-in load-displacement curves of Zn-Ni demonstrate different load profiles with the highest load occurring about 0.4 inch pushing distance (Figure 14(a)). This could be explained that after pushing 0.4 inch distance, the rest of the 0.1 inch height of aluminum was deformed or expanded, resulting in reduced resistance and load drop. Therefore, we can conclude that Elisha Zn-Ni coating with secondary lubrication demonstrated low dynamic pressure friction coefficient, dense structure, high strength of coating, and good bonding strength between coating and base metal

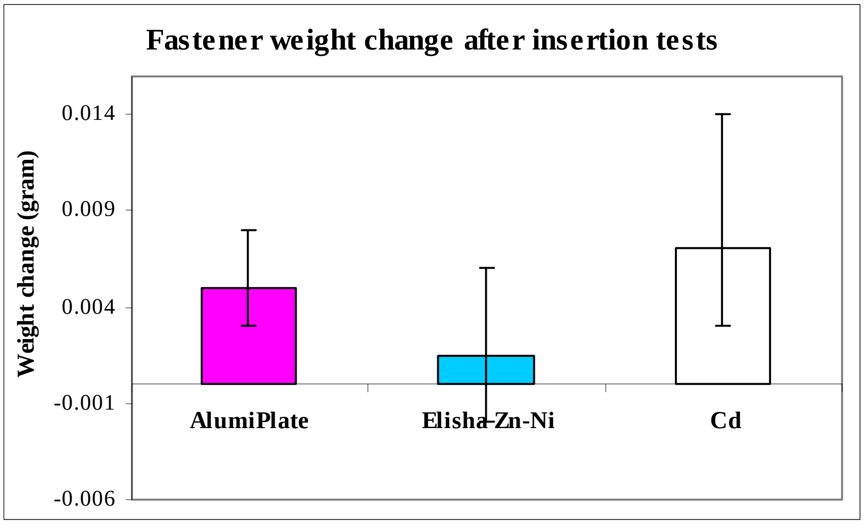


(a) (b)

Figure 13: Schematic representation of (a) push-in and interference test set up and (b) maximum push-in insertion load for various coatings.



(a)



(b)

Figure 14. Typical (a) push-in load and displacement curves and (b) fasteners weigh changes before and after insertion tests for various coatings.

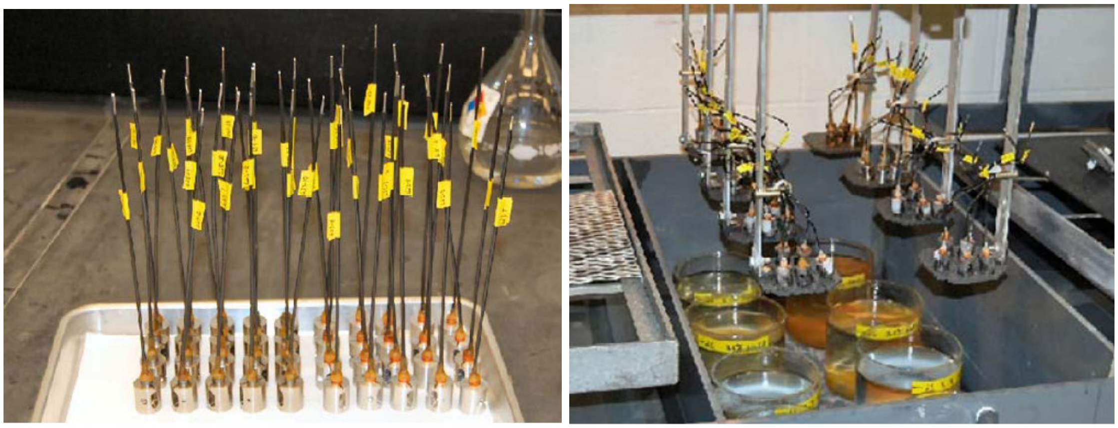
3.7 Corrosion resistance

3.7.1 Stress corrosion test (SCC)

SCC testing was performed on all coatings schemes per the method described in National Aerospace Standard NASM1312-9, Standard Practice, Fastener Test Methods, Section 9, Stress Corrosion. In this method, fasteners are threaded into a test collar and tightened. The stressed specimens were then exposed to an alternate immersion exposure of 3.5 weight % NaCl in deionized water. A separate container of solution was used for coating/collar combination. The alternate immersion cycle calls for a 10-minute immersion, followed by a 50-minute drying period. This 1-hour cycle is repeated continuously. Figure 15 illustrates the stressed samples with their lead-wire attached (a) and a set of replicate samples prepared for alternate immersion (b). The samples were exposed for a period of 60 days. Periodic visual examinations revealed no evidence of stress corrosion. It should be noted, however, that after several weeks, some general corrosion of the collars developed and the resultant corrosion product made it difficult to make visual examinations. Figure 16 provides low-magnification stereoscope images of representative samples at the end of the 60 day test. The presence of corrosion products from the collars is evident in each photograph. At the end of the 60 day test, all samples were examined in this manner, and no evidence of SCC was observed.

Two of the five replicates from each coating/collar set were disassembled by un-threading the fastener and removing it from the collar. The fasteners were cleaned of corrosion product from the collars as best as possible, using a stiff nylon brush and soap and water. This provided an opportunity for a better visual and low-magnification stereoscope examination. Again, no evidence of SCC was seen. One replicate of each coating/collar set was then optically cross-sectioned to determine if there was any evidence of incipient stress corrosion cracking. These micrographs were provided in Figure 17. No evidence of SCC was observed.

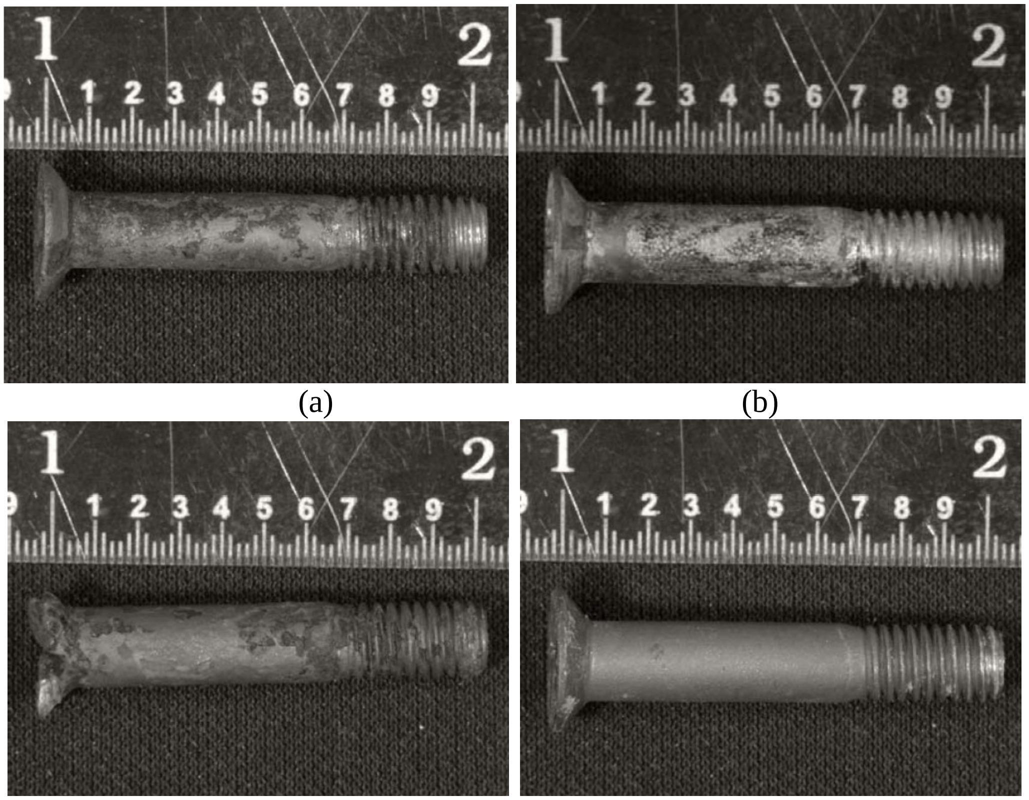
The corrosion potential of each fastener was periodically measured. The results for individual replicates were very consistent. Figure 18 provides the average value for each set of replicates, for each coating system and both collar materials. Also included in Figure 18, for reference, are the approximate ranges for corrosion potential for common aerospace aluminum alloys in the same environment. The initial obvious observation is the impact of the collar material. The stainless collars shift the potentials on order of ~200 mV in the anodic direction. This is as expected, given the galvanic potential of the stainless steel. The shift in potential, relative to an aluminum collar, is most likely unrealistic with respect to the in-service application. However, it is a conservative shift, since the more-positive potentials will tend to promote more corrosion of the fasteners in the steel collars, relative to those in the aluminum collars. A second observation is that for each set of collars, the Cd-plated fasteners are the most negative in potential, although all of the coatings reside in a relatively tight band of potential. It is difficult to interpret any significant differences in performance based on the potential values. For the more-realistic case of aluminum collars, all of the potentials are essentially in the same location, relative to the aluminum alloys used in aerospace. Hence, any galvanic impact of the fastener/air-frame couple should be similar to what is presently seen for Cd-plated fasteners.



(a) (b)

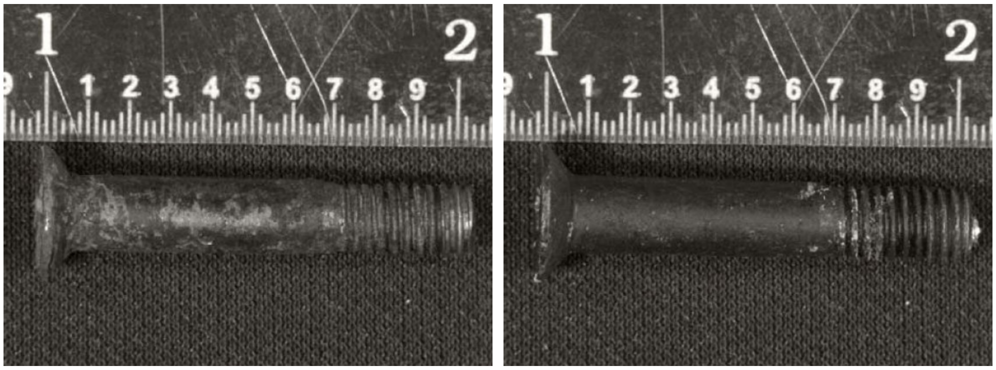
Figure 15 showed stressed Specimens prepared for test, with lead-wires attached for corrosion potential measurements (a) and stressed specimens in test in alternate Immersion (b).

Figure 16 shows typical SCC Specimen in a steel collar, after 60 days test.



(a) (b)

(c) (d)



(e) (f)

Figure 17 showed typical images of SCC testing with Cd coating in steel fixture (a) and aluminum fixture (b), AlumiPlate coating in steel fixture (c) and aluminum fixture (d), and Elisha coating in steel fixture (e) and aluminum fixture (f).

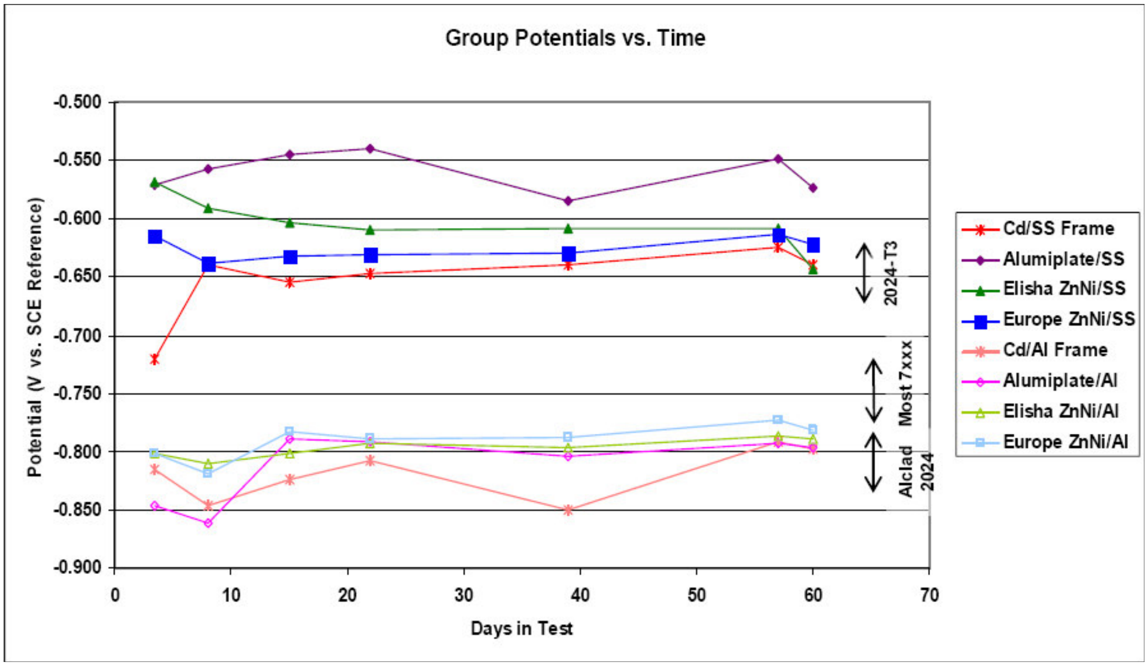


Figure18 showed corrosion potentials as measured during the SCC Testing.

3.7.2 Salt fog test

Five replicate samples of each coating system were exposed to a continuous Salt Spray exposure, per ASTM B117, “Standard Practice for Operating Salt Spray (Fog) Apparatus”. This is a continuous exposure to a spray/fog of 5 weight % NaCl in deionized water, at a temperature of 95°F ± 3°F. The fasteners were unstressed; i.e., no collars. Exposure period was 1000 hours. Similar to the SCC samples, the corrosion potential was measured on a weekly basis. The samples were immersed in a beaker of 5% NaCl in order to make the corrosion potential measurement. Figure 19 illustrates the appearance of the fasteners after 1,000 hours of salt spray. All replicates looked essentially identical. In general, all of the coated fasteners looked very good. While some showed mild staining and discoloration (not uncommon after salt spray exposure), none of them exhibited any evidence of a “structural” corrosion behavior, e.g., in the form of pits or cracks.

Fig 20 illustrates the corrosion potentials of each coating system, taken periodically during the salt spray exposure. In general, with the exception of Elisha Zn-Ni, the coating systems all exhibit essentially an identical corrosion potential. The reason for the difference on the Elisha Zn-Ni may be related to the surface silicate layer due to surface mineralization. Nonetheless, even though it is slightly more positive, its relative placement, with respect to the bare steel and the aluminum aerospace alloys, is similar to the other coatings. Certainly, this difference in potential is not a cause for concern, or a basis to consider the Elisha Zn-Ni as a poor candidate. For all coatings, there is a significant potential difference relative to the bare steel fastener. This indicates that all coating materials will serve well in their role as sacrificial coatings, geared to protect the steel. Also, none of the coatings has a potential that is markedly more-negative than the Cd-plated fasteners. A markedly more-negative potential, and hence a much larger potential difference with the bare steel, could indicate a concern for increased risk of hydrogen embrittlement of the steel fastener. Given none are markedly more negative than the Cd-plating, there should be no increased concern for hydrogen embrittlement.

Figure 19 illustrates the appearance of the typical fasteners after 1,000 hours of salt spray test.

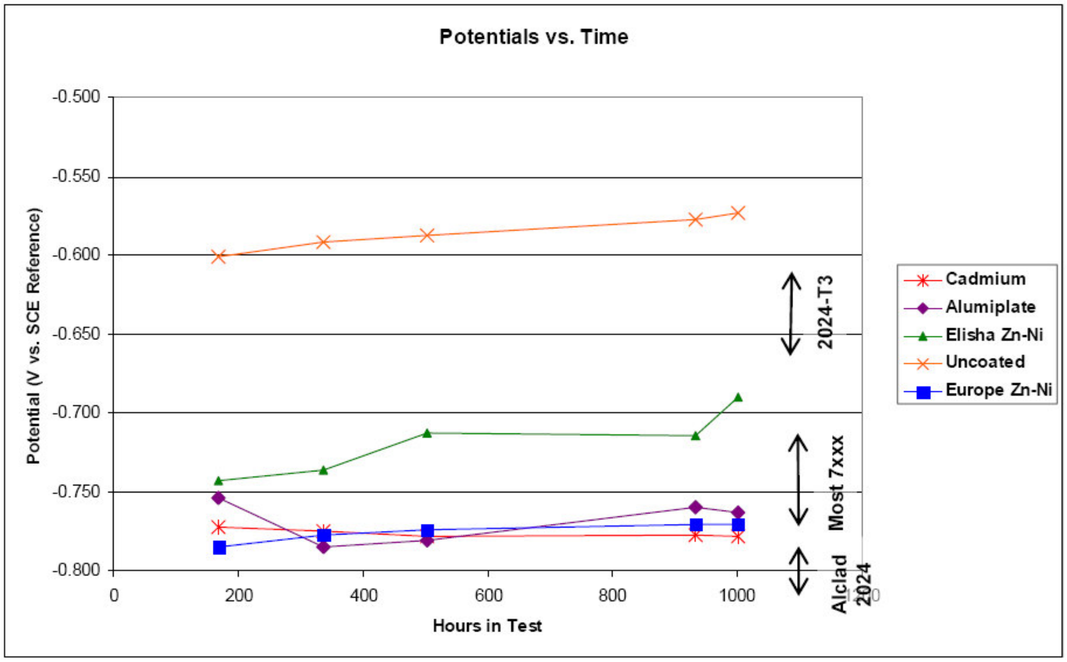


Figure 20 shows corrosion potentials as measured during the salt spray testing**.**