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IMPROVED LHE ZN-NI AND CD PLATING PROCESS PHASE II: FINAL REPORT

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ACRONYMS ABBREVIATIONS

ASF	Amps per Square Foot
BR&T	Boeing Research & Technology
Cd	Cadmium
HAFB	Hill Air Force Base
HE	Hydrogen Embrittlement
HSS	High Strength Steel
IAW	In Accordance With
LHE	Low Hydrogen Embrittlement
Ni	Nickel
NFS	Notch Fracture Strength
SBIR	Small Business Innovation Research
TDS	Thermal Desorption Spectroscopy
UTS	Ultimate Tensile Strength
USAF	United States Air Force
Zn	Zinc
Zn-Ni	Zinc-Nickel

REFERENCES

1. ASTM F519: Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments
2. ASTM F1624 (10/5/1,2): Standard test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Load Technique
3. MIL-STD-870: Cadmium Plating, Low Embrittlement Electrodeposition
4. USAF-DWG-20102456: Low Hydrogen Embrittlement Plating Process Specification Zinc-Nickel

1 Background

Low Hydrogen Embrittlement (LHE) cadmium (Cd) and LHE zinc-nickel (Zn-Ni) are currently used as sacrificial coatings on high strength steel (HSS) aircraft landing gear components to prevent corrosion. During the SBIR Phase II project, *Development of Cd Plating Replacement on High Strength Steel*, that qualified LHE Zn-Ni as a replacement for Cd, it was recognized that the time required to bake hydrogen out of the electroplated component may be reduced. The 309 CMXG Production Group is the single largest producer of electroplated components in the USAF, therefore a reduction in the hydrogen embrittlement (HE) relief bake time could reduce energy consumption, costs, and time to repair and overhaul aircraft components.

Current LHE Cd (MIL-STD-870) and LHE Zn-Ni (USAF DWG 201027456) plating processes require that components be baked at 375°F ± 25°F for a minimum time of 23 hours. The 417 SCMS/GUEA Landing Gear Engineering Group is responsible for ~95% of all USAF landing gear components. This equates to an estimated 1000+ components undergo the required 23 hour HE relief bake annually.

2 Scope

This SBIR Phase II project determined the feasibility of reducing the HE relief bake time by sustained load testing, incremental step load (ISL) testing, thermal desorption spectroscopy (TDS) testing, and the LECO test. A reduction in the HE relief bake time will ultimately lead to significant savings in time, energy, and cost during the overhaul process of landing gear components.

3 Objective

The overall objective of this SBIR Phase II project was to reduce the HE relief bake time requirement after the electroplating process. This objective was met by performing the sustained load, ISL, TDS and LECO tests mentioned above. Test subjects were prepared IAW their respective standards, drawings, and ASTM F519: *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments.*

To achieve the desired results, the measurement in the reduction of time required to pass accepted hydrogen embrittlement tests was correlated to the amount of hydrogen that was in the coupon which was measured by TDS, and LECO.

4 Procedures

HE testing due to electrodeposition is outlined in MIL-STD-870 (Cd) and USAF DWG 201027456 (Zn-Ni). Each respective standard outlines the criteria for pass/failure of the ASTM F519 embrittlement test. Testing used the ASTM F519 Type 1a.1 coupon (4340 and 300M) for hydrogen embrittlement testing. A 1.3” x 1.3” square block with variable thickness made from 300M steel heat treated to 280-300 ksi UTS (Figure 1) was used for hydrogen content bakeout.

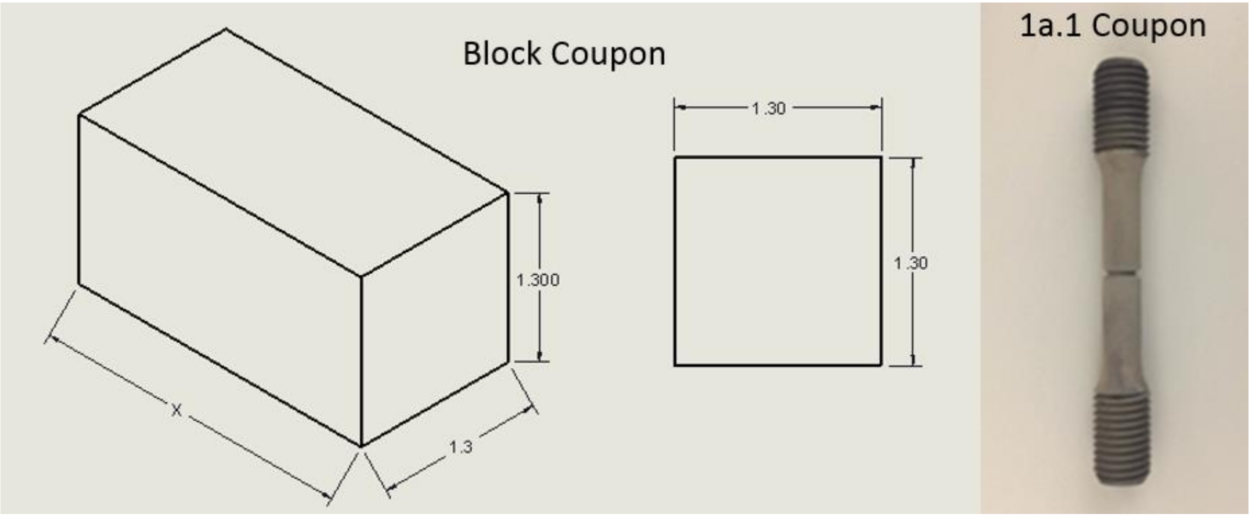

Figure 1: ASTM F519 1a.1 (Right) and Thick Block (Left) Test Coupons

Square block coupons were plated with Electroless Ni (E-Ni) at ES3’s facilities before they were shipped to McGill University for LHE Zn-Ni plating and TDS testing.

4.1 Coupon Preparation

Coupons were tested in various conditions to quantify the required bake time and amount of hydrogen in the coupons. The coupons were tested bare (without any plating), standard plating practices (23 hr bake) and varying bake times. This was designed to validate introduction of hydrogen from the plating process as well as reduction of hydrogen through the baking process. The plated coupons were subjected to the most embrittling parameters that may be seen while plating during normal plating processes. The current density was adjusted to the low end of the respective specifications and the plating thickness was increased to the high end of the spectrum as shown in the following plating procedures. The bake temperature was also reduced to the low end of the acceptable bake spectrum of 355°F ±5°F.

4.1.1 LHE Zn-Ni Plating Procedure

LHE Zn-Ni electroplated coupons were prepared IAW USAF DWG 201027456 Section 5.5.5 Qualification embrittlement test except for coupon type and bakes times, which were adjusted to study the impact of reducing the 23-hour bake time.

The following plating procedure for LHE Zn-Ni was followed.

a. ASTM F519 Type 1a.1(4340 vacuum melt and 300M steel) or 1.3” x 1.3” square block (300M) test specimen were prepared.
b. Specimen were prepared for and plated in accordance with the requirements of the standard. During plating, the specimen was mounted symmetrically on a rack by themselves. All areas of the rack except the contact area was coated with a suitable maskant. An ammeter having a sensitivity of 0.5 amperes or better was connected between the specimen rack and the cathode. The specimen was plated at 40 ASF to a thickness of 0.8-1.0 mil (0.0008 - 0.001 in.).
c. The specimen underwent, sustained load, ISL, TDS, or LECO testing.
d. Test specimen were loaded onto the respective test frame within one hour of baking or plating, except for the “no bake” test subjects for the sustained load testing.

4.1.2 Cd Plating Procedure

LHE Cd electroplated coupons were prepared IAW MIL-STD-870 Section 5.7 Qualification embrittlement test except for the coupon type and bake times, which were adjusted to study the impact of reducing the 23-hour bake.

The following plating procedure for Cd was followed:

a. ASTM F519 Type 1a.1(4340 vacuum melt and 300M steel) or 1.3” x 1.3” square block (300M) test specimen were prepared.
b. Specimen were prepared for and plated in accordance with the requirements of the standard. During plating, the specimen was mounted symmetrically on a rack by themselves. All areas of the rack except the contact area was coated with a suitable maskant. An ammeter having a sensitivity of 0.5 amperes or better was connected between the specimen rack and the cathode. The specimen was plated at 50 ASF to a thickness of 0.8-1.0 mil (0.0008 - 0.001 in.).
c. The specimen underwent, sustained load, ISL, TDS, or LECO testing.
d. Test specimen were loaded onto the respective test frame within one hour of baking or plating, except for the “no bake” test subjects for the sustained load testing.

4.1.3 Electroless Nickel Plating Procedure

Four sides of the block coupons were E-Ni plated, and afterwards the remaining two sides of the block coupon were LHE Zn-Ni plated. Figure 2 below shows the LHE Zn-Ni and E-Ni plated areas. Medium phosphorus (5-7%) E-Ni plating was completed to a thickness of approximately 0.0005”. This provides a barrier to hydrogen evolution out of the nickel plated sides forcing the flow of hydrogen out of the Cd or LHE Zn-Ni plated ends.

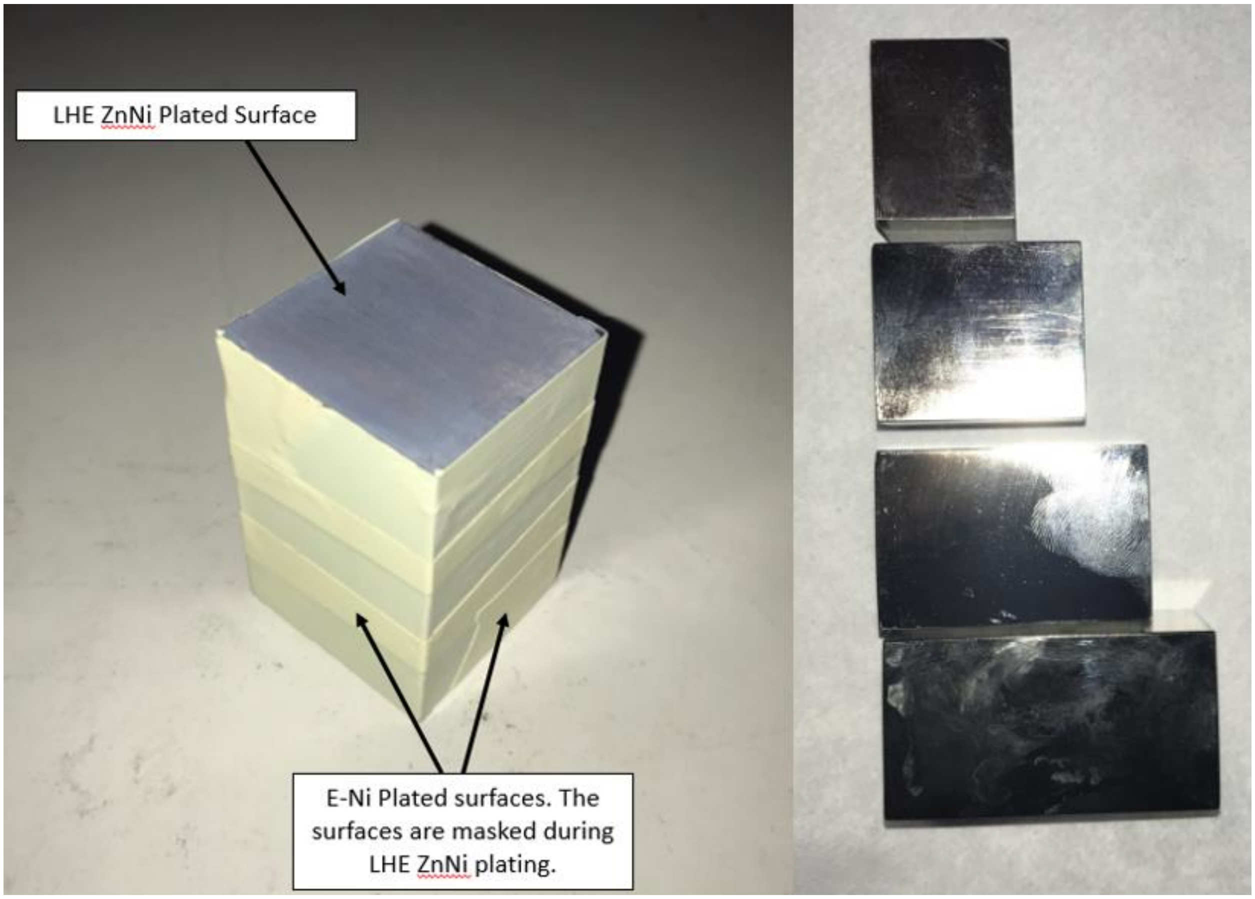

Figure 2: LHE Zn-Ni and E-Ni Plated Areas for Block Test Coupons (Left) and E-Ni Plated Areas (Right)

4.2 Testing Procedure

4.2.1 Sustained Load Testing

Sustained load testing was completed IAW ASTM F519: *Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments.* Testing consisted of 4 Type 1a.1 coupons being baked for the reduced times and then loaded to

75% NFS for 200 hours. Pass criteria states that if all 4 coupons withstand the 200-hour sustained load, the test is passed. If 1 of the test coupons break, the remaining three coupons must complete the 200-hour sustained load. After completion of the 200-hour sustained load, the coupons are then step loaded by 5% every 2 hours, up to 90% NFS. If all 3 remaining coupons withstand the load of 90% NFS for 2 hours, the test is considered passed.

Sustained load testing and plating was completed at HAFB and ES3 facilities.

4.2.2 Incremental Step Load Testing

ISL testing was completed in accordance with ASTM F1624 (10/5/1,2). Testing consists of 3 coupons loaded incrementally by 5% NFS every hour for 10 steps (50% NFS is reached). The coupons are then incrementally loaded by 5% NFS every two hours until fracture occurs. Coupons that can sustain 90% NFS for two hours are non-embrittled. All three coupons must reach 90% NFS for two hours to pass.

ISL testing and plating was completed at McGill University, and Boeing Research and Technology (BR&T) St. Louis.

4.2.3 Thermal Desorption Spectroscopy Testing

TDS testing consists of placing the plated material into a vacuum chamber and then heating the material. By heating the material, hydrogen molecules that have adsorbed to the surface are released. TDS quantifies the amount of hydrogen molecules released during testing. Un-plated coupons, plated and unbaked, as well as plated coupons that have been through the standard 23-hour bake were used to find a baseline of hydrogen concentration. The amount of hydrogen released on the test coupons was compared to the baseline.

TDS testing and plating was completed at McGill University.

4.2.4 LECO Testing

LECO testing is performed by cutting a small piece of material out of a coupon. This piece is then melted and the amount of hydrogen that was in the piece is measured to determine the total hydrogen in the coupon. The test is completely destructive of the base material. All hydrogen is released from the coupon in this case including bonded hydrogen and hydrogen that is in trap sites in the material. This testing was completed by BR&T St. Louis.

5 Results and Discussion

5.1 Sustained Load Results

ASTM F519 type 1a.1 coupons were LHE Zn-Ni or Cd plated and baked for the reduced times outlined in Table 1 below. Testing stopped when a bake time produced a passing test result. These results show that for test coupons that a reduction in bake time is feasible.

Figure 3: Sustained Load Test Frame

Table 1: HAFB Sustained Load Test Matrix and Results

Bake Time	Bake Temperature	HSS	Plating Type	Coupon Geometry	# of Coupons	Pass/Fail
23 hr. std.	355 ± 5 ˚F	4340	Cd	1a.1	4	Pass
No Bake	355 ± 5 ˚F	4340	Cd	1a.1	4	Fail
15 min	355 ± 5 ˚F	4340	Cd	1a.1	4	Pass
23 hr. std.	355 ± 5 ˚F	300M	Cd	1a.1	4	Pass
No Bake	355 ± 5 ˚F	300M	Cd	1a.1	4	Fail
15 min	355 ± 5 ˚F	300M	Cd	1a.1	4	Fail
30 min	355 ± 5 ˚F	300M	Cd	1a.1	4	Pass
23 hr. std.	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Pass
No Bake	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Fail
15 min	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Fail
30 min	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Pass
23 hr. std.	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Pass
No Bake	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Fail
15 min	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Fail
30 min	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Pass

These tests were performed to simulate the most embrittling parameters within the specification allowances. Under these conditions the coupons were able to pass using bake times ranging from 15 to 30 minutes.

Increasing the temperature of the embrittlement relief bake decreases the amount of time required to ensure that the coupons have been sufficiently processed to remove hydrogen. The following data shows that baking at higher temperatures allows for a coupon to pass a test where it had failed in the previous test at the lower bake temperature. The initial testing was performed at 355 ± 5 ˚F the following data is performed at 395 ± 5 ˚F.

Table 2: Higher Temperature Bake

Bake Time	Bake Temperature	HSS	Plating Type	Coupon Geometry	# of Coupons	Pass/Fail
355˚F Pass **(15 min)**	395 ± 5 ˚F	4340	Cd	1a.1	4	Pass
355˚F Pass **(30 min)**	395 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Pass
355˚F Fail **(15 min)**	395 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	4	Pass
355˚F Pass **(30 min)**	395 ± 5 ˚F	300M	Cd	1a.1	4	Pass
355˚F Fail **(15 min)**_	395 ± 5 ˚F	300M	Cd	1a.1	4	Pass
355˚F Pass **(30 min)**	395 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Pass
355˚F Fail __(**15 min)**_	395 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	4	Pass

This testing showed that an increase in bake temperature allowed coupons that had previously failed the HE testing can had the hydrogen baked out and pass testing in a reduced bake time.

5.1.1 Sustained Load Conclusions

Coupon that were tested using the 200-hr sustained load testing results show that LHE Zn-Ni and Cd electroplated test coupons, both 300M and 4340 HSS, can pass the 200-hr sustained load test with reduced bake times. Reducing the HE bake time to 30 minutes for all test scenarios.

5.2 Incremental Step Load Results

ASTM F519 type 1a.1 coupons were LHE Zn-Ni or Cd plated and baked for the reduced times outlined in Table 3 - Table 4 below.

Table 3: BR&T ISL Test Matrix and Results

Bake Time	Bake Temperature	HSS	Plating Type	Coupon Geometry	Notch Fracture Strength	Pass/Fail
23 hr. std.	355 ± 5 ˚F	4340	Cd	1a.1	100%	Pass
No Bake	-	4340	Cd	1a.a	55%	Fail
15 min	355 ± 5 ˚F	4340	Cd	1a.1	66%	Fail
30 min	355 ± 5 ˚F	4340	Cd	1a.1	95%	Pass
No Bake	355 ± 5 ˚F	300M	Cd	1a.1	50%	
15 min	355 ± 5 ˚F	300M	Cd	1a.1	40%	Fail
30 min	355 ± 5 ˚F	300M	Cd	1a.1	50%	Fail
1 hr.	355 ± 5 ˚F	300M	Cd	1a.1	60%	Fail
2 hr.	355 ± 5 ˚F	300M	Cd	1a.1	50%	Fail
3 hr.	355 ± 5 ˚F	300M	Cd	1a.1	95%	Pass
No Bake	355 ± 5 ˚F	4340	Zn-Ni	1a.1	30%	Fail
15 min	355 ± 5 ˚F	4340	Zn-Ni	1a.1	45%	Fail
30 min	355 ± 5 ˚F	4340	Zn-Ni	1a.1	85%	Fail
1 hr	355 ± 5 ˚F	4340	Zn-Ni	1a.1	90%	Pass
15 min	355 ± 5 ˚F	300M	Zn-Ni	1a.1	55%	Fail
30 min	355 ± 5 ˚F	300M	Zn-Ni	1a.1	55%	Fail
30 min	355 ± 5 ˚F	300M	Zn-Ni	1a.1	90%	Pass
1 hr	355 ± 5 ˚F	300M	Zn-Ni	1a.1	70%	Fail
1 hr	355 ± 5 ˚F	300M	Zn-Ni	1a.1	90%	Pass
2 hr	355 ± 5 ˚F	300M	Zn-Ni	1a.1	90%	Pass

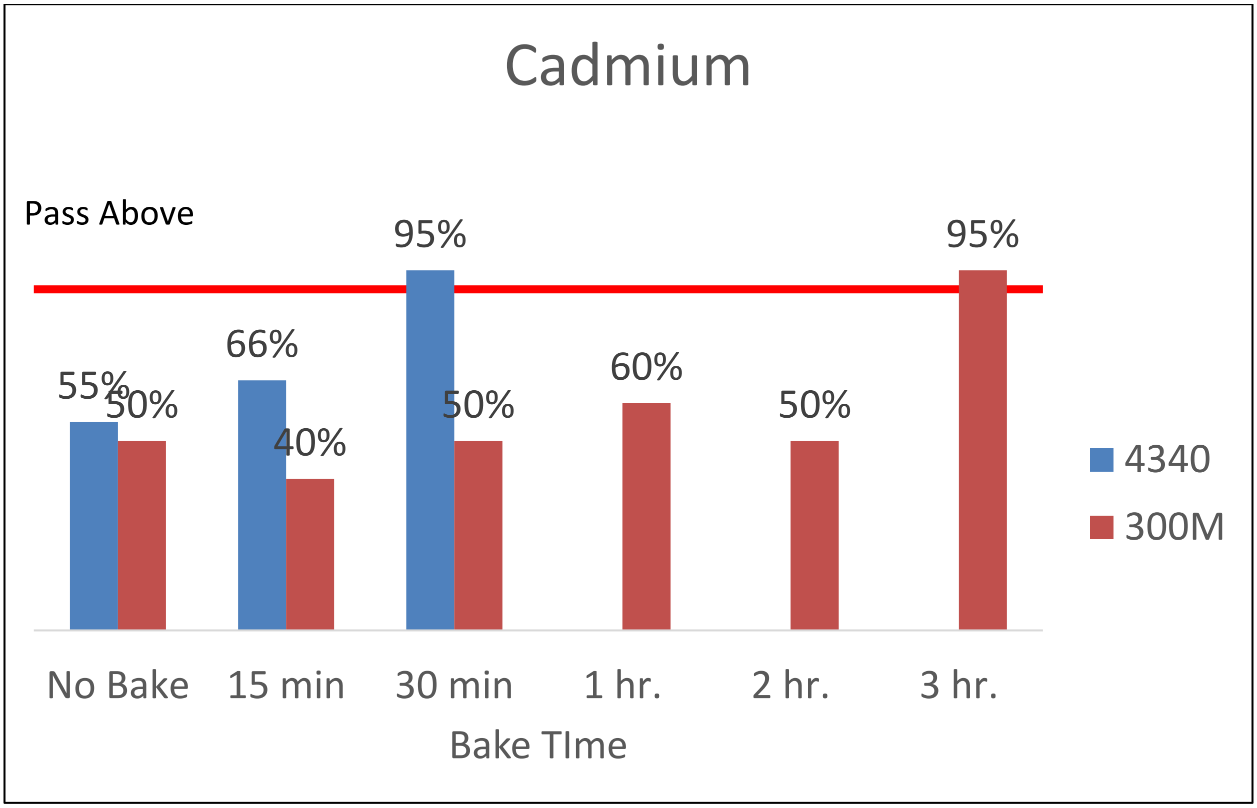

Figure 4: Cadmium SL Results - Testing Discontinued After a Pass

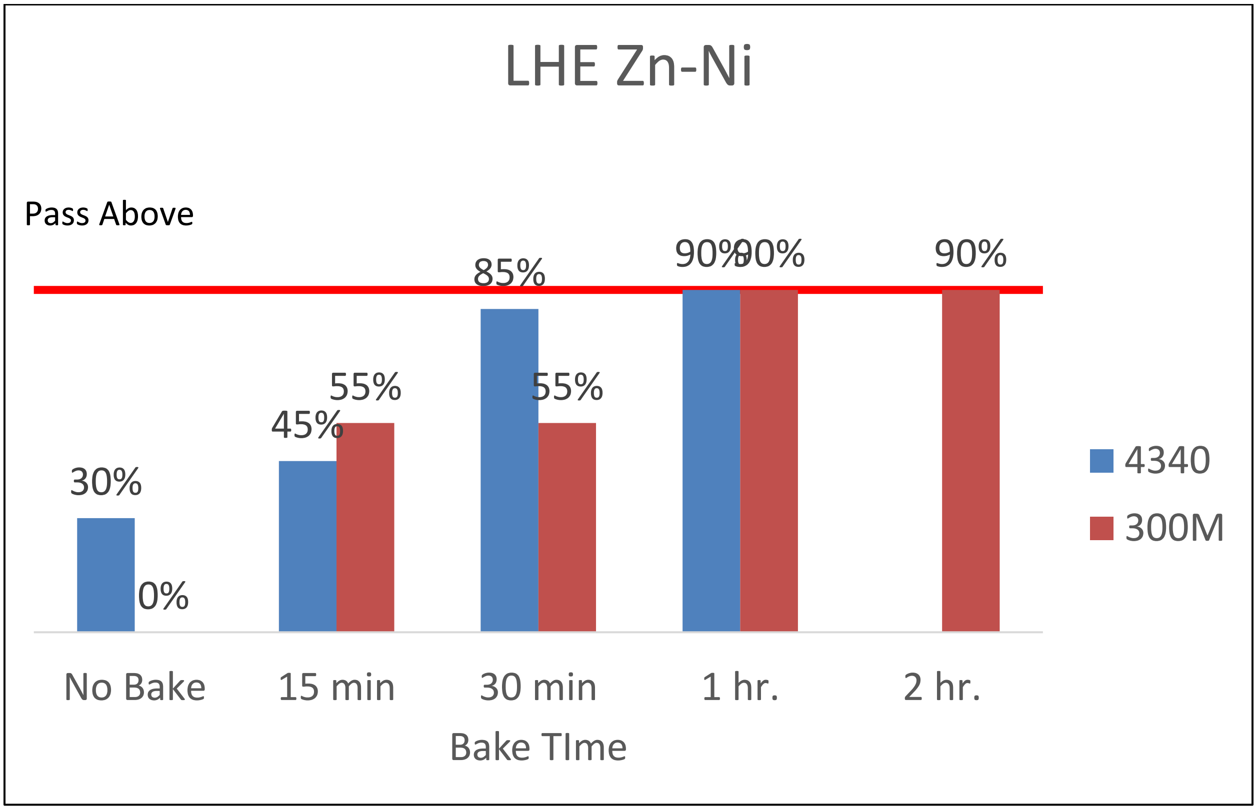

Figure 5: LHE Zn-Ni SL Results - Testing Discontinued After a Pass

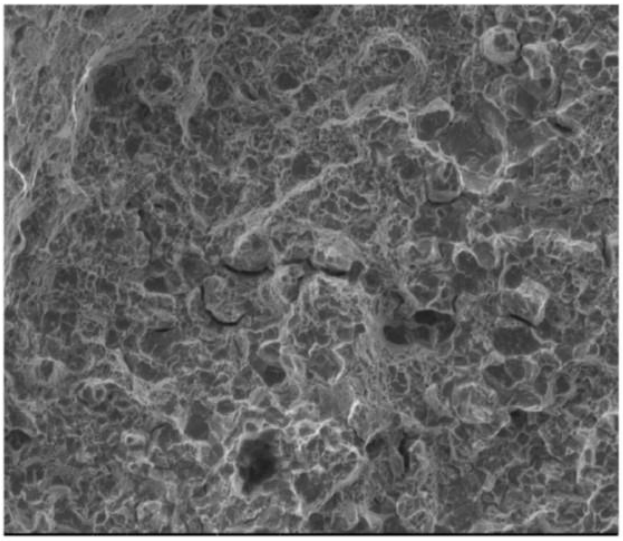

Figure 6: 4340 Cd - No Bake Brittle Fracture

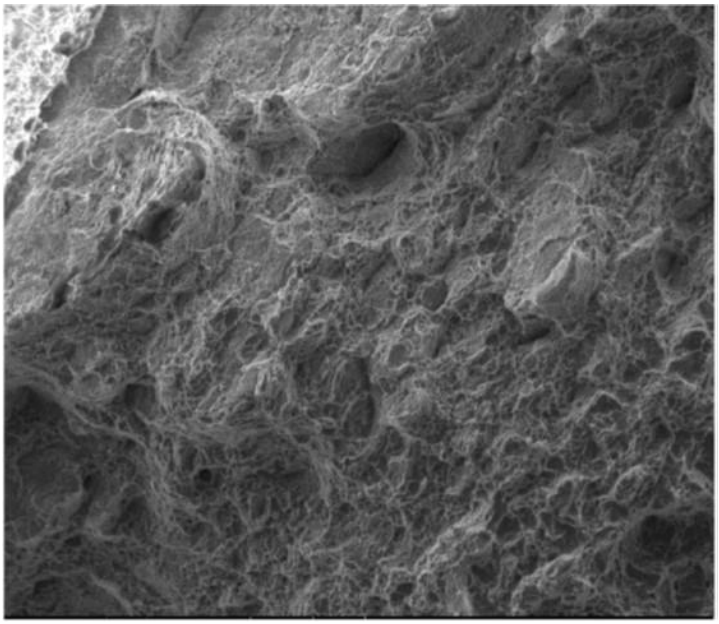

Figure 7: 4340 Cd - 30 min Bake Ductile Fracture

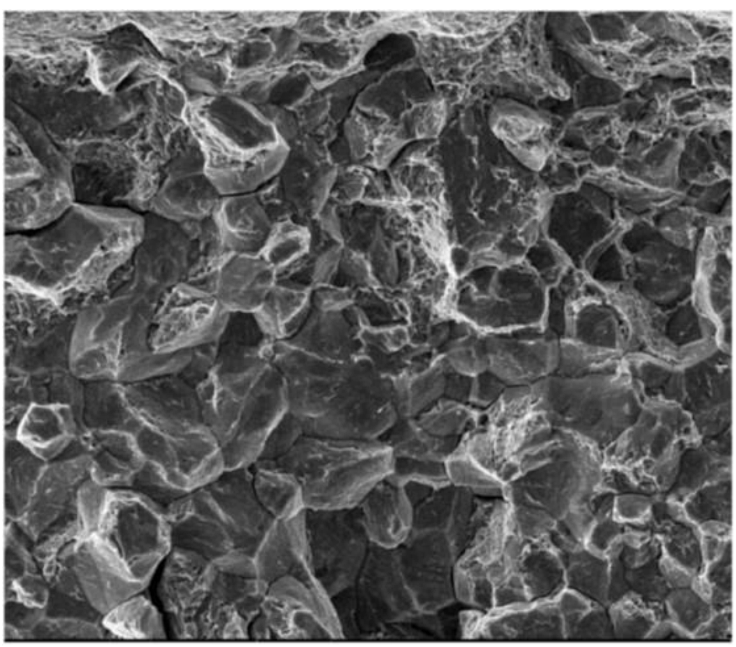

Figure 8: 4340 LHE Zn-Ni - 15 min Bake Brittle Fracture

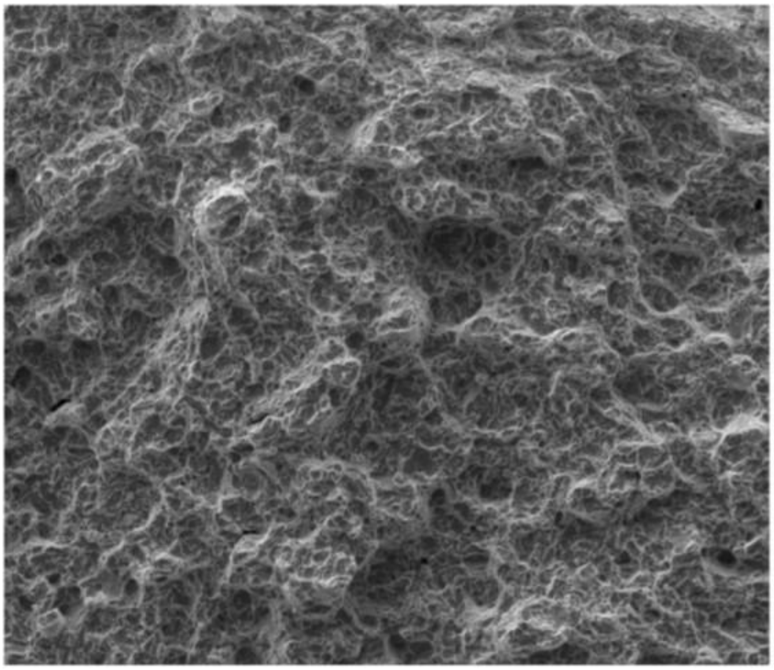

Figure 9: 4340 Cd - 1 hr Bake Ductile Fracture

ISL test specimens that failed the ISL test (< 90% NFS) showed signs predominantly of brittle fracture, and ISL test specimens that passed the ISL test ($\geq$ 90% NFS) showed signs of predominantly ductile fracture

Table 4: McGill University ISL Test Matrix and Results

Bake Time	Bake Temperature	HSS	Plating Type	Coupon Geometry	Notch Fracture Strength	Pass/Fail
No Bake	355 ± 5 ˚F	4340	Cd	1a.1	45%	Fail
15 min	355 ± 5 ˚F	4340	Cd	1a.1	93%	Pass
30 min	355 ± 5 ˚F	4340	Cd	1a.1	93%	Pass
No Bake	355 ± 5 ˚F	300M	Cd	1a.1	95%	Pass

No Bake	-	4340	LHE Zn-Ni	1a.1	49%	Fail
15 min	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	71%	Fail
30 min	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	94%	Pass
23 hr. std.	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	102%	Pass
No Bake .	-	300M	LHE Zn-Ni	1a.1	57%	Fail
15 min	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	87%	Fail
30 min	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	100%	Pass

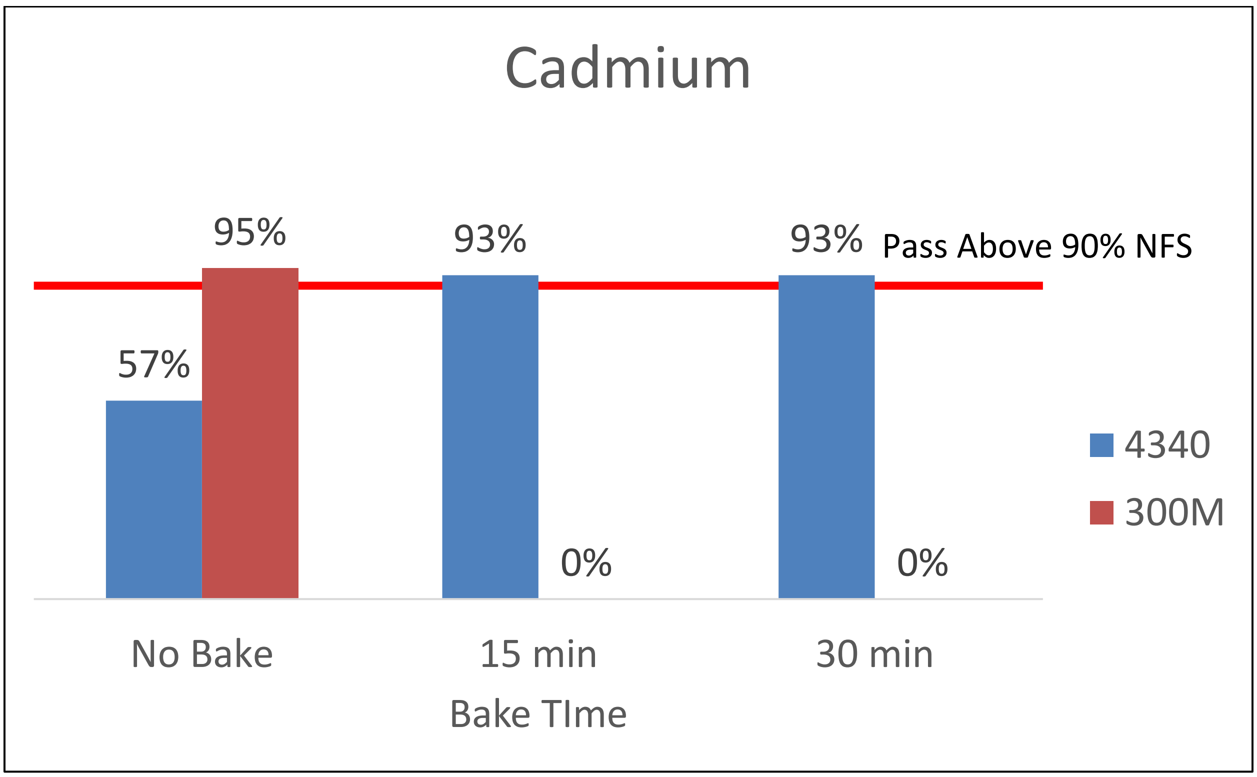

Figure 10: Cadmium ISL McGill Results - Testing Discontinued After a Pass

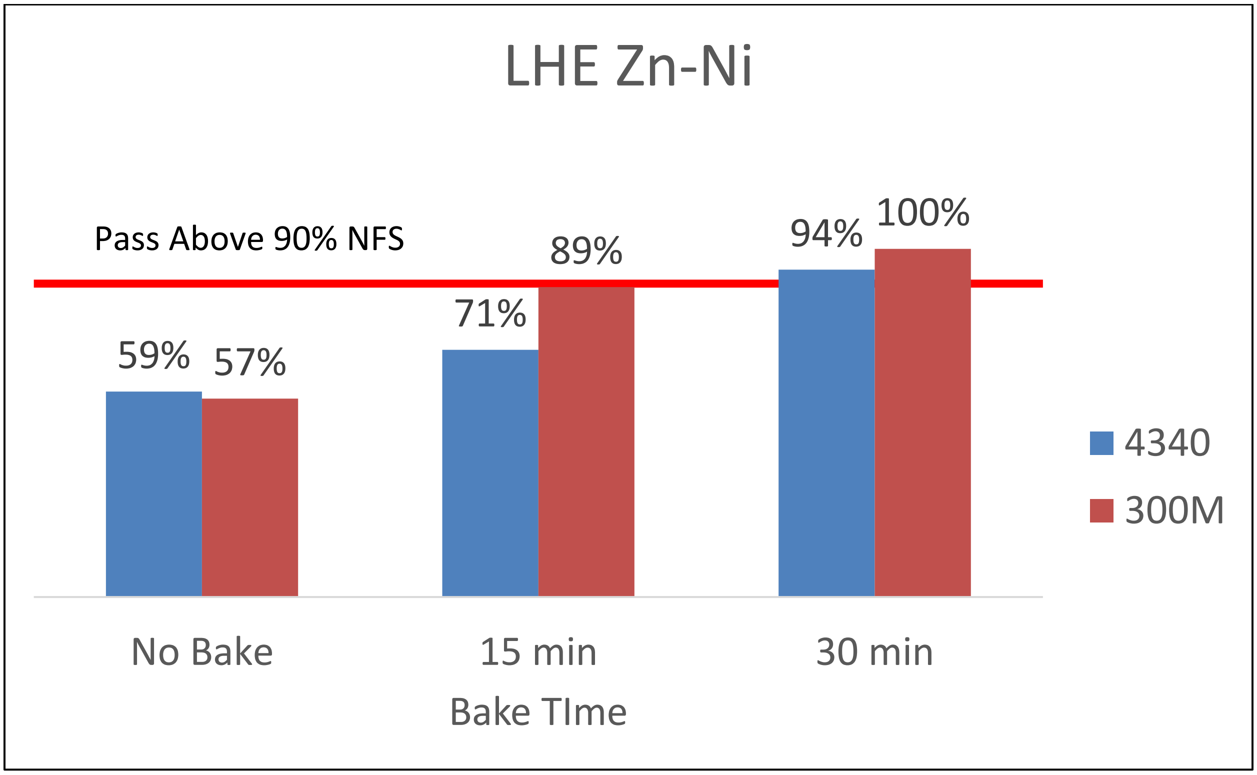

Figure 11: LHE Zn-Ni ISL McGill Results - Testing Discontinued After a Pass

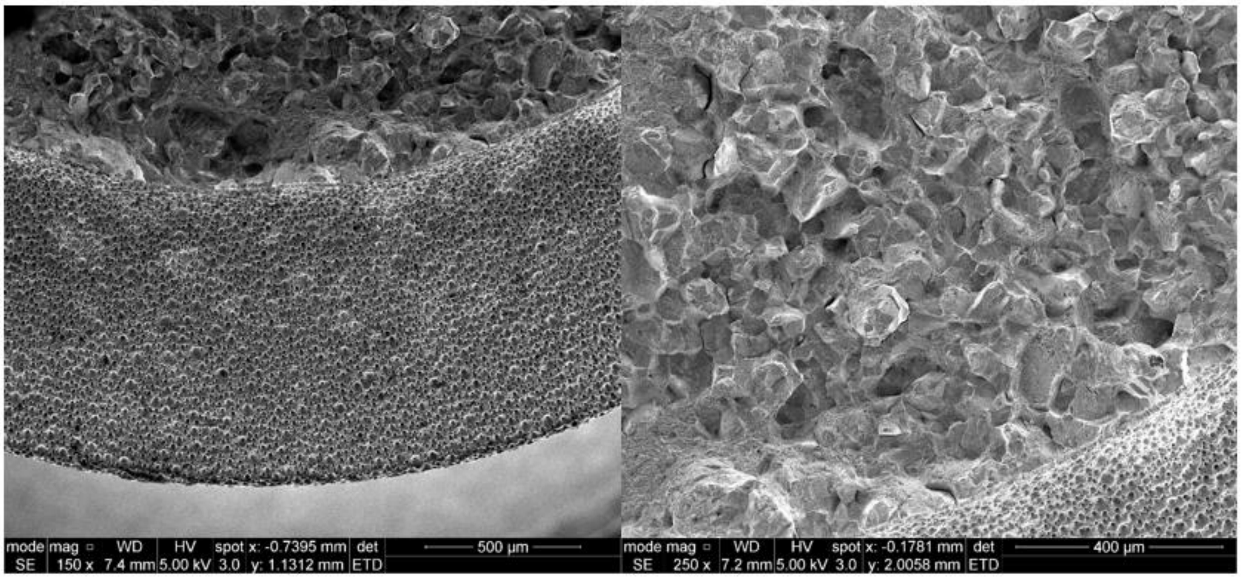

Figure 12: 300M ISL fracture location – No Bake

5.2.1 Incremental Step Load Conclusions

ISL testing showed that Cd and LHE Zn-Ni plated coupons that underwent reduced bake times passed at significantly lower bake times than the currently required 23 hour bake. The two locations that performed the ISL testing had varying results when compared with each other. However, there was agreement in the data that a significant reduction in bake time can be achieved.

5.3 Thermal Desorption Spectroscopy 1a.1 Coupon Results

Bare coupons underwent the TDS test to measure the amount of hydrogen (if any) released from 4340 vacuum melt and 300M steel during testing. A group of LHE Zn-Ni and Cd coupons were plated and underwent TDS testing without the HE relief bake and again with the standard 23-hour relief bake to set a baseline. The amount of hydrogen released from coupons baked for reduced times was compared to the baseline.

Coupons that were not plated were measured using TDS to have a baseline reference to the hydrogen that is naturally in the steel. Comparisons of the plated and baked coupons can be made to identify the effectiveness of the bake process to reduce the hydrogen content to levels that are found in the steel at its reference state.

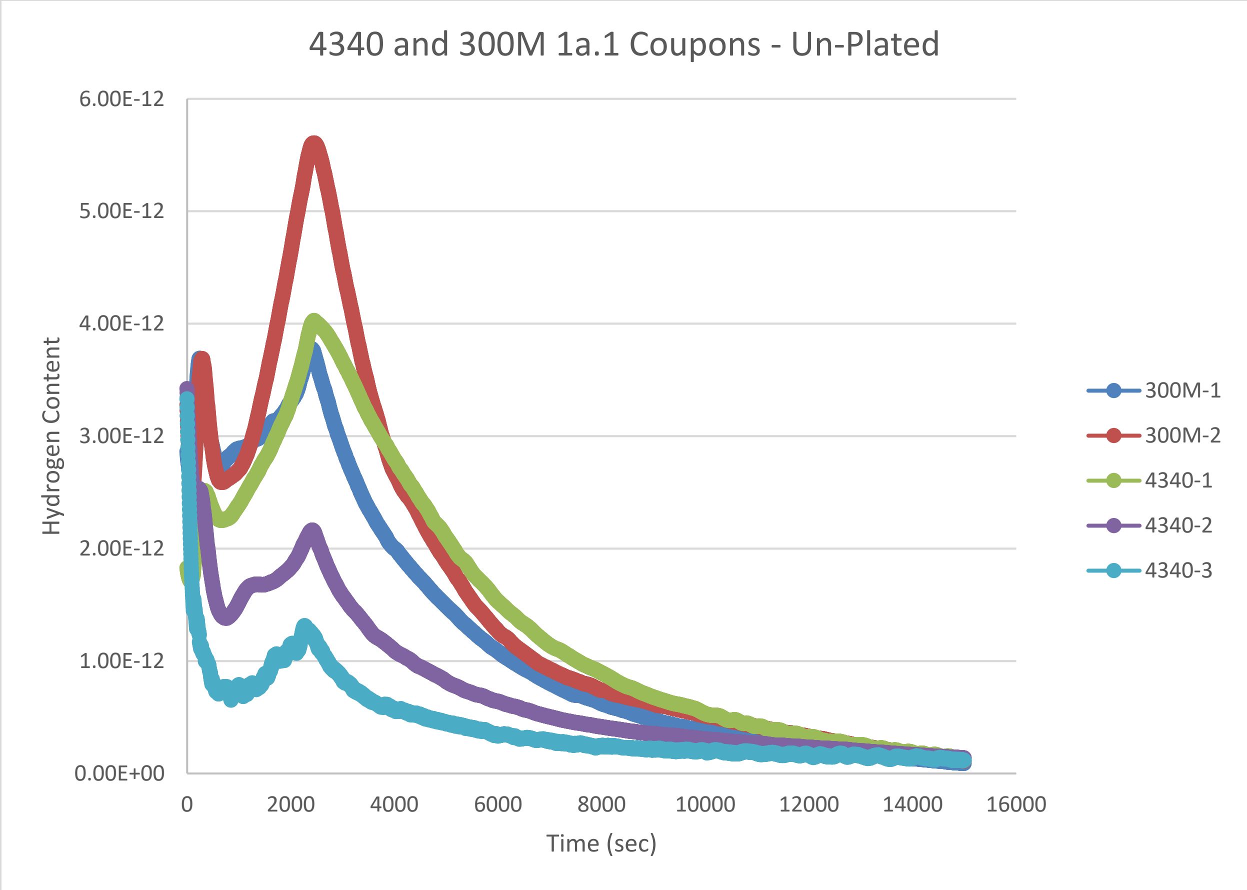

Figure 13: Un-Plated Steel Coupons

The following graphs show three specimens in which the hydrogen content was measured after Cd plating on 4340 steel. There is variation between the amount of hydrogen in each of the three coupons processed under the same parameters.

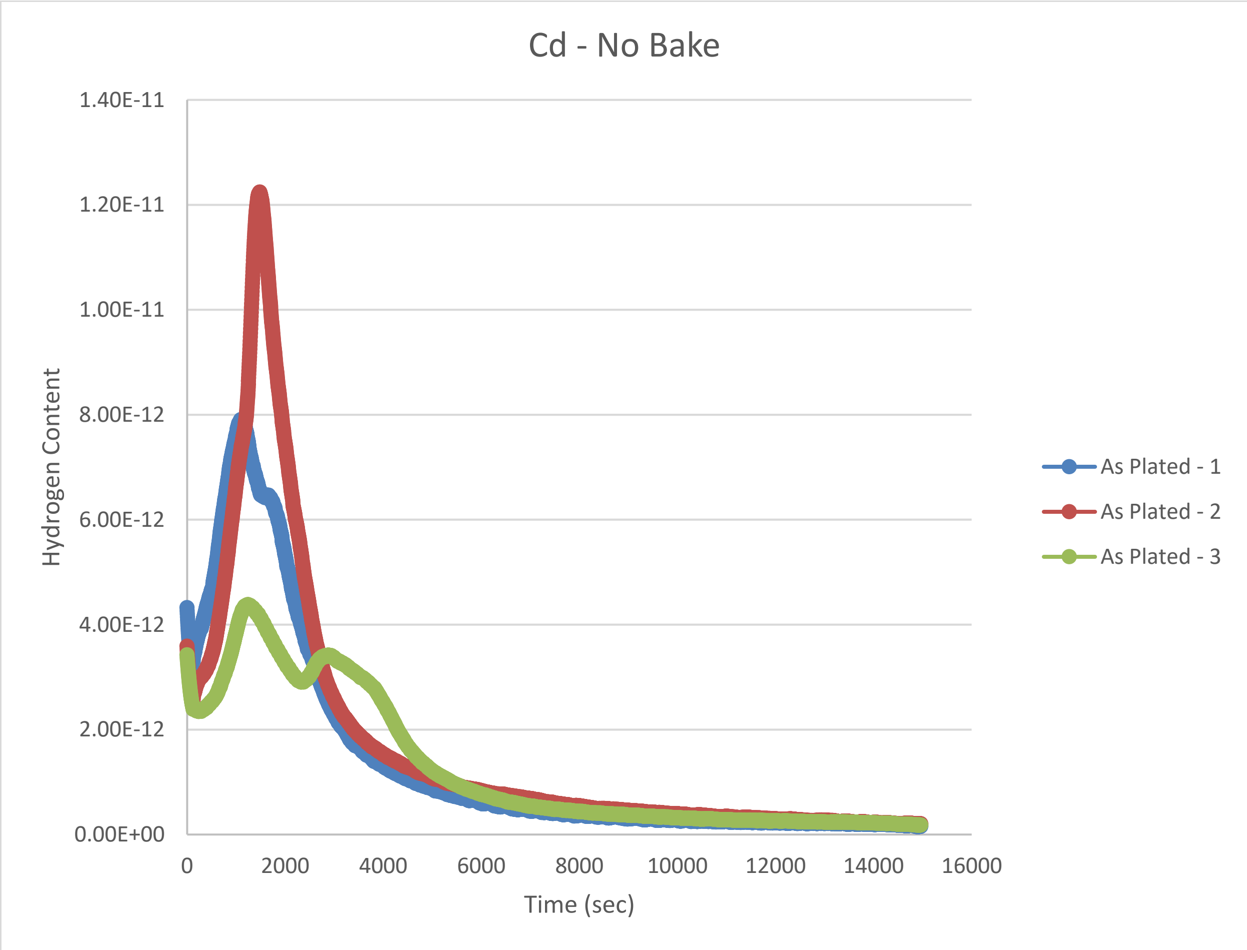

Figure 14: Cd Plated Coupons - No Baking

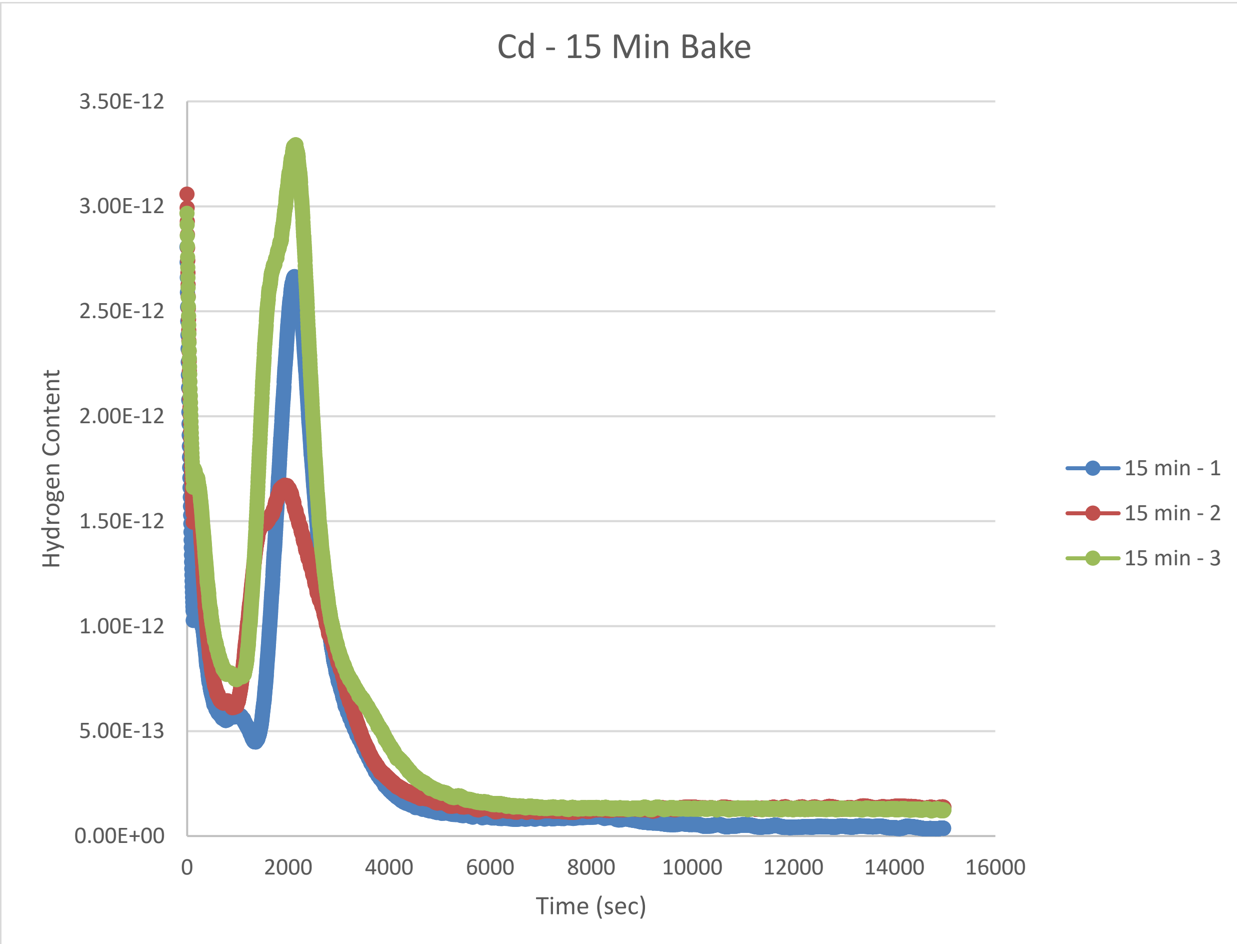

Figure 15: Cd Plated Coupons - 15 min Bake

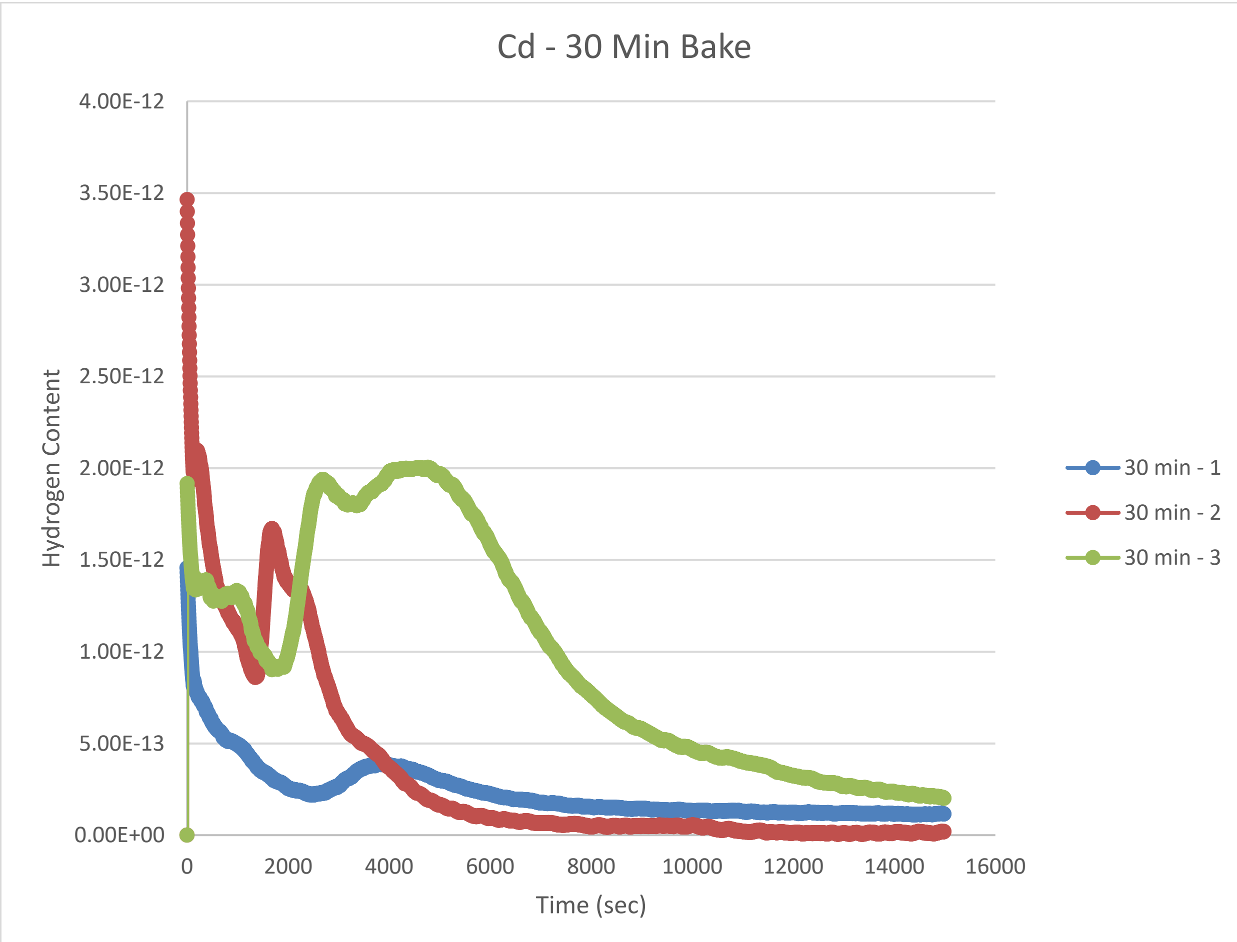

Figure 16: Cd Plated Coupons - 30 min Bake

The following graph shows a comparison of the amount of hydrogen between the unbaked, baked for 15 min and baked for 30 min. The displayed results show the lowest hydrogen content in the unbaked coupon compared to the highest measured hydrogen from the 15 min and 30 min baked coupons. The results show that there is a reduced amount of hydrogen in the coupon from the varied bake times.

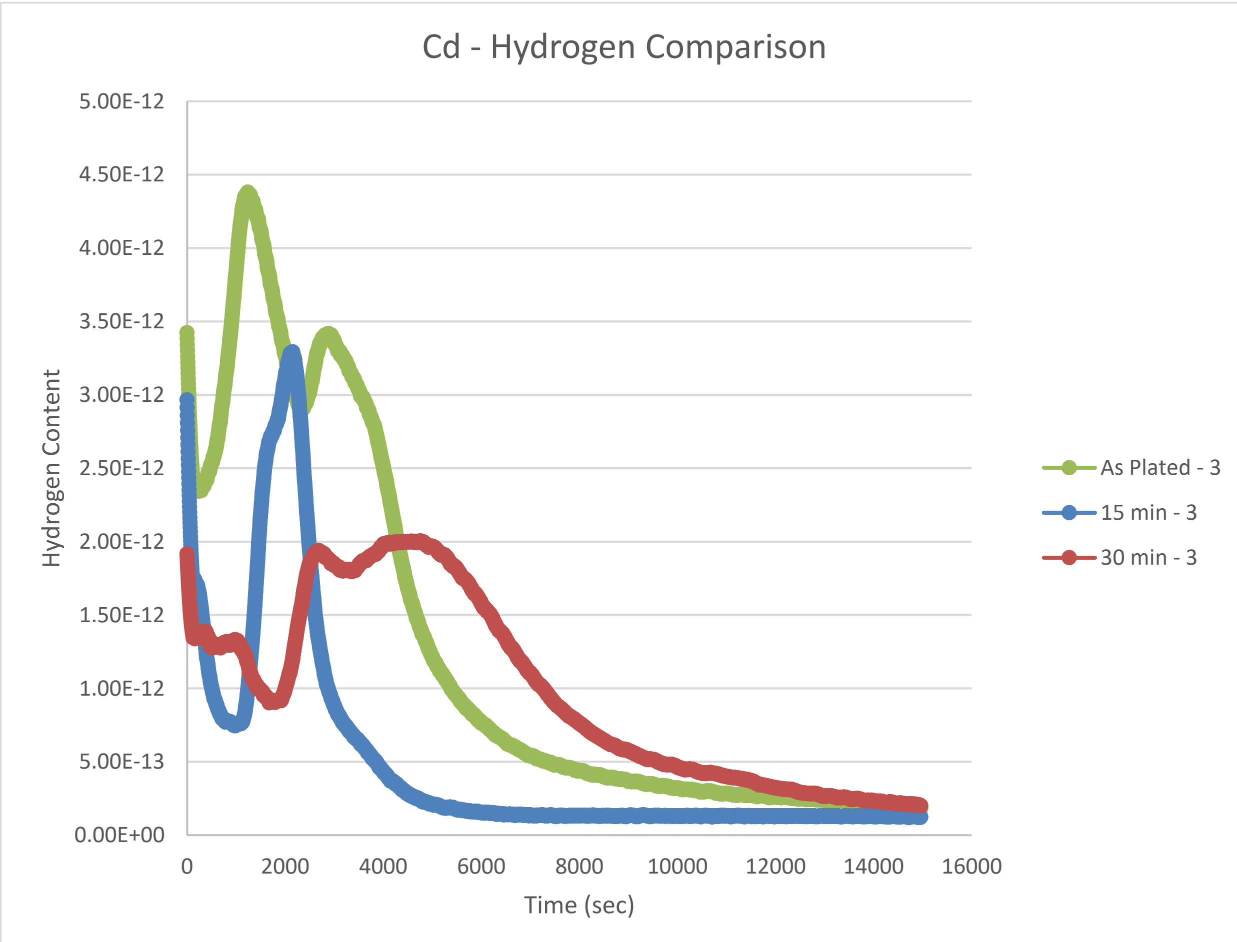

Figure 17: Cd Plated Coupons - Hydrogen Bake Comparison

The un-plated coupon data was added in to show the background level of hydrogen found in the coupons. The data shows that baking the coupon, even for 15 minutes removes the hydrogen to levels near the un-plated levels.

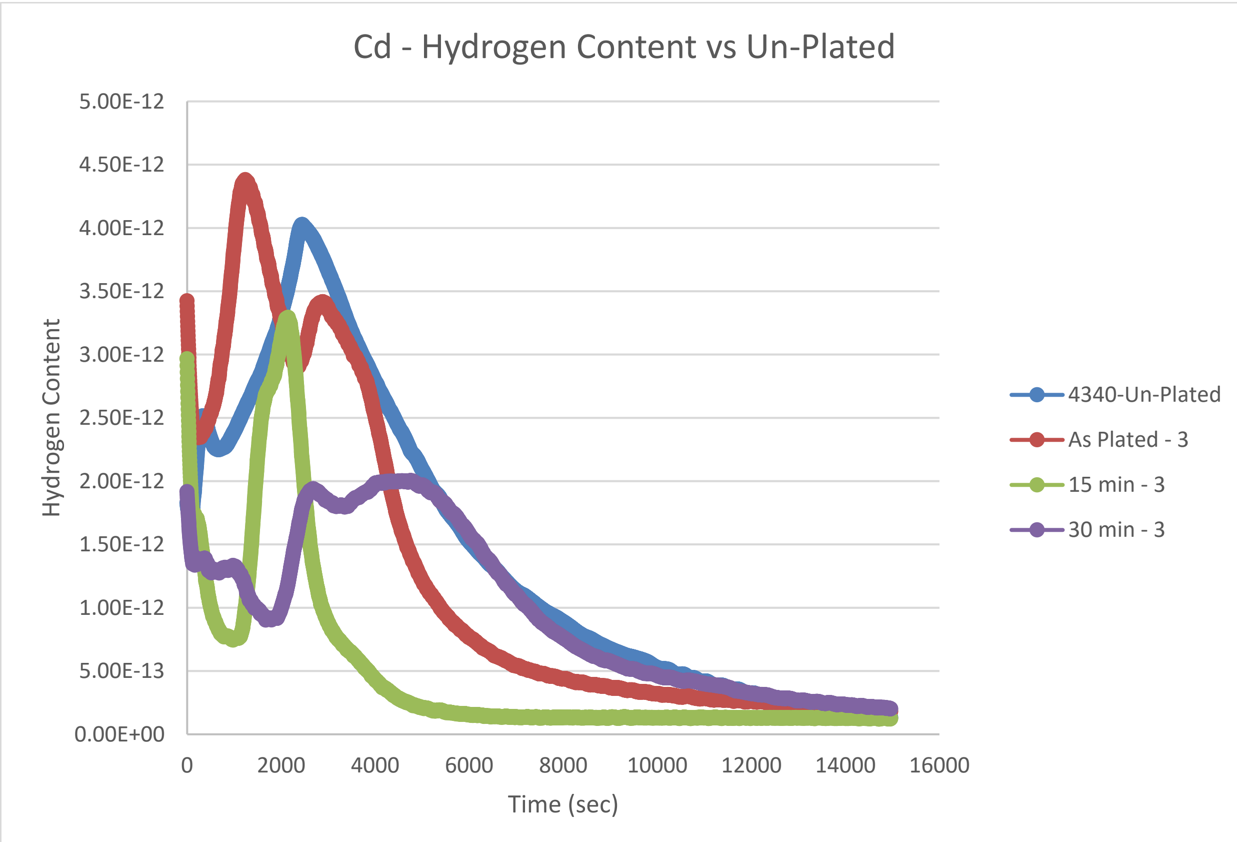

Figure 18: Hydrogen Content Compared to Un-Plated

The following graph shows the greatest amount of hydrogen measured compared to the least amount of hydrogen measured in the coupons that were plated and baked.

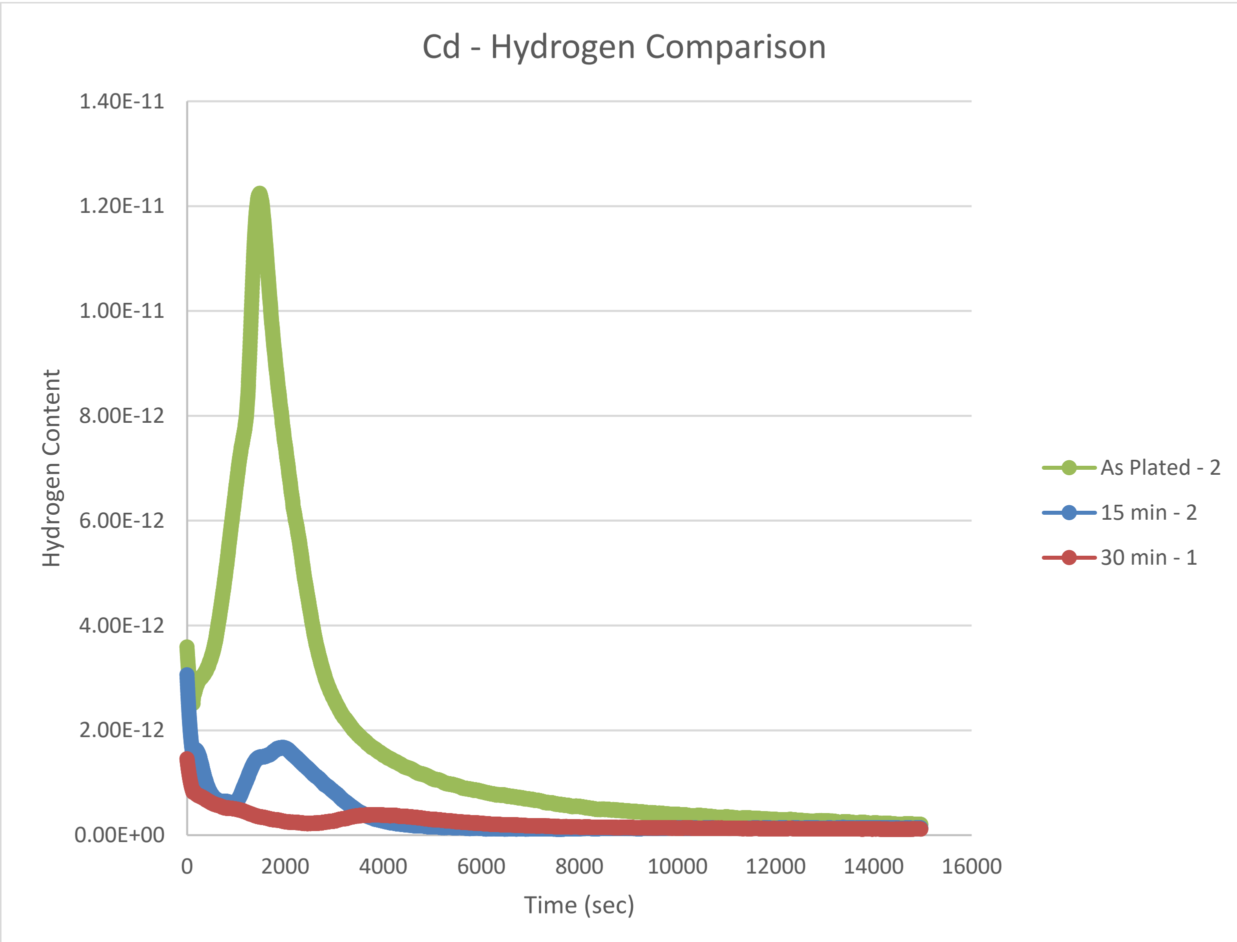

Figure 19: Cd Plated Coupons - Hydrogen Bake Comparison

5.3.1 Thermal Desorption Spectroscopy 1a.1 Coupon Conclusions

Based upon the data gathered the bake time of coupons after electroplating can be reduced. Levels of hydrogen measured before plating, after plating and after bake show that the amount of hydrogen is reduced from baking to levels near the un-plated coupons within 15 minutes.

5.3.2 Thermal Desorption Spectroscopy Block Testing

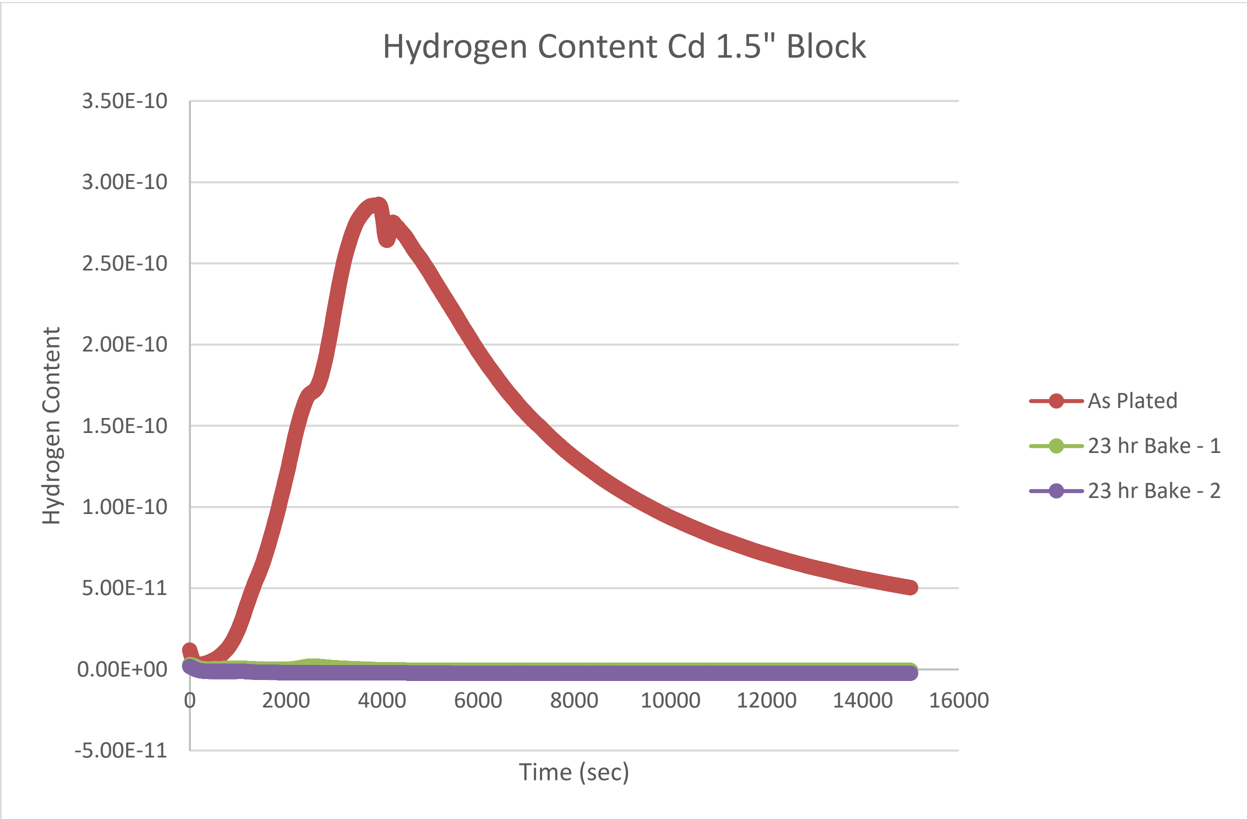

Figure 20: Hydrogen Content Pre and Post Bake

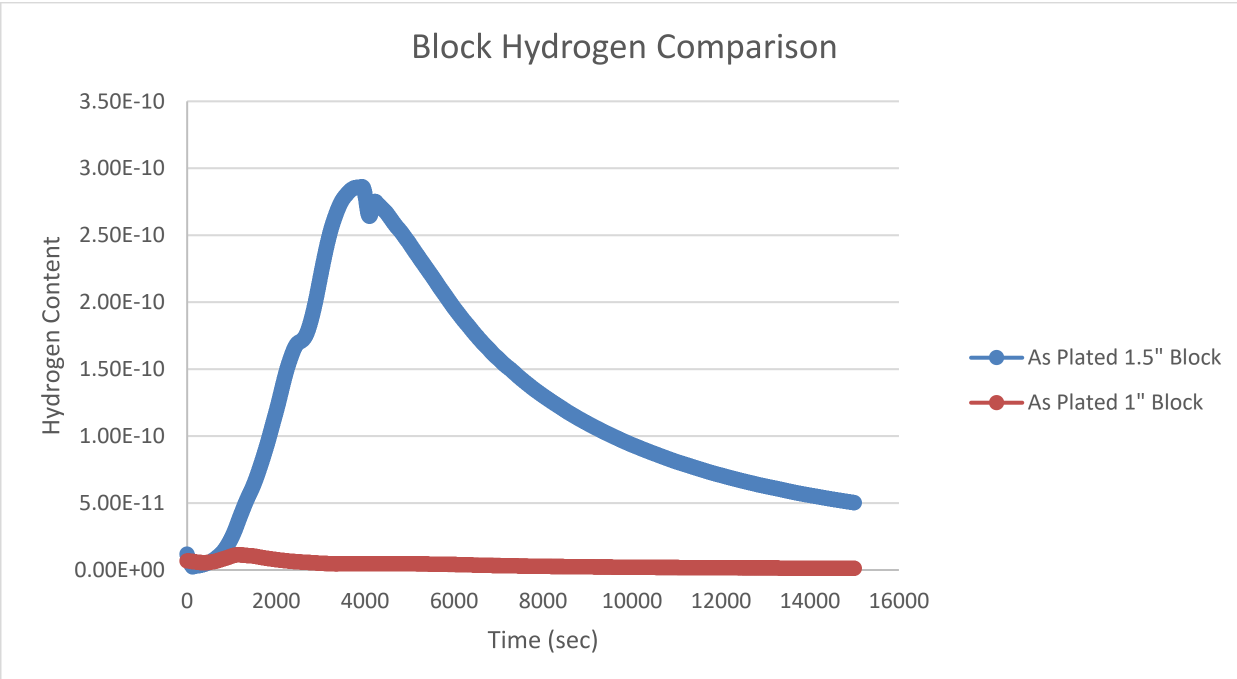

Figure 21: Hydrogen Content 1" Block vs 1.5" Block

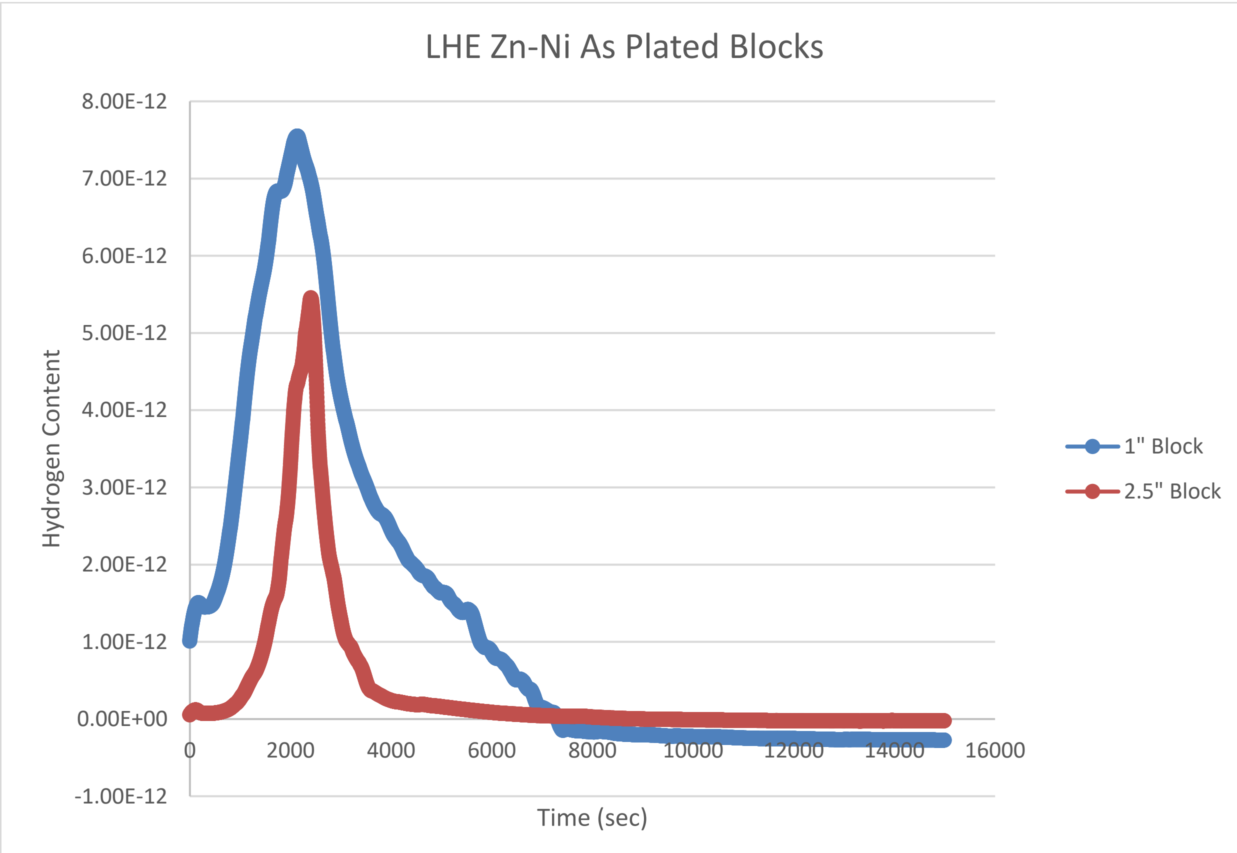

Figure 22: Hydrogen Content 1" Block vs 2.5" Block

5.3.3 Thermal Desorption Spectroscopy Conclusions

From all measurements made there are some trends that can be seen. Baking reduces the amount of hydrogen in the test coupons. A review of the data indicates that testing was accomplished near the acceptable limit of hydrogen to not cause HE failures. Testing should be conducted at a bake time that is further away from the critical bake time to provide more defined results.

5.4 LECO Results

Table 5: BR&T LECO Test Matrix and Results

	Hydrogen (ppm)						
Type 1a.1 Test Specimen	1st Reading	2nd Reading	3rd Reading	Average	Std. Dev.	TOTAL - AVERAGE	TOTAL -STANDARD DEVIATION
4340-1 Bare Steel*	0.2	3.2	0.3	1.23	1.70	1.17	1.08
4340-2 Bare Steel*	1	1.2	1.1	1.10	0.10		
300M-1 Bare Steel*	4.2	3.2	2.5	3.30	0.85	1.82	1.72
300M-2 Bare Steel*	0.2	0.3	0.5	0.33	0.15		
4340-3 Bare Steel	0.6	0.9	0.9	0.80	0.17	0.75	0.16
4340-4 Bare Steel	0.8	0.8	0.5	0.70	0.17		
300M-3 Bare Steel	0.8	0.7	0.5	0.67	0.15	0.53	0.24
300M-4 Bare Steel	0.2	0.3	0.7	0.40	0.26		
4340 ZnNi No Bake - 1	9.27	12.8	7.31	9.79	2.78	8.18	3.12
4340 ZnNi No Bake - 2	5.34	9.92	4.41	6.56	2.95		
4340 Cd No Bake - 1	7.7	8.09	7.33	7.71	0.38	8.03	0.75
4340 Cd No Bake - 2	9.48	7.83	7.73	8.35	0.98		
4340 ZnNi 24 Hour Bake - 1	4.91	5.27	6	5.39	0.56	6.13	0.89
4340 ZnNi 24 Hour Bake - 2	6.61	7.06	6.93	6.87	0.23		
4340 Cd 24 Hour Bake - 1	3.98	6.94	7.19	6.04	1.79	5.85	1.16
4340 Cd 24 Hour Bake - 2	5.31	5.76	5.9	5.66	0.31		
4340 ZnNi 1 Hour Bake - 1	2.18	3.34	5.49	3.67	1.68	4.82	1.72
4340 ZnNi 1 Hour Bake - 2	6.57	6.26	5.08	5.97	0.79		
4340 Cd 0.5 Hour Bake - 1	3.12	2.17	4.59	3.29	1.22	5.35	2.91
4340 Cd 0.5 Hour Bake - 2	10.1	7.31	4.82	7.41	2.64		
300M ZnNi No Bake - 1	2.94	1.82	4.42	3.06	1.30	3.38	1.12
300M ZnNi No Bake - 2	2.49	4.1	4.52	3.70	1.07		
300M Cd No Bake - 1	2.85	4.45	3.22	3.51	0.84	4.64	1.58
300M Cd No Bake - 2	6.04	4.34	6.93	5.77	1.32		

*4340-1 and 2 and 300M-1 and 2 hydrogen data was supect and repeated with -3 and -4 samples.
Repeat data was acceptable for as-received bare steel Type 1a.1 test specimens.

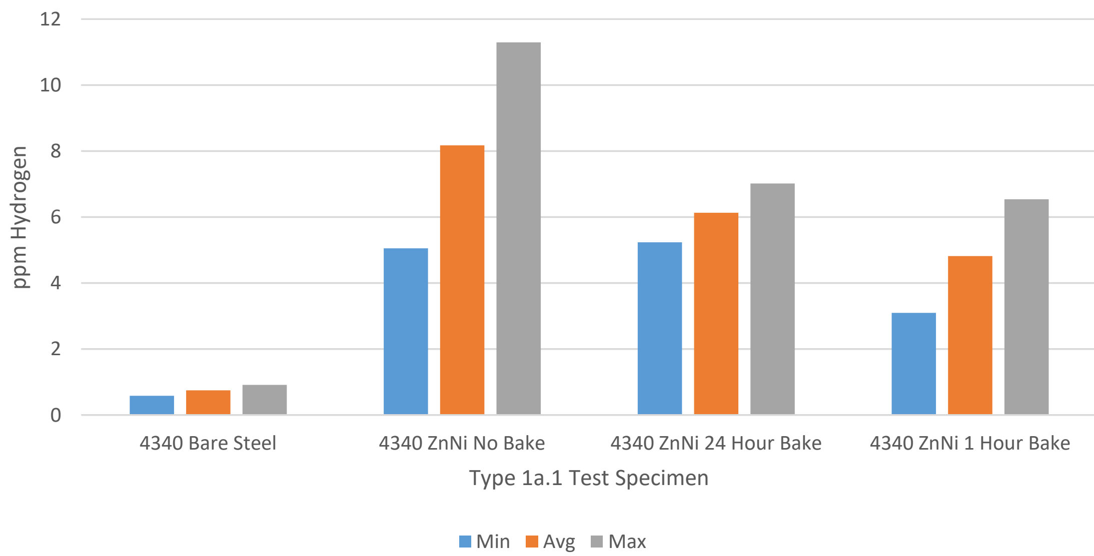

Figure 23: LECO 4340 LHE Zn-Ni

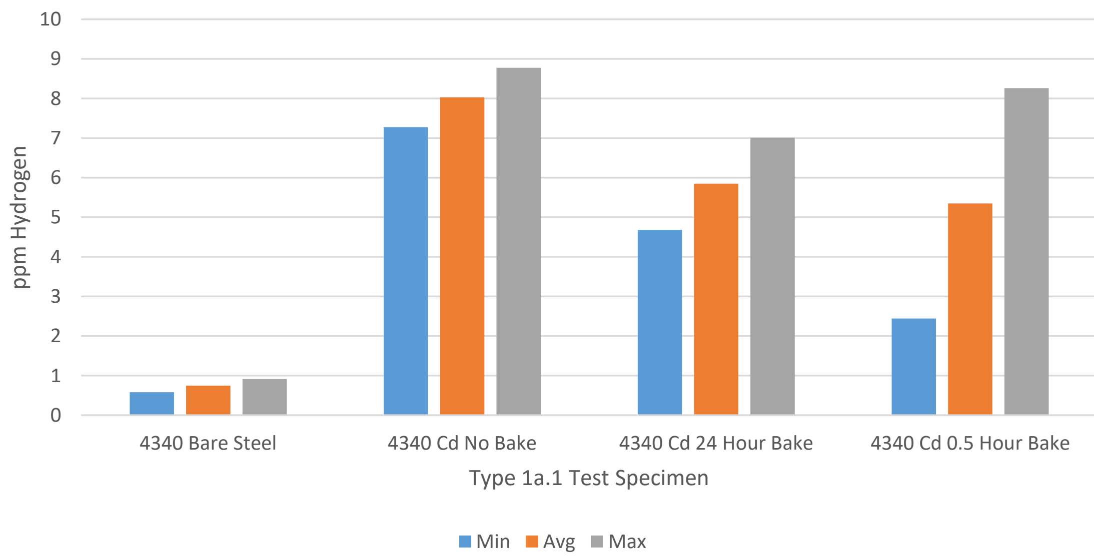

Figure 24: LECO 4340 Cd

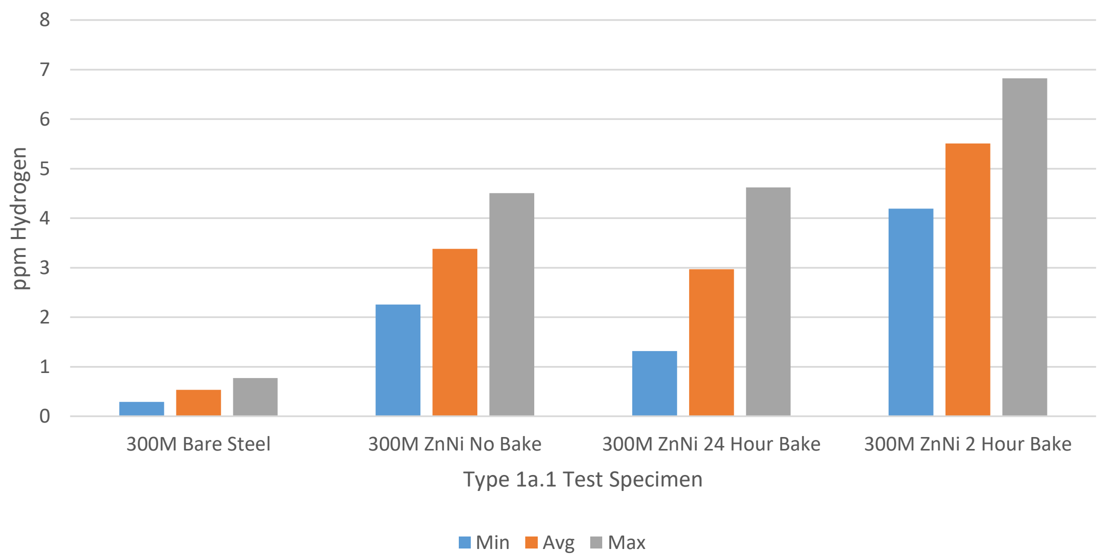

Figure 25: LECO 300M LHE Zn-Ni

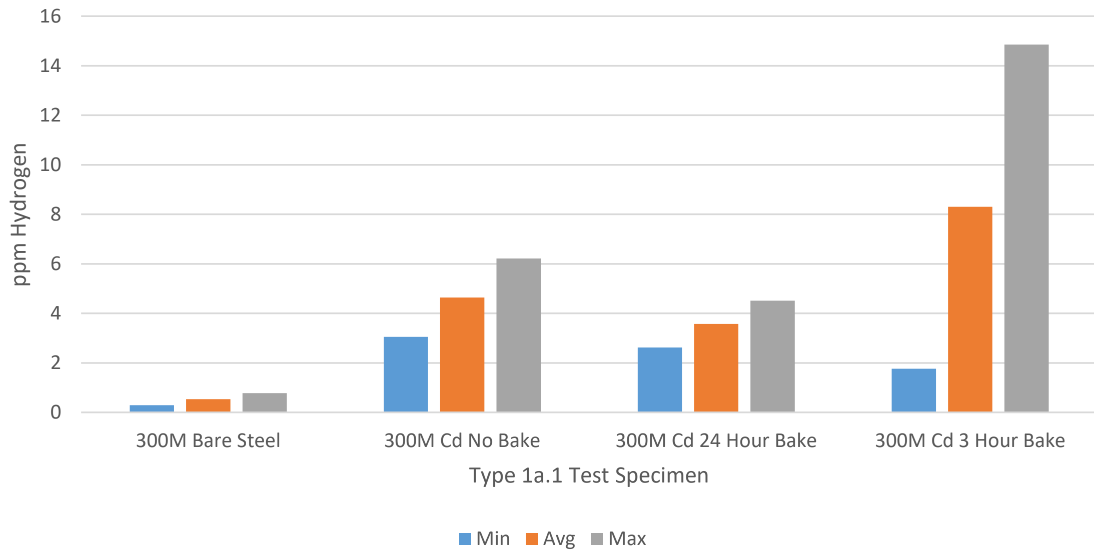

Figure 26: LECO 300M Cd

5.4.1 LECO Conclusions

Hydrogen concentration was the lowest for the bare steels (4340 and 300M) and rose significantly after either Cd or Zn-Ni plating, but baking for 23 hours at 355^o F could not restore the steel to the original low concentration levels for bare steel, and baking for the reduced times showed no significant difference when compared to baking at 23 hours.

Due to the data scatter, LECO hydrogen measurements should not solely be used to determine if a steel has been embrittled or non-embrittled.

It appears that during embritlement relief baking the hydrogen is either moving to trap sites in the steel or in the plating in order to pass the embritlement test. However, when the LECO test is done on these baked test specimens, and the steel sample (with plating) is melted and analyzed for hydrogen in the LECO equipment, the hydrogen is released from the trap sites, and/or plating, and produces a high concentration of hydrogen.

The values measured using LECO evaluation shows a definite increase of hydrogen due to the electroplating process. The reduction of hydrogen is also evident in the test measurements due to baking the specimen. The amount of hydrogen measured in a 24 hr bake is similar to the amount of hydrogen measured in a 1 hr bake for LHE Zn-Ni and 30 min bake for Cd. This data shows that from the coupons the hydrogen is able to be baked out of the coupon at a reduced bake time.

5.4.2 Low Current Density Plating

Low current density plating was tested to identify characteristics that may occur from plating levels below the approved specification levels. The current density on a part is not 100% uniform. Dependent upon various factors from part geometry to anode distance and area ratio between the part and anode, there may me areas on the part that have a lower current density than required in the plating specification.

Table 6: Low Current Density (20 ASF) HE Testing

Bake Time	Bake Temperature	HSS	Plating Type	Coupon Geometry	Notch Fracture Strength	Pass/Fail
1 hr	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	55%	Fail
2 hr	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	55%	Fail
4 hr	355 ± 5 ˚F	4340	LHE Zn-Ni	1a.1	93%	Pass
1 hr	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	35%	Fa
2 hr	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	35%	Fail
4 hr	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	50%	Fail
4 hr	355 ± 5 ˚F	300M	LHE Zn-Ni	1a.1	90%	Pass

5.4.3 Low Current Density Plating Conclusions

There is an increase in required bake time to allow for coupons to pass the HE testing. The this is expected as the coupon is exposed to an embrittling environment for a longer period of time and the porosity of the coating is reduced due to plating factors.

5.4.4 Residual Stress and Electroplating

Hydrogen migrates to high stress locations on parts and collect causing HE. The effects of residual stress from overhaul or manufacturing practices has been identified as a potential area of concern during electroplating processes. If the residual stress is too high the component may facture during or shortly after plating.

To investigate this phenomenon, 300M HSS ASTM F519 type 1d (notched c-ring) coupons were LHE Zn-Ni plated under stress, see Table 7. After plating, C-rings underwent the 200-hr sustained load test at 75% NFS.

Figure 27: Coupon Electroplated Under Stress

Table 7: Stressed C-Ring Sustained Load Test Results

% Notch Fracture Strength During Plating	200-hr SLT Pass/Fail	Notes
45%	Fail	Failed during plating
35%	Fail	Failed during Plating
25%	Fail	Failed 200-hr sustained load test
15%	Pass	

Testing showed that indeed if the stresses in the part are too high there may be fracture either during plating or after plating under load. For these coupons the stress level at 15% is approximately 44 ksi. At this level of stress there was no failure in the material.

Figure 28: Coupon That Fractured During Plating

6 Recommendations

Reduction in the required bake time has been demonstrated as a truly viable option. Coupons were able to pass a reduced bake time to levels lower than a quarter of the 23 hr required bake time. Coupons however are relatively small in comparison to full sized landing gear parts. A large part such as an outer cylinder was monitored to identify when the part reached the oven temperature. The part took 5 hours to reach the oven temperature of 375°F.

A review of the data indicates that coupon testing was accomplished near the acceptable limit of hydrogen to not cause HE failures. Testing should be conducted at a bake time that is further away from the critical bake time to provide more defined results.

Scaling the testing from a coupon sized piece to a full size landing gear component for HE testing is recommended. As previously mentioned the time that it takes for a large part can take hours before reaching the oven temperature. Data gathered using the block coupons to simulate thick sections of landing gear did not produce enough data to provide confidence in the results. Testing using thick HE coupon specimen would be recommended to represent the thick sections of landing gear components. A demonstration of incrementally stepped up coupon thickness may achieve desired results without having to go straight to the thickest 2.5” section.

7 Appendices

Appendix A – Certificates of Manufacture

GREEN SPECIALTY
greenspecialty.com

20 December 2016

ES3 INC.
550 W. C Street Suite 1630
San Diego , CA 92101

ATTN: Chuck CFO

CERTIFICATION OF MANUFACTURE

Quantity of Parts:250

Condition:260-280 KSI

Purchase Order Number: 11601

Job Number: 87045

Part Number: GSS1001

Grain Direction:LONGITUDINAL

Identification Number: 16V

Inventory Item Number:84772

Material:AISI4340 SAE AMS-S-5000 (AMS6414L,AMS 6415 T)

Material Identity:REPUBLIC # G22264K02

Chemical Analysis: Report # 4676

RC Hardness Avg: 51.53

Heat Treat: Report # 34-132874

Tensile Test: Report # 4500 Avg=262.325 KSI

Notch Tensile: Report # 4666

Notch Tensile Strength: 8514 Lbs

Sensitivity: Report # 16-52540, Complies with ASTM F519 -13

Sensitivity Test PSI = 350,370 psi

Bake: 4 Hours at 375° F after Final Machining

Process: Manufactured in accordance with Figure 8 of ASTM E8/E-15a, ASTM F519-13 (1a1) and standards AMS-QQ-P-416, AMS-QQ-N-290, AMS-QQ-C-320 and AMS-2460. This is to certify that all other dimensions in FIG. A1.1 were inspected and Conforms to the FIG. A1.1 Dimensional requirements for Type 1A1 Specimen.

Thank You,

Richard G. Green
President

Green Specialty Service Inc. 301 West Morningside Drive Fort Worth, Texas 76110
Phone (817) 924-4323 Fax (817) 924-3461 Email-sales@greenspecialty.com

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GREEN SPECIALTY
greenspecialty.com

16 December 2016

ES3 INC.
550 W. C Street Suite 1630
San Diego, CA 92101

ATTN: Jeremy Hall

CERTIFICATION OF MANUFACTURE

Quantity of Parts:250

Condition:280-300 KSI

Purchase Order Number: 11601

Job Number: 87045

Part Number: GSS1001 280-300

Grain Direction: LONGITUDINAL

Identification Number: 06A

Inventory Item Number: 79286

Material: AISI4340M (300M) AMS 6417F
Chemical Analysis:

Material Identity: HT # 560713
RC Hardness Avg:

Heat Treat:

Tensile Test: 295.5 KSI

Notch Tensile: **Notch Tensile Strength:** 9538 Lbs

Sensitivity: , Complies with ASTM F519 -12A

Requirements = 392,510 psi

Bake: 4 Hours at 375° F after Final Machining

Process: Manufactured in accordance with Figure 8 of ASTM E8/E-09, ASTM F519-12A (1A1) (Exception – Condition and Material Above) .

Thank You,

Richard G. Green
President

GREEN SPECIALTY
greenspecialty.com

16 December 2016

ES3 INC.
550 W. C Street Suite 1630
San Diego, CA 92101

ATTN: Jeremy Hall

CERTIFICATION OF MANUFACTURE

Quantity of Parts:250

Condition:260-280 KSI

Purchase Order Number: 11601

Job Number: 87045

Part Number: GSS1001

Grain Direction: LONGITUDINAL

Identification Number: 58C

Inventory Item Number: 85551

Material: AISI4340 SAE AMS-S-5000 (AMS 6415 T)

Material Identity: REPUBLIC # 8198530

Chemical Analysis: Report # 4057

RC Hardness Avg: 51.71

Heat Treat: Report # 34-131815

Tensile Test: Report # 3777 Avg=270.775 KSI

Notch Tensile: Report # 4276

Notch Tensile Strength: 8465 Lbs

Sensitivity: Report # 16-52204, Complies with ASTM F519 -13

Sensitivity Test PSI = 348,354 psi

Bake: 4 Hours at 375° F after Final Machining

Process: Manufactured in accordance with Figure 8 of ASTM E8/E-15a, ASTM F519-13 (1a1) and standards AMS-QQ-P-416, AMS-QQ-N-290, AMS-QQ-C-320 and AMS-2460. This is to certify that all other dimensions in FIG. A1.1 were inspected and Conforms to the FIG. A1.1 Dimensional requirements for Type 1A1 Specimen.

Thank You,

Richard G. Green
President

Green Specialty Service Inc. 301 West Morningside Drive Fort Worth, Texas 76110
Phone (817) 924-4323 Fax (817) 924-3461 Email-sales@greenspecialty.com

Appendix B – Photos and Figures

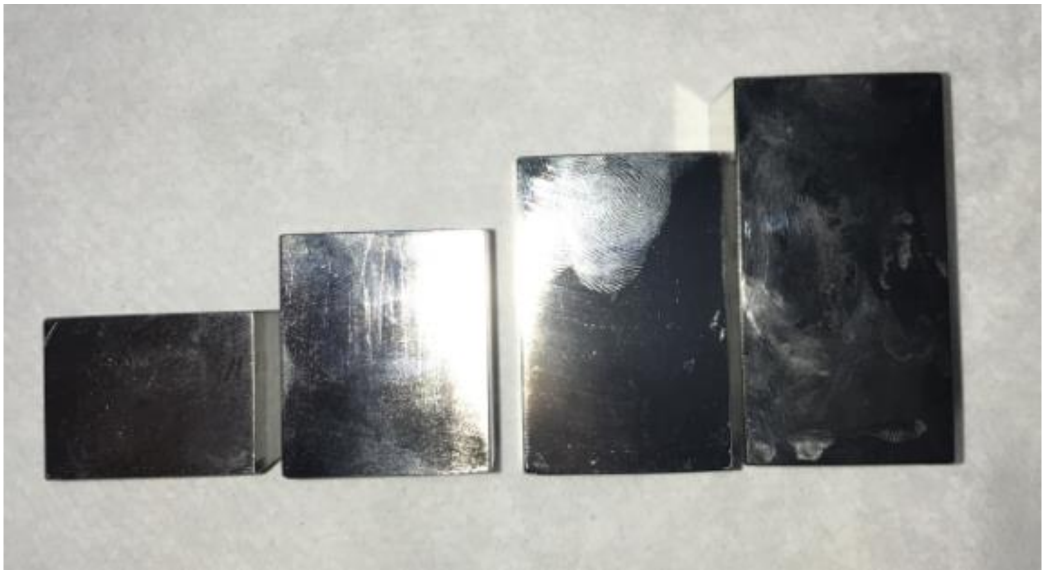

Figure 29: Electroless Nickel Plated 300M Block Test Coupons

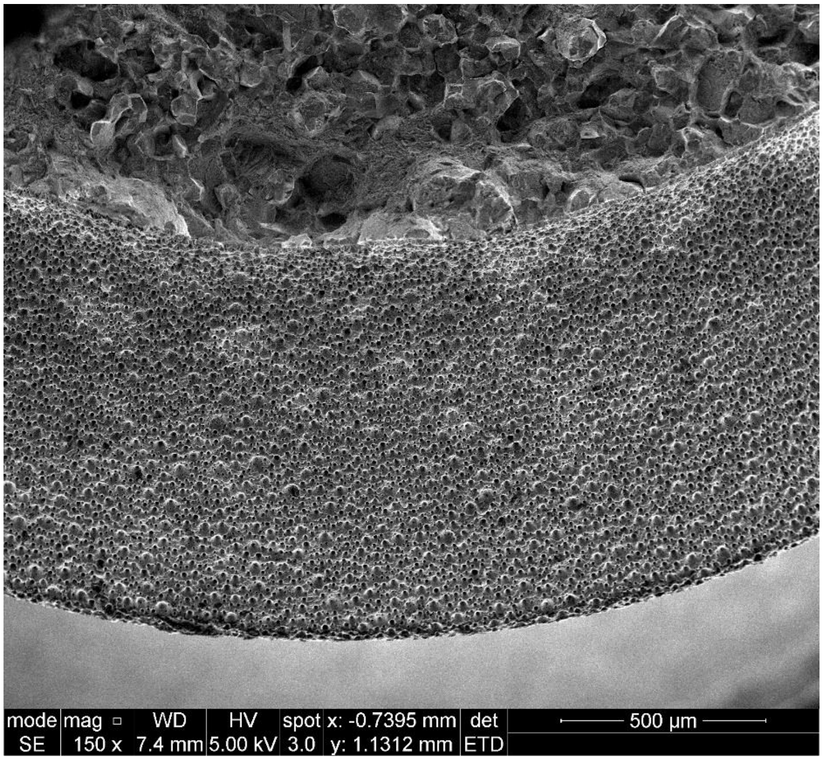

Figure 30: 300M 1a.1 ISL No Bake

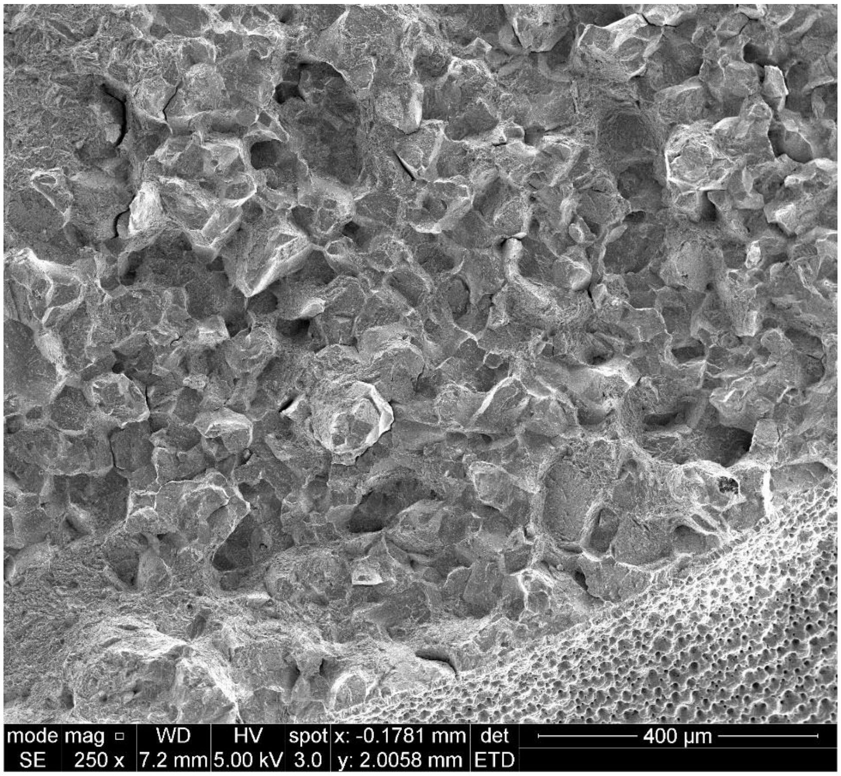

Figure 31: 300M 1a.1 ISL No Bake