

# **Alternative to Nitric Acid Passivation**

NASA Corrosion Technology Laboratory

&

NASA Technology Evaluation for Environmental Risk Mitigation

# Alternative to Nitric Acid Passivation

## Outline

- **Background**
- **Risk**
- **Specifications**
- **Benefits**
- **Objective**
- **Testing**
- **Collaboration**
- **Summary**



# Alternative to Nitric Acid Passivation

## Background

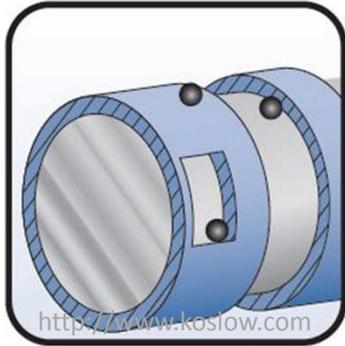
- Corrosion is an extensive problem that affects the National Aeronautics and Space Administration (NASA) and European Space Agency (ESA).
- The deleterious effects of corrosion result in steep costs, asset downtime affecting mission readiness, and safety risks to personnel.
- It is vital to reduce corrosion costs and risks in a sustainable manner.



# Alternative to Nitric Acid Passivation

## Background

- The standard practice for protection of stainless steel is passivation.
- Passivation works by forming a shielding outer (metal oxide) layer that reduces the impact of deleterious environmental factors such as air or water.
  - Typical passivation procedures call for the use of nitric acid; however, there are a number of environmental, worker safety, and operational issues associated with its use.



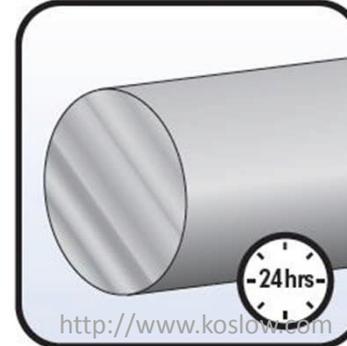
### Non-Passive Stainless Steel

- Free iron particles (un-alloyed iron)
- Damage or a scratch to the passive layer
- Also called active because surface can be prone to corrosion



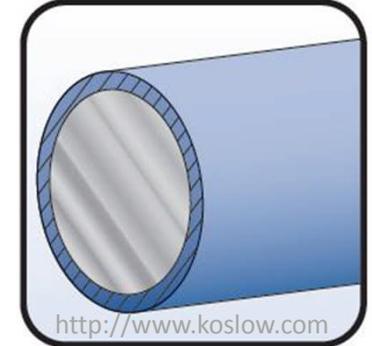
### Passivation Process

- Stainless steel is degreased, cleaned and prepped
- Stainless steel is immersed in an acid bath and rinsed



### Stripped Down to the Bare Metal

- Raw stainless steel after damaged passive film and contaminants have been dissolved
- Allow 8 – 24 hours to allow stainless steel to oxidize



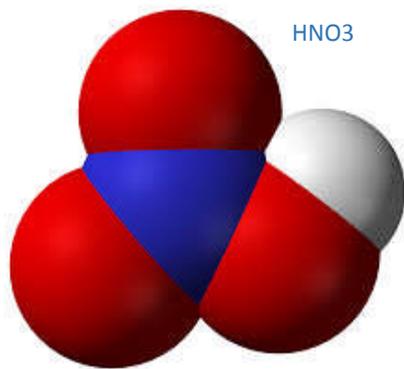
### Return to Passive Stainless Steel

- The spontaneous formation of a fresh passive film.
- Stainless steel now ready for a corrosion free service

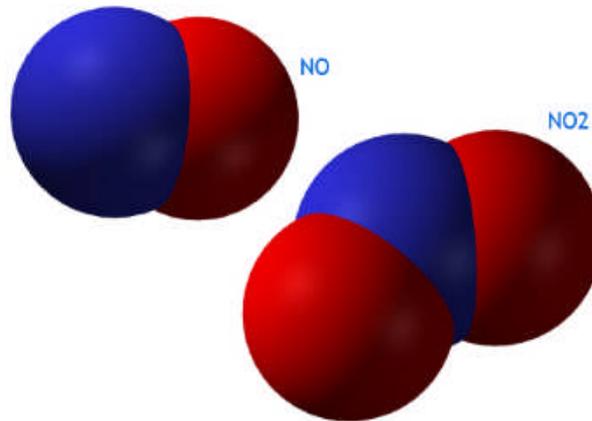
## Alternative to Nitric Acid Passivation

### Risk

- Nitric acid passivation results in fumes that contain nitrogen dioxide and nitrogen oxide (NO<sub>x</sub>) emissions which are considered greenhouse gases; Best Available Technology (BAT) to be employed to control nitric acid and NO<sub>x</sub> emissions
- Nitric acid passivation requires 25% or 50% concentration of the strong acid.
- Wastewater generated from the passivation process is regulated under the U.S. Environmental Protection Agency's (EPA) Metal Finishing Categorical Standards
- Nitric acid can remove beneficial heavy metals (nickel, chromium, etc.) that give stainless steel its desirable properties.



<http://commons.wikimedia.org>



<http://www.offshoreenergytoday.com>



<http://www.theguardian.com>

## Alternative to Nitric Acid Passivation

### Specification

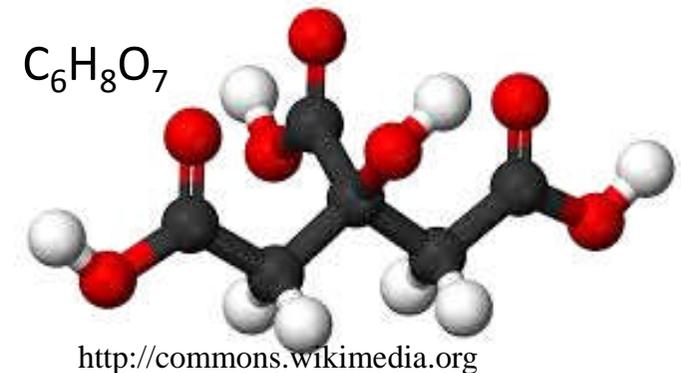
- ASTM A 967 (*Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts*) and AMS 2700 (*Passivation Treatments for Corrosion-resistant Steel*), both allow for the use of citric acid in place of nitric acid.
- Citric acid is similarly called out in the ASTM A 380 (*Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems*) standard.
- Citric acid passivation is not a new technology; it was developed (many years ago) for the beverage industry in Germany to process containers that were free of iron which causes an unwanted taste to the beverage.
- While citric acid use has become more prominent in industry in the U.S., there is little evidence that citric acid is a technically sound passivating agent, especially for the unique and critical applications encountered by NASA and ESA.



## Alternative to Nitric Acid Passivation

### Benefits of Citric Acid Passivation

- Citric acid is a bio-based material that helps government agencies meet the procurement requirements of the Farm Security and Rural Investment Act of 2002
- There are no toxic fumes created during the citric acid passivation process making it safer for workers.
- Nitric acid passivation requires 25% or 50% concentrations of the strong acid which are extremely corrosive and hazardous to workers.
- Citric acid removes iron from the surface more efficiently than nitric acid and therefore uses much lower concentrations reducing material costs.
- Citric acid-based processing baths retain their potency for longer periods requiring less frequent refilling and reduced volume and potential toxicity of effluent and rinse water.



## Alternative to Nitric Acid Passivation

### Benefits of Citric Acid Passivation

KSC Corrosion Lab recorded the following data:

- 4% Citric Acid has a pH of 2.39
- 50% nitric acid had a pH < 1

KSC Process Waste Questionnaire Technical Response Package =  
TCLP METALS BELOW RCRA REGULATORY LEVELS

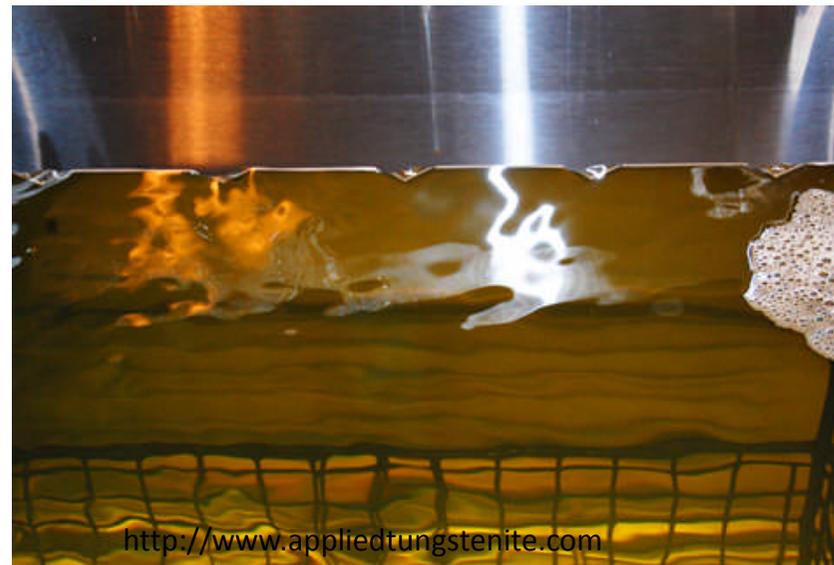
- Estimated costs for nitric or citric acid with a pH of < 2 would be about \$235/55 gal drum {€207/208L}
- Estimated costs for these wastes with pH > 2 and no other hazardous waste concerns, such as toxic metals, would be about \$80/55 gal drum {€71/ 208L}



# Alternative to Nitric Acid Passivation

## Objective

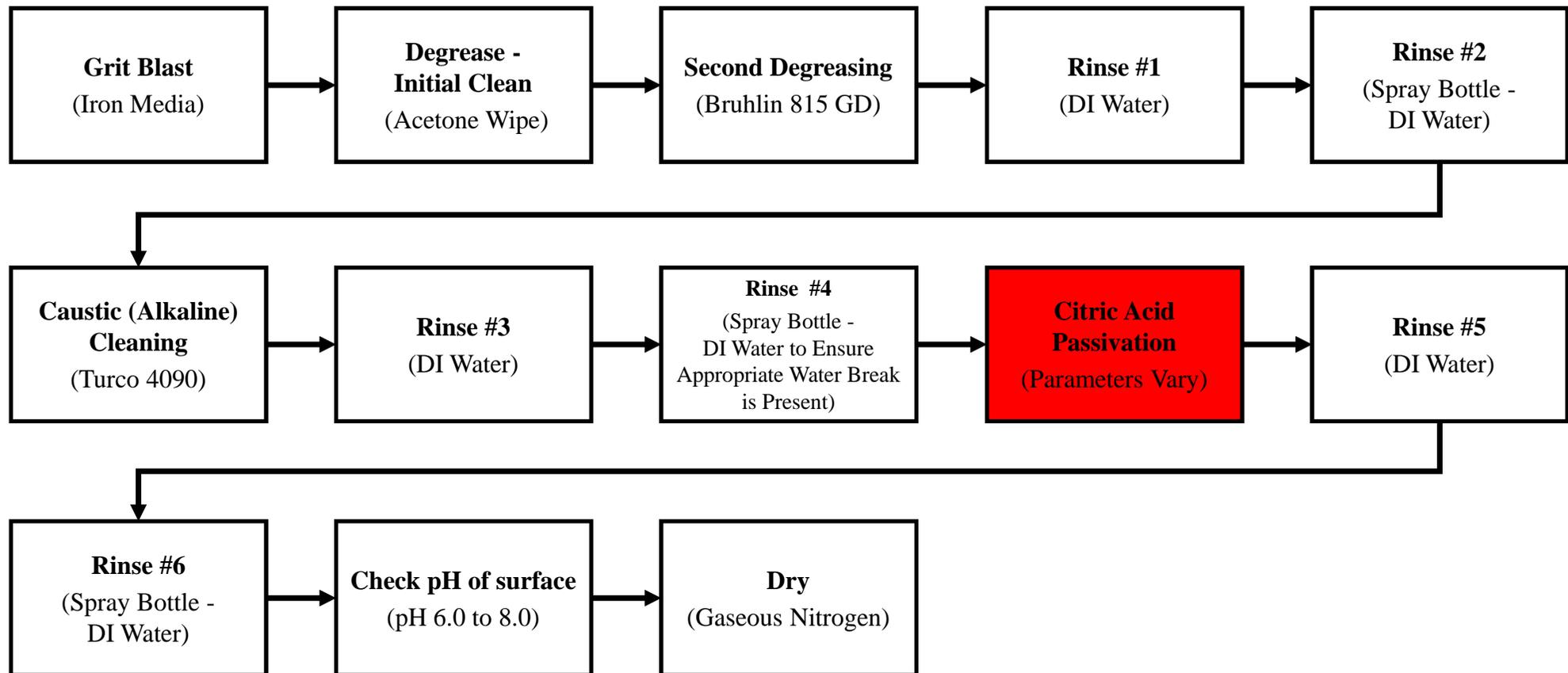
- The primary objective of this effort is to qualify citric acid as an environmentally-preferable alternative to nitric acid for passivation of stainless steel alloys.



# Alternative to Nitric Acid Passivation

## Test Specimen Preparation

The NASA Corrosion Technology Lab followed the United Space Alliance (USA) procedure for passivation:



# Alternative to Nitric Acid Passivation

## Parameter Optimization

Test panels of each stainless steel alloy were prepared using various process parameters

- Citric Acid Concentration: 4%
- Immersion Times: 60, 90, and 120 minutes
- Bath Temperatures: 100, 140, and 180°F
- Salt Spray Testing per ASTM B 117
- Corrosion Resistance Evaluation per ASTM D 610 every 168 hours
- Parameters resulting in the best corrosion resistance shall be used for preparation of that substrate's test panels for the remainder of the testing

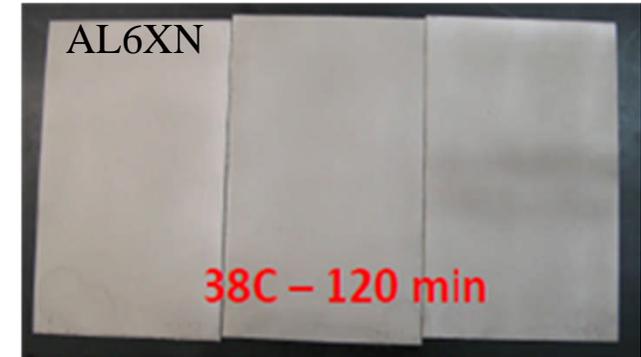


## Alternative to Nitric Acid Passivation

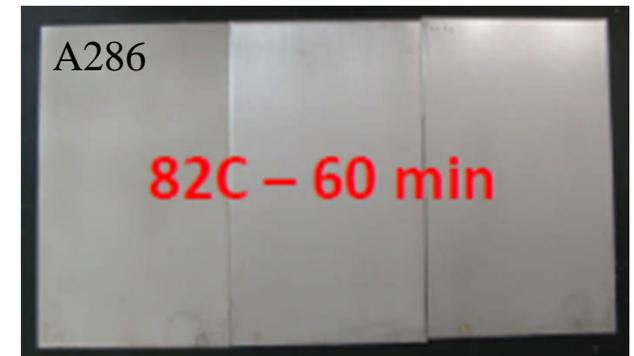
Process Parameters Used for Testing				
Alloy	Passivation	Concentration (%)	Bath Temperature (°C)	Dwell Time (minutes)
AL6XN	Nitric Acid	22.5	60	20
	Citric Acid	4	38	120
A286	Nitric Acid	50	64	30
	Citric Acid	4	82	60
304	Nitric Acid	22.5	60	20
	Citric Acid	4	49	120
17-4PH <sup>1</sup>	Nitric Acid	50	64	30
	Citric Acid	4	38	30
316	Nitric Acid	22.5	60	20
	Citric Acid	4	60	90
321	Nitric Acid	22.5	60	20
	Citric Acid	4	82	60
410	Nitric Acid	50	64	30
	Citric Acid	4	82	60
440C	Nitric Acid	50	64	30
	Citric Acid	4	60	60
15-5PH	Nitric Acid	50	64	30
	Citric Acid	4	82	60
17-7 PH	Nitric Acid	50	64	30
	Citric Acid	4	82	60

Note 1 = Citric acid parameters were initially determined by USA

All other citric acid parameters were determined by KSC Corrosion Lab



@ 504 Hours of ASTM B117 Exposure



@ 504 Hours of ASTM B117 Exposure

## Alternative to Nitric Acid Passivation

### Stainless Steel Alloy Composition

Alloy	C	Mn	Cr	Mo	Ni	Fe	Si	P	S	Al	Cu	Ti
AL6XN	0.03	2	20 - 22	6 - 7	23.5 - 25.5	BAL	1	0.04	0.03		0.75	
A286	0.08	2	13.5 - 16	1 - 1.5	24 - 27	BAL	1	0.025	0.025	0.35	0.5	1.9 - 2.35
304	0.08	2	18 - 20		8 - 10.5	BAL	0.75	0.04	0.03			
17-4PH	0.07	1	15 - 17.5		3 - 5	BAL	1	0.04	0.03		3 - 5	
316	0.08	2	16 - 18	2 - 3	10 - 14	BAL	0.75	0.04	0.03			
321	0.08	2	17 - 19		9 - 12	BAL	0.75	0.04	0.03			0.7
410	0.15	1	11.5 - 13.5			BAL	1	0.04	0.03			
440C	0.95 - 1.2	1	16 - 18	0.75		BAL	1	0.04	0.03			
15-5PH	0.07	1	14 - 15.5		3.5 - 5.5	BAL	1	0.04	0.03		2.2 - 4.50	
17-7PH	0.09	1	16 - 18		6.5 - 7.5	BAL	1	0.04	0.03	0.75 - 1.5		



<http://periodictable.com>

## Alternative to Nitric Acid Passivation

### Testing

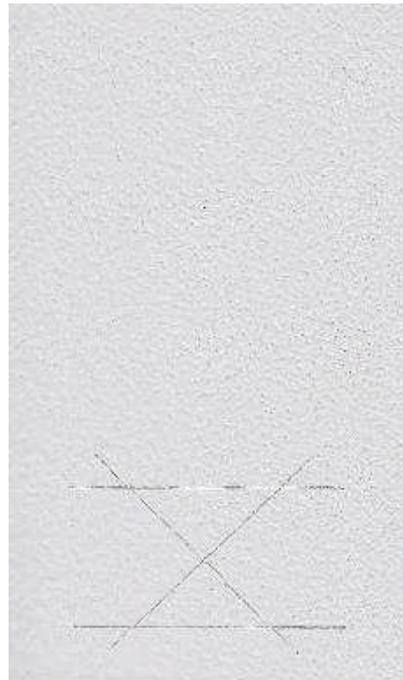
Test	Test Methodology References	Acceptance Criteria	Location
X-Cut Adhesion by Wet Tape	ASTM D 3359	Alternative performs as well or better than control process	NASA Corrosion Technology Lab
Tensile (Pull-off) Adhesion	ASTM D 4541		
Cyclic Corrosion Resistance	GMW 14872		
Atmospheric Exposure Testing	ASTM D 610		NASA Corrosion Technology Lab Atmospheric Exposure Site
	ASTM D 714		
	NASA-STD-5008		
Stress Corrosion Cracking	ASTM B 117		NASA Corrosion Technology Lab
	ASTM E 4		
	ASTM E 8		
	ASTM G 38		
	ASTM G 39		
Fatigue*	ASTM E 466		
Hydrogen Embrittlement**	ASTM F 519		
* = Only one alloy was tested; 17-4PH			
** = Test specimens were made of AISI 4340 alloy steel, this is considered worst case			

# Alternative to Nitric Acid Passivation

## X-Cut Adhesion by Wet Tape Testing



24 Hour Immersion @ Ambient Temperature



X-cut Scribed into the Surface



Masking Tape is Affixed to the Surface Using a Roller; Within 90 Seconds, the Tape is removed, Pulling (180-degree angle) Rapidly back upon Itself

# Alternative to Nitric Acid Passivation

## X-Cut Adhesion by Wet Tape Testing

Performs as well or better than control process

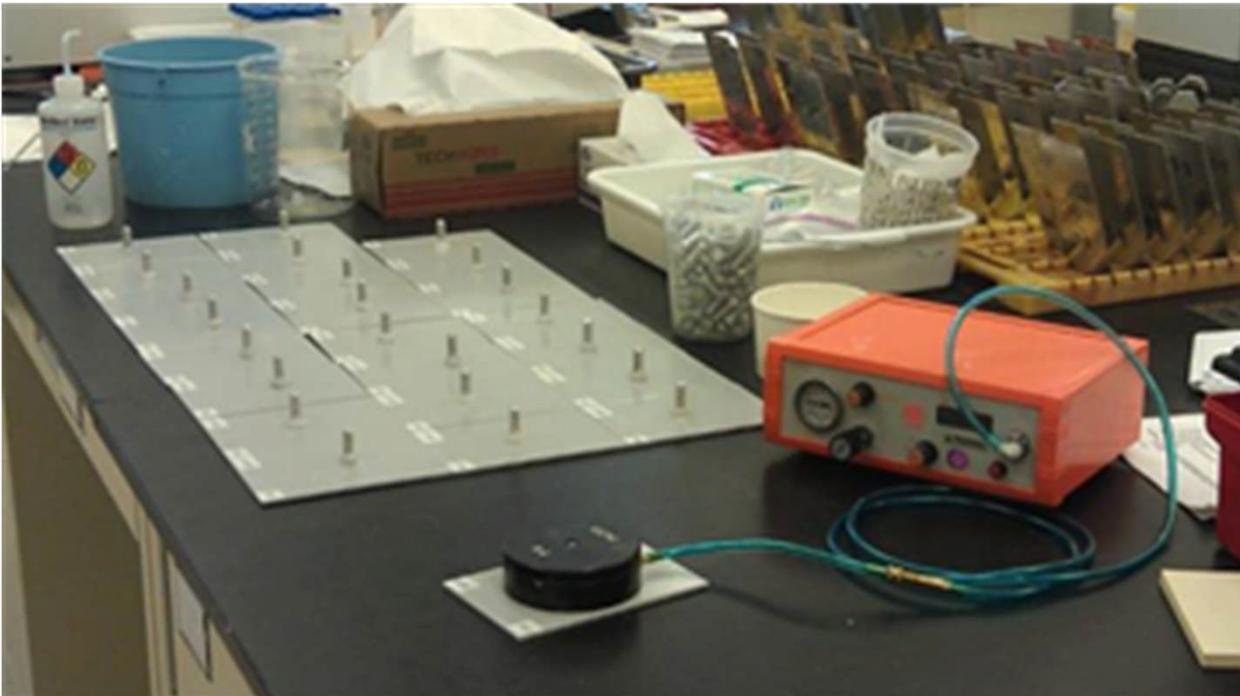
Alloy	Passivation	Coating Systems				
		Primer / Topcoat				
		Sherwin Williams E90H226 & V93V227 / Sherwin Williams F93G504 & V93V502	Sherwin Williams E90H226 & V93V227 / Sherwin Williams F93G116	Sherwin Williams E90W501 & V93V505 / Sherwin Williams F93G106	Carboline Carboguard 893 / Carboline Carbothane 134 MC	Sherwin Williams Polysiloxane XLE2
AL6XN	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
17-4PH	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
286	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
304	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
17-7PH	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
410	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
155	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
321	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
316	Citric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>
	Nitric	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>	5A <sup>1</sup>

Note <sup>1</sup> = 5A is the highest rating available; no peeling or removal of the coating at the scribe

## Alternative to Nitric Acid Passivation

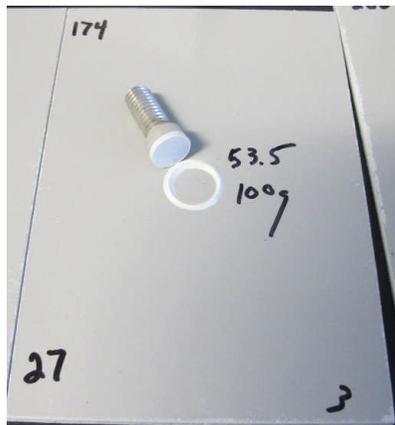
### Tensile (Pull-Off) Adhesion

The pull-off test is performed by securing a loading fixture (dolly) to the surface of the coating with an adhesive. After the adhesive is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension normal to the test surface. The fixture is pulled from the surface of the panel and the burst pressure is recorded, that value is converted to produce a value for pull-off tensile adhesion (POTS).



## Alternative to Nitric Acid Passivation

### Tensile (Pull-Off) Adhesion – Phase I



Alloy	Primer Only	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
A-286	Carboline Carboguard 893 <sup>1</sup>	1504	1064
304	Carboline Carboguard 893 <sup>1</sup>	847	1383
AL6XN	Carboline Carboguard 893 <sup>1</sup>	1297	1292
17-4PH	Carboline Carboguard 893 <sup>1</sup>	1131	1292

Note<sup>1</sup> = NASA-STD-5008 Approved Products List

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

All pull-off values were over 500 psi, and the mode of failure was predominantly related to the adhesive used to glue the dolly to the surface of the panel.

# Alternative to Nitric Acid Passivation

## Tensile (Pull-Off) Adhesion – Full Alloy Set

Alloy	Primer Only	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Sherwin Williams E90H226 & V93V227 <sup>1</sup>	987	985
17-4PH		704	753
286		704	841
304		699	757
17-7PH		739	816
410		858	946
155		979	1127
321		995	945
316		753	978

Note<sup>1</sup> = Chemical Agent Resistant Coatings

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

Alloy	Primer Only	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Sherwin Williams E90W501 & V93V505 <sup>1</sup>	1016	1005
17-4PH		2159	2390
286		775	748
304		631	678
17-7PH		531	707
410		756	819
155		683	784
321		757	726
316		713	735

Note<sup>1</sup> = Chemical Agent Resistant Coatings

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

Alloy	Primer Only	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Carboline Carboguard 893 <sup>1</sup>	504	517
17-4PH		1052	1252
286		1045	1212
304		459*	512
17-7PH		536	534
410		481*	528
155		496*	523
321		523	523
316		489*	572

Note<sup>1</sup> = Coatings on the NASA-STD-5008 Approved Products List

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

Note\* = 100% glue failure

Alloy	Coating	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Sherwin Williams Polysiloxane XLE <sup>1</sup>	1259	1266
17-4PH		1164	1040
286		1025	1105
304		771	918
17-7PH		402*	438*
410		432*	479*
155		472*	434*
321		364*	417*
316		434*	452*

Note<sup>1</sup> =

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

Note\* = 100% glue failure

Alloy	Plating	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Hard Chrome Plating <sup>1</sup>	957	958
17-4PH		1871	1989
286		2359	2113
304		1542	2287
17-7PH		1255	1049
410		1086	1189
155		942	1284
321		958	981
316		880	830

Note<sup>1</sup> =

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

Alloy	Plating	Result PSI (ave) <sup>2</sup>	
		Passivation	
		Nitric	Citric
AL6XN	Cadmium Plating <sup>1</sup>	1201	1221
17-4PH		1629	1718
286		1857	2069
304		1368	1553
17-7PH		516	680
410		770	894
155		803	830
321		620	716
316		709	745

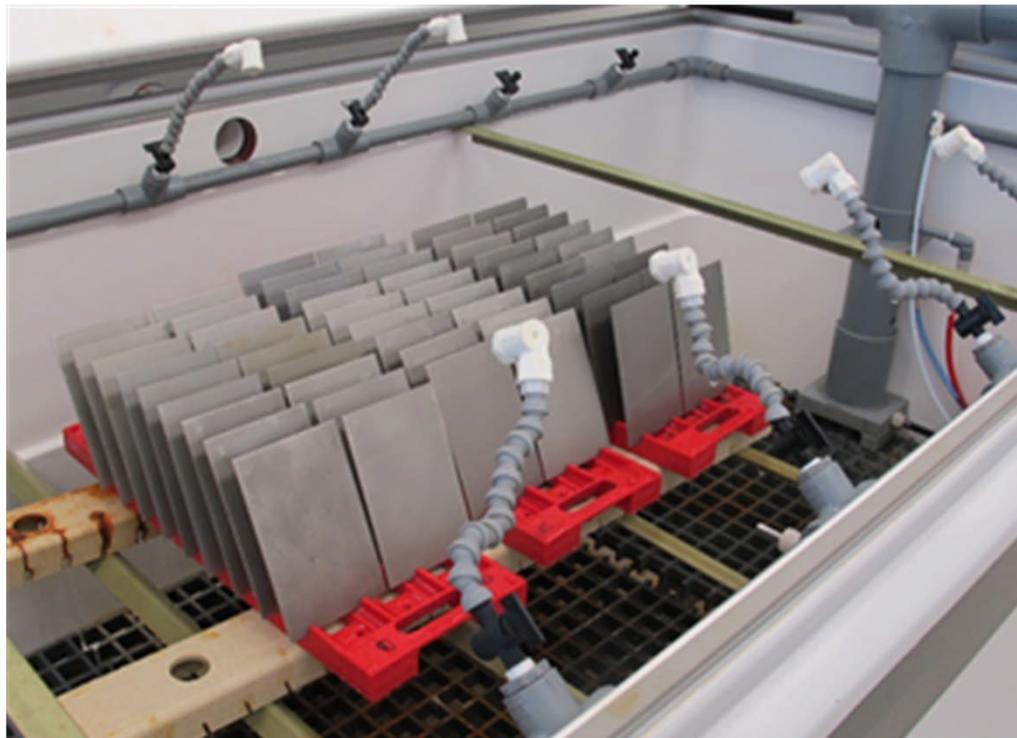
Note<sup>1</sup> = Coatings on the NASA-STD-5008 Approved Products List

Note<sup>2</sup> = Pull-off values over 500 psi are considered passing

## Alternative to Nitric Acid Passivation

### GMW 14872 Cyclic Corrosion Resistance - 80 Cycles

Each cycle consists of an 8 hour exposure under ambient conditions (25°C, 45% RH), an 8 hour exposure under high humidity conditions (49°C, 100% RH), and 8 hours under drying conditions (60°C,  $\leq$  30% RH). During the initial ambient stage, the specimens are sprayed with a solution comprised of sodium chloride (0.90%), calcium chloride (0.10%), sodium bicarbonate (0.075%) and water (98.925%).



## Alternative to Nitric Acid Passivation

### ASTM D 610:

Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces

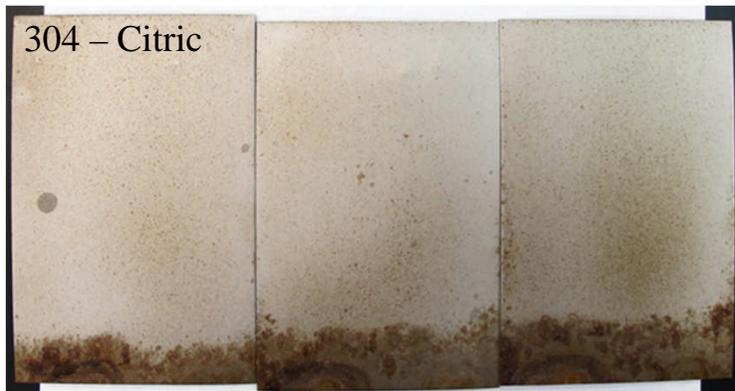
Rust Grade	Percent of Surface Rusted
10	Less than or equal to 0.01 percent
9	Greater than 0.01 percent and up to 0.03 percent
8	Greater than 0.03 percent and up to 0.1 percent
7	Greater than 0.1 percent and up to 0.3 percent
6	Greater than 0.3 percent and up to 1.0 percent
5	Greater than 1.0 percent and up to 3.0 percent
4	Greater than 3.0 percent and up to 10.0 percent
3	Greater than 10.0 percent and up to 16.0 percent
2	Greater than 16.0 percent and up to 33.0 percent
1	Greater than 33.0 percent and up to 50.0 percent
0	Greater than 50 percent

## Alternative to Nitric Acid Passivation

**GMW 14872**

**Cyclic Corrosion Resistance**

**Performs as well or better than control process**



Alloy	Passivation	Cyclic Corrosion
		Average Ratings After 80 Cycles
AL6XN	Citric	9
	Nitric	9
17-4PH	Citric	3
	Nitric	3
286	Citric	5
	Nitric	5
304	Citric	5
	Nitric	3
17-7PH	Citric	5
	Nitric	5
410	Citric	0
	Nitric	0
155	Citric	4
	Nitric	3
321	Citric	6
	Nitric	4
316	Citric	5
	Nitric	4



# Alternative to Nitric Acid Passivation

## Atmospheric Exposure Test

Phase I



Phase II



 National Aeronautics and  
Space Administration  
John F. Kennedy Space Center

**Corrosion Technology  
Laboratory  
Beach  
Atmospheric  
Exposure Site**

Authorized Personnel Only

## Alternative to Nitric Acid Passivation

### ASTM D 610:

Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces

Rust Grade	Percent of Surface Rusted
10	Less than or equal to 0.01 percent
9	Greater than 0.01 percent and up to 0.03 percent
8	Greater than 0.03 percent and up to 0.1 percent
7	Greater than 0.1 percent and up to 0.3 percent
6	Greater than 0.3 percent and up to 1.0 percent
5	Greater than 1.0 percent and up to 3.0 percent
4	Greater than 3.0 percent and up to 10.0 percent
3	Greater than 10.0 percent and up to 16.0 percent
2	Greater than 16.0 percent and up to 33.0 percent
1	Greater than 33.0 percent and up to 50.0 percent
0	Greater than 50 percent

## Alternative to Nitric Acid Passivation

### Atmospheric Exposure Test – Passivated Only – (Phase I Samples and Exposure)

**Performs as well or better than control process**

Alloy	Passivation	Atmospheric Exposure Test			
		1 Month Average Ranking	3 Month Average Ranking	6 Month Average Ranking	18 Month Average Ranking
A286	Citric	6	5	5	4
	Nitric	5	4	3	3
304	Citric	5	5	3	3
	Nitric	4	4	2	2
AL6XN	Citric	9	8	8	7
	Nitric	7	7	7	5
17-4PH	Citric	4	3	3	2
	Nitric	4	3	3	2

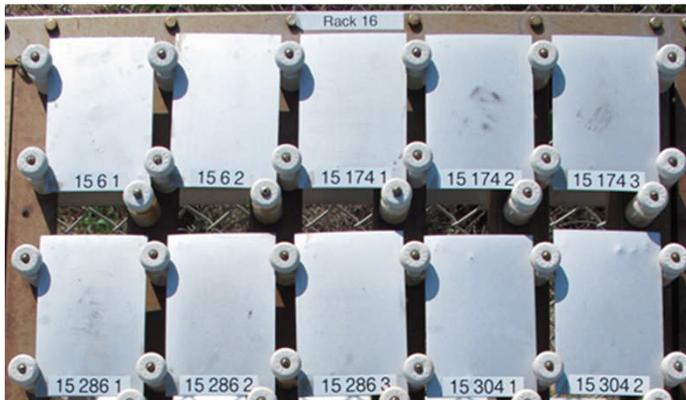


## Alternative to Nitric Acid Passivation

### Atmospheric Exposure Test – Passivated Only - (Phase II and III Samples)

**Performs as well or better than control process**

**Performs worse than control process**



Alloy	Passivation	Atmospheric Exposure Test			
		1 Month Average Ranking	3 Month Average Ranking	6 Month Average Ranking	12 Month Average Ranking
AL6XN	Citric	10	8	6	5
	Nitric	10	5	4	4
17-4PH	Citric	3	2	2	2
	Nitric	4	3	3	3
286	Citric	5	4	4	3
	Nitric	5	4	3	3
304	Citric	4	3	3	3
	Nitric	2	2	2	2
17-7PH	Citric	4	4	3	3
	Nitric	4	4	3	2
410	Citric	4	2	2	0
	Nitric	3	1	1	0
155	Citric	4	3	3	3
	Nitric	4	3	3	3
321	Citric	4	3	2	2
	Nitric	2	2	2	2
316	Citric	5	3	3	3
	Nitric	2	2	2	2

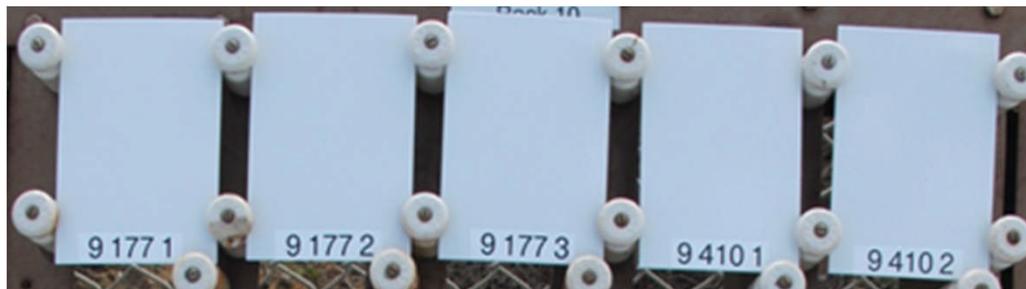
# Alternative to Nitric Acid Passivation

## Atmospheric Exposure Test – Passivated & Coated – (Phase I Samples Only)

**Performs as well or better than control process**

Alloy	Passivation	Primer	Topcoat	Atmospheric Exposure Test			
				1 Month Average Ranking	3 Month Average Ranking	6 Month Average Ranking	18 Month Average Ranking
A286	Citric	Carboline Carboguard	Carboline Carbothane	10	10	10	10
	Nitric	893 <sup>1</sup>	134 MC <sup>1</sup>	10	10	10	10
304	Citric	Carboline Carboguard	Carboline Carbothane	10	10	10	10
	Nitric	893 <sup>1</sup>	134 MC <sup>1</sup>	10	10	10	10
AL6XN	Citric	Carboline Carboguard	Carboline Carbothane	10	10	10	10
	Nitric	893 <sup>1</sup>	134 MC <sup>1</sup>	10	10	10	10
17-4PH	Citric	Carboline Carboguard	Carboline Carbothane	10	10	10	10
	Nitric	893 <sup>1</sup>	134 MC <sup>1</sup>	10	10	10	10

Note<sup>1</sup> = NASA-STD-5008 Approved Products List



## **Alternative to Nitric Acid Passivation**

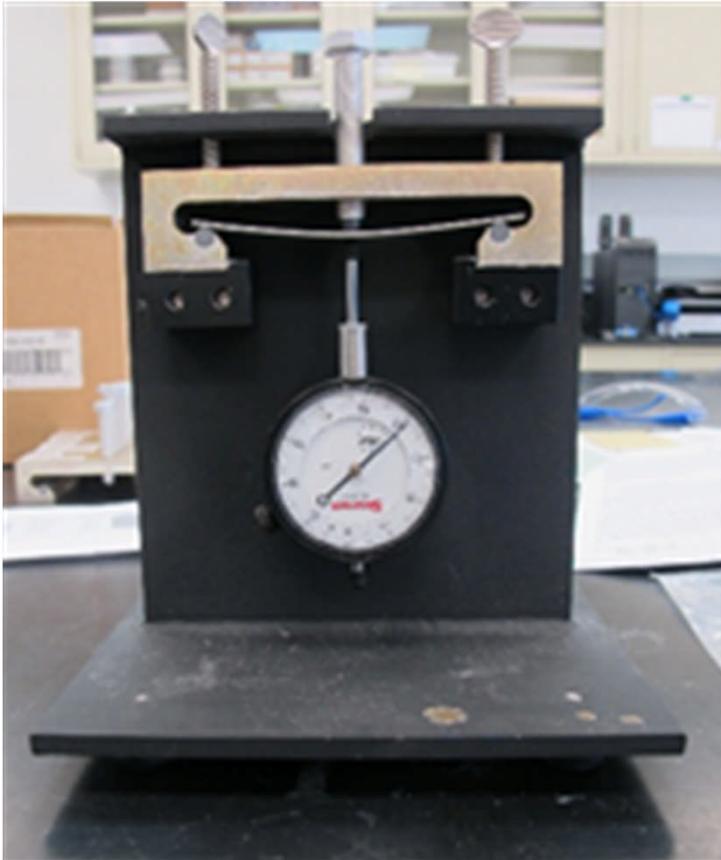
**Atmospheric Exposure Test – Passivated & Coated – Phase II and Phase III**

**Testing On-going**

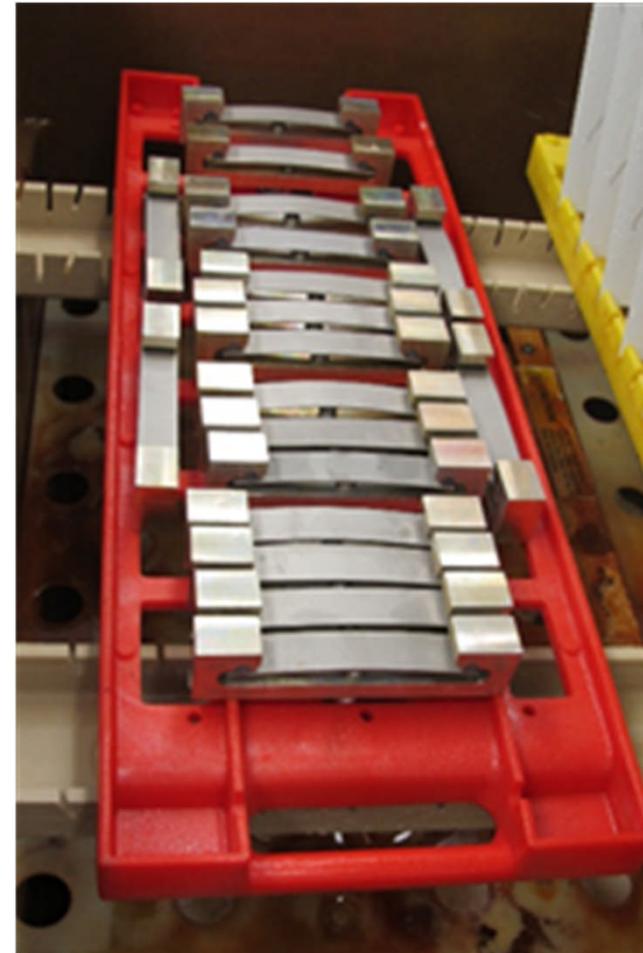
## Alternative to Nitric Acid Passivation

### Stress Corrosion Cracking

All alloys were loaded into test fixtures and were stressed according to the requirements of ASTM G39.



Samples are then Placed in ASTM B 117 Salt Spray Testing – 1,000 Hours



## Alternative to Nitric Acid Passivation

### Stress Corrosion Cracking

After 1000 hours of the salt spray exposure, the samples were removed, photographed, and microscopically inspected for signs of stress corrosion cracking.



**Performs as well or better than control process**

Alloy	Passivation	Stress Corrosion Cracking
AL6XN	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
17-4PH	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
286	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
304	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
17-7PH	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
410	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
155	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
321	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	
316	Citric	Microscopic evaluations showed that none of the samples showed any signs of stress corrosion cracking
	Nitric	

## Alternative to Nitric Acid Passivation

### Fatigue

- 17-4PH alloy only
- Specimen with Continuous Radial Bends Between Ends; Subjected to a Constant Amplitude, Periodic Forcing Function in Air at Room Temperature
- Stress loads and cycles selected for each substrate were based on historical S-N Curve data in air at ambient temperature.



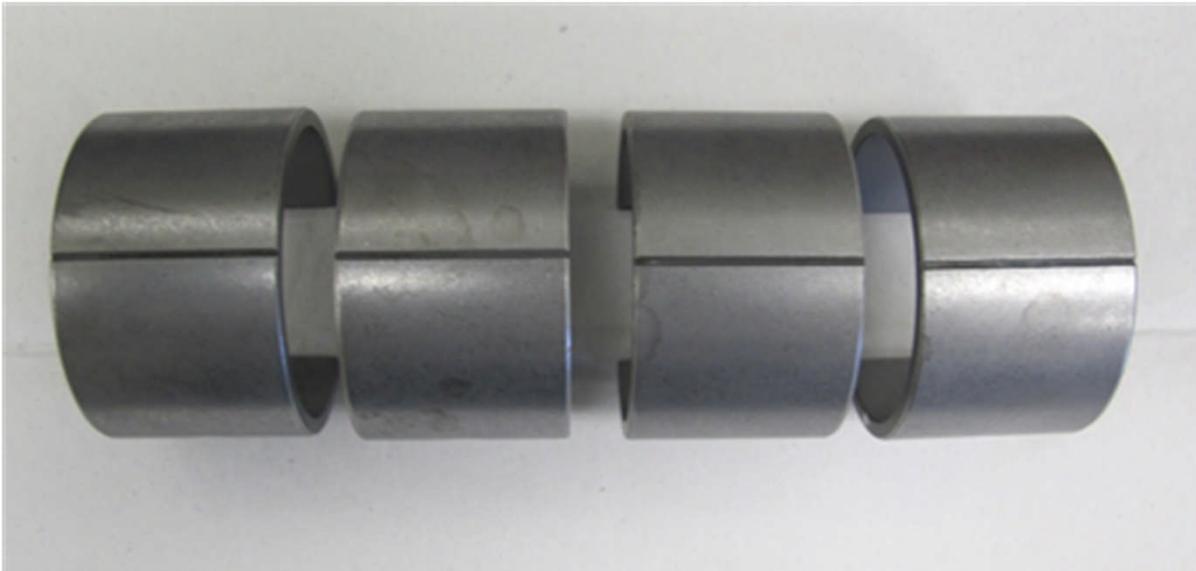
# Alternative to Nitric Acid Passivation

**Fatigue**

Testing On-going

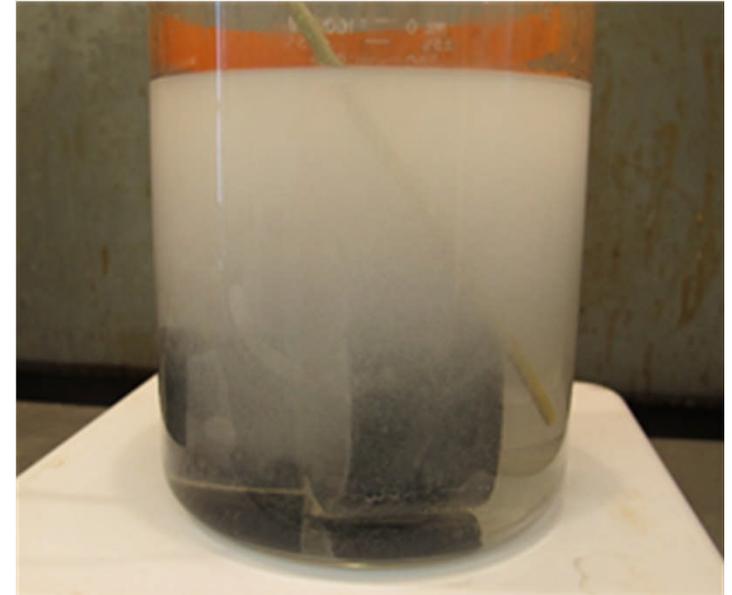
## Alternative to Nitric Acid Passivation

### Hydrogen Embrittlement



Cleaned ASTM F519-13 {Type 1D C-ring - AISI 4340 alloy steel}  
Test Specimens Prior to Passivation

Upon removal from the citric acid bath, it was noticed that the C-Rings were covered with a glossy black film (magnetite). This film remained after the C-Rings were rinsed with deionized water



4% citric acid solution at 82°C for 2  
hours



## Alternative to Nitric Acid Passivation

### Hydrogen Embrittlement

- 2.45 turns of the bolt were required to produce a 75% (to failure) loading. All C-Rings were compressed by 2.45 turns of the nut on the 1/4" – 28 steel bolt to produce the C-Rings for evaluation for potential cracking. All four samples were exposed under ambient condition in the laboratory.
- After 200 hours of exposure to ambient laboratory conditions, no fractures due to hydrogen embrittlement were visible on any sample {Type 1D C-ring - AISI 4340 alloy steel}



# Alternative to Nitric Acid Passivation

## Kourou Exposure Test Campaign



## Alternative to Nitric Acid Passivation

### Test Panels @ ESA

Alloy	Passivation	Number of Panels
304	Nitric	3
	Citric	3
316	Nitric	3
	Citric	3



## Alternative to Nitric Acid Passivation



# **Alternative to Nitric Acid Passivation**

ESA Update

## **Alternative to Nitric Acid Passivation**

### Next Steps

- Additional process optimization = only evaluated citric acid @ 4% concentration
  - It is suggested that the NASA Corrosion Technology Laboratory optimize the passivation process for the 17-4 samples
- Long term analysis of pitting of the samples at the NASA Beach Site
- Determine ESA needs and requirements for future testing

# Alternative to Nitric Acid Passivation

## Summary

- Corrosion is an extensive problem that affects the National Aeronautics and Space Administration (NASA) and European Space Agency (ESA).
- The deleterious effects of corrosion result in steep costs, asset downtime affecting mission readiness, and safety risks to personnel.
- The standard practice for protection of stainless steel is passivation.
- Typical passivation procedures call for the use of nitric acid; however, there are a number of environmental, worker safety, and operational issues associated with its use.
- Citric acid removes iron from the surface more efficiently than nitric acid and therefore uses much lower concentrations reducing material costs.
- There are no toxic fumes created during the citric acid passivation process making it safer for workers.
- For a citric acid passivation concentration of 4%, the stainless steel alloys tested performed as well, and in some cases better than nitric acid passivated panels.
- NASA and ESA will collaborate on a joint project to evaluate citric acid passivation of stainless steel alloys.