

Alternative to Nitric Acid Passivation of Stainless Steel Alloys

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ABSTRACT

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

The DoD and NASA have numerous structures and equipment that are fabricated from stainless steel. The standard practice for protection of stainless steel is a process called 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 offers a variety of benefits including increased safety for personnel, reduced environmental impact, and reduced operational cost.

DoD and NASA agreed to collaborate to validate citric acid as an acceptable passivating agent for stainless steel. This paper details our investigation of prior work developing the citric acid passivation process, development of the test plan, optimization of the process for specific stainless steel alloys, on-going and planned testing to elucidate the process' resistance to corrosion in comparison to nitric acid, and preliminary results.

Key words: passivation, stainless steel, nitric acid, citric acid, corrosion

INTRODUCTION

The costs associated with corrosion are varied and great; not only financially, but with respect to asset downtime, worker safety, and environmental risks. Therefore, it is imperative that the DoD and NASA

consider all contributing factors when making decisions regarding the protection of valuable assets. Because of the harsh environments to which the assets are subject to, both DoD and NASA require assets to be fabricated from stainless steel for the protection from the deleterious effects of corrosion.

The most common method used to increase the corrosion protection afforded by stainless steel is passivation. Passivation is defined by *The American Heritage Dictionary of the English Language* as "to treat or coat (a metal) in order to reduce the chemical reactivity of its surface."¹ Passivation works by forming a shielding outer (metal oxide) layer that reduces the impact of deleterious environmental factors such as air or water. Consequently, this process necessitates a final product that is very clean and free of iron and other contaminants.

The longtime military specification for passivation of stainless steel was Federal Specification QQ-P-35C², which details the specific processing conditions to passivate a stainless steel surface using nitric acid. While nitric acid is very effective at producing a properly passivated surface, there are numerous safety and environmental concerns associated with its use.

1. Nitric acid passivation results in fumes that contain nitrogen oxide (NOx) emissions which are considered greenhouse gases and volatile organic compounds that contribute to smog. There are several federal and state regulations that must be considered and may require permits for the use of nitric acid.³
2. NOx emissions are also dangerous to workers and require the use of general area ventilation or local ventilation of the passivation tank.³
3. Wastewater generated from the passivation process is regulated under the United States (U.S.) Environmental Protection Agency's (EPA) Metal Finishing Categorical Standards (40 CFR 433⁽¹⁾) as well as your wastewater treatment facility and may require permits and pretreatment prior to discharge.³
4. Nitric acid can remove beneficial heavy metals (nickel, chromium, etc.) that give stainless steel its desirable properties.⁴

The QQ-P-35 specification was cancelled in favor of ASTM⁽²⁾ A 967⁵ and Aerospace Material Specifications (AMS) 2700⁶, both of which allow for the use of citric acid in place of nitric acid. Citric acid is similarly called out in the ASTM A 380⁷ standard. In addition, many Services/Agencies have internal requirements for the processing of stainless steel parts using citric acid.

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. It was determined that nitric acid passivation could not provide the degree of passivation required. Other industries in the U.S. have only recently begun using citric acid.⁸ There are a variety of benefits from the substitution of citric acid for nitric acid for passivation.

1. 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⁽³⁾, Executive Order (EO) 13423 (Strengthening Federal Environmental, Energy, and Transportation Management)⁽⁴⁾ and EO 13514 (*Federal Leadership in Environmental, Energy, and Economic Performance*)⁽⁵⁾.⁹
2. There are no toxic fumes created during the citric acid passivation process making it safer for workers.⁴
3. Citric acid removes iron from the surface more efficiently than nitric acid and therefore uses much lower concentrations reducing material costs.⁴

⁽¹⁾ "Metal Finishing Point Source Category," Title 40 *Code of Federal Regulations*, Pt. 433. 2005 ed.

⁽²⁾ ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

⁽³⁾ Farm Security and Rural Investment Act of 2002, Pub. L. No. 107-171, § 9002, 116 STAT. 134 (2002).

⁽⁴⁾ Exec. Order No. 13,423, 72 Fed. Reg. 17 (Jan. 26, 2007).

⁽⁵⁾ Exec. Order No. 13,514, 74 Fed. Reg. 194 (Oct. 8, 2009).

4. 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.⁴

While citric acid use has become more prominent in industry in the U.S. and the benefits are well documented, there is little evidence that citric acid is a technically sound passivating agent, especially for the unique and critical applications encountered by DoD and NASA.

In 2008, NASA's Materials Advisory Working Group requested that United Space Alliance (USA), Ground Operations at NASA John F. Kennedy Space Center (KSC), evaluate a procedure that employs citric acid as a replacement for nitric acid passivating baths. USA began work to investigate the corrosion resistance afforded by citric acid passivation and optimize the processing parameters for the process. USA concluded that citric acid most likely performs as well or better than nitric acid and that in general, high temperature, low concentration, and longer processing time provided the optimal level of corrosion resistance.¹⁰

The USA study included a limited number of alloys and only considered corrosion resistance. Yet there are many other concerns that must be evaluated prior to acceptance of the citric acid process. Therefore, DoD and NASA decided to build off of the USA effort and work together to demonstrate the possibility of using citric acid for the passivation of a variety of stainless steel alloys typically used by the Agencies.

This paper highlights testing of the citric acid passivation process on stainless steel alloys that are frequently used in the field by the stakeholders (DoD and NASA). The project aims to investigate the process for a set of 10 alloys, but due to limitations due to incremental funding associated with the project, only four (4) of the alloys have been tested as of the writing of this paper. This paper reports on the testing protocol that is required for the full set of 10 alloys, and details the results for the four (4) alloys that have been investigated to date.

EXPERIMENTAL PROCEDURE

A joint task group consisting of technical representatives from the DoD and NASA worked together to reach technical consensus on alloys of interest and performance requirements. This information is documented in the *Joint Test Protocol for Validation of Citric Acid as an Alternative to Nitric Acid for Passivation of Stainless Steels*.¹¹

Selection of Alloys

The project stakeholders identified a variety of stainless steel alloys from which their assets are typically fabricated. The alloys that were selected for testing are shown in Table 1.

Table 1
Stainless Steel Alloys Selected for Demonstration

Type	Alloy	Unified Numbering System (UNS) ⁽⁶⁾ Number
Super Austenitic	AL-6XN	N08367
200 Series Austenitic	A286	S66286
300 Series Austenitic	304	S30400
300 Series Austenitic	316	S31600
300 Series Austenitic	321	S32100

⁽⁶⁾ Unified Numbering System for Metals and Alloys (UNS). UNS numbers are listed in Metals & Alloys in the Unified Numbering System, 11th ed. (Warrendale, PA: SAE International and West Conshohocken, PA: ASTM International, 2008).

Type	Alloy	Unified Numbering System (UNS) ⁽⁶⁾ Number
400 Series Martensitic	410	S41000
400 Series Martensitic	440C	S44004
Precipitation-Hardened Martensitic	15-5PH	S15500
Precipitation-Hardened Martensitic	17-4PH	S17400
Precipitation-Hardened Martensitic	17-7PH	S17700

Performance Requirements

The joint group defined critical tests with procedures, methodologies, and acceptance criteria necessary to qualify citric acid passivation for use by the respective Agencies (Table 2).

Table 2
Performance Requirements, Acceptance Criteria, and Test Methodology

Test	Acceptance Criteria	Test Methodology References
Parameter Optimization Testing	Best performing parameters	ASTM B 117 ¹² ASTM D 610 ¹³
X-Cut Adhesion by Wet Tape	Alternative performs as well or better than control process	ASTM D 3359 ¹⁴
Tensile (Pull-off) Adhesion		ASTM D 4541 ¹⁵
Cyclic Corrosion Resistance		GMW 14872 ¹⁶
Atmospheric Exposure Testing		ASTM D 610 ASTM D 714 ¹⁷ NASA-STD-5008 ¹⁸
Stress Corrosion Cracking		ASTM B 117 ASTM E 4 ¹⁹ ASTM E 8 ²⁰ ASTM G 38 ²¹ ASTM G 39 ²² ASTM G 44 ²³ MSFC-STD-3029 ²⁴
Fatigue		ASTM E 466 ²⁵
Hydrogen Embrittlement		ASTM F 519 ²⁶
Liquid Oxygen (LOX) Compatibility	Twenty samples must not react when impacted at 72 ft-lbs (98 J). If one sample out of 20 reacts, 40 additional samples must be tested without any reactions.	NASA-STD-6001 ²⁷

Test Specimen Preparation

The cleaning and degreasing procedures were based upon those used by USA in their passivating activities for aerospace flight hardware. Figure 1 outlines the panel preparation and passivation process for this effort. Test specimens were processed the same in all respects other than the actual

passivation. Citric acid time/temperature passivation conditions were optimized as part of this experimental effort. Nitric acid passivation conditions were employed per Federal Specification QQ-P-35C (et al.). Finally, the final rinse, pH verification, and drying of the samples were performed according to the USA process for aerospace flight hardware.

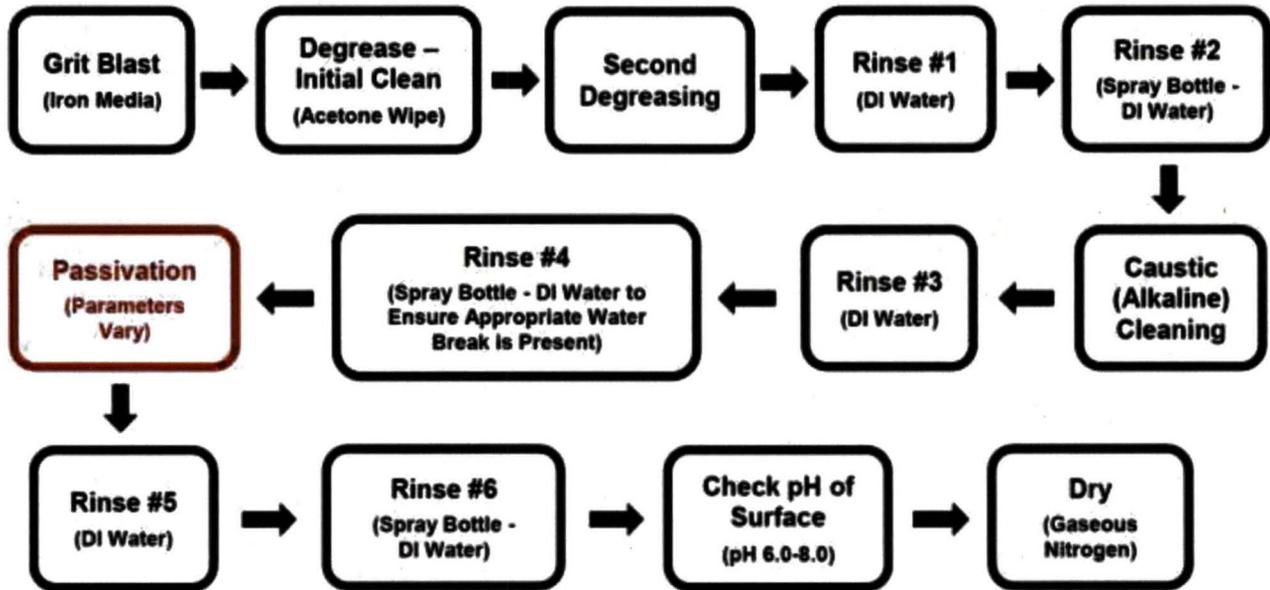


Figure 1: Process flow diagram detailing the preparation of test specimens

The citric acid passivation procedure varied by alloy. Previous USA testing showed that different citric acid time/temperature/concentration process conditions resulted in varying degrees of corrosion resistance. Therefore, Parameter Optimization was imperative to ascertain the appropriate citric acid processing conditions to determine which process parameters resulted in superior performance. Those alloys included in the USA testing did not undergo this test; their processing parameters were based on USA results. The optimized parameters were then used to prepare panels for the remainder of testing.

One side of each panel was blasted with a fine steel grit to impart contamination to the surface. Perfectly clean panels would be unlikely to show much corrosion within the testing period. The steel grit also removed the protective passivation layer provided by the manufacturer for storage and transport. Consequently, steel grit was used for the blasting process to leave a smooth finish on the surface of the metal.

After grit blasting, the panels were carefully cleaned through the series of degreasing, cleaning, and rinsing steps shown in Figure 1. Initially, acetone was used to remove excess steel grit and grease. This step prevented unwanted contamination of the cleaning and passivating tanks.

A second degreasing was performed with a cleaner in deionized (DI) water. The panels were removed from the degreaser and subsequently placed into a heated DI water bath to remove excess contaminants and the degreasing agent. The panels were then rinsed using an ambient temperature DI water spray.

Following the second rinse, a caustic cleaning was performed. The panels were then rinsed in a heated DI water trough, followed by a DI spray-down with ambient temperature DI water. In order to verify that the degreasing and cleaning steps were performed successfully prior to passivation, the water break of the final rinse was inspected to ensure cleanliness of the product.

Cleaned panels were placed in passivation baths containing either nitric acid or citric acid solutions using varying parameters. Ordinary bath trays (using covers to limit evaporation) were used. After each passivation was performed, rinses were conducted according to the USA process. After the rinsing process was performed, the pH of the water on the surface of each panel was checked to ensure that it remained at a pH between 6.0 to 8.0. Ordinary pH paper was used to perform this function. The pH is typically of concern when a high volume of panels is being processed due to the increased possibility of residual passivation solution contaminating the rinse tanks. Although this study processed a relatively small number of panels, this step was still included. Verifying that this parameter was met, the panels were dried with gaseous nitrogen.

RESULTS

All testing for this effort has not been completed; therefore, the following gives only data gathered to date. Of the ten (10) identified alloys, this paper focuses on the testing of the following alloys:

- UNS N08367
- UNS S66286
- UNS S30400
- UNS S17400

Parameter Optimization Testing

As a result of previous work by USA, it was determined that the parameters for citric acid passivation resulted in various levels of corrosion related performance as a function of the substrate. The data obtained during the original USA study was used for the preparation of test specimens composed of those alloys. Parameter optimization testing, however, was necessary to determine the best parameters for the alloys that were not part of the USA study.

The USA study determined that a citric acid concentration of 4% provided optimum performance for all substrates and therefore a 4% concentration shall be used for the optimization testing in this test plan. The USA study also found that treatments with elevated temperature and longer immersion times (temperature > 38 °C and time > 30 minutes) provided significantly better corrosion protection than treatments under ambient conditions.

Based on available information, the following parameters were used to evaluate the best possible combination of immersion time and temperature conditions:

- Time: 60 minutes, 90 minutes, and 120 minutes
- Temperature: 38 °C, 60 °C, and 82 °C

After citric acid passivation was performed using the appropriate time/temperature conditions, test specimens were placed in a salt fog chamber and tested in accordance with ASTM B 117. Test specimens were removed from the salt fog chamber and evaluated every 168 hours.

Stage I of this effort only included four (4) of the alloys listed in Table 1. The USA effort included UNS S30400 and UNS S17400 so those alloys were not included in the Parameter Optimization Testing; this testing only included UNS N08367 and UNS S66286.

After exposure in a salt fog chamber for 504 hours, there were no distinguishable differences in corrosion performance in UNS N08367. None of the samples showed signs of corrosion on the surface of the samples. Figure 2 illustrates the lack of corrosion to the face of the UNS N08367 panels as a function of the shortest (60 min.) and longest (120 min.) processing times with the 4% citric acid bath at 38 °C.

Both processing times would be suitable for the passivation of the stainless steel alloy, though consideration was given to prior testing for the UNS S30400 alloy, which is also an austenitic stainless steel. USA process optimization for that alloy indicated that the 120-minute passivation procedure worked best. Consequently, the same processing time and temperature was chosen for UNS N08367 as well (38 °C and 120 minutes).

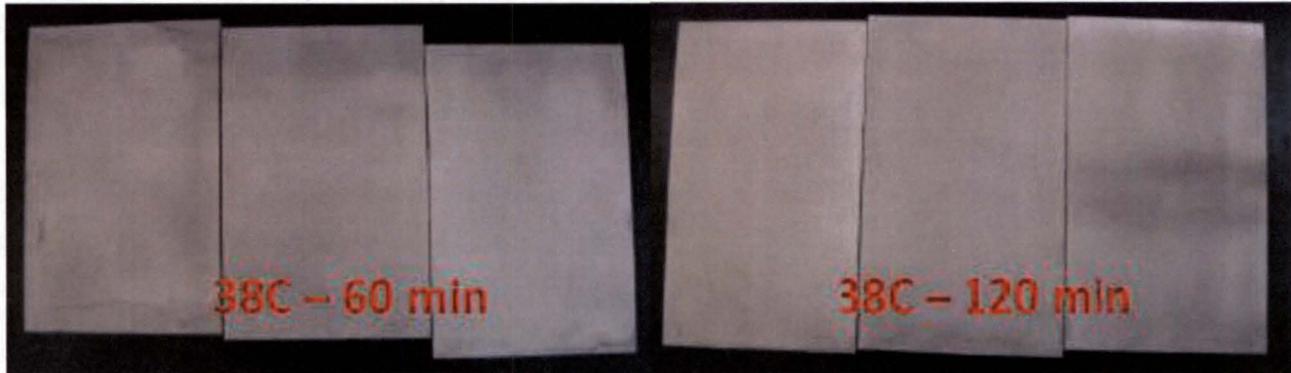


Figure 2: UNS N08367 test specimens after 504 hours ASTM B 117 exposure

Citric acid optimization for the UNS S66286 indicated that processing time had little effect on the corrosion related discoloration on the sample surface. As shown in Figure 3, the 60-minute and 120-minute processing times exhibited little difference in appearance at 38 °C. The same held true for the samples processed at 82 °C. Conversely, the 82 °C processing conditions showed a reduction in discoloration in comparison to the panels processed at 38 °C. Consequently, the recommendation was made to passivate all UNS S66286 samples in the 4% citric acid solution at 82 °C for 60 minutes.

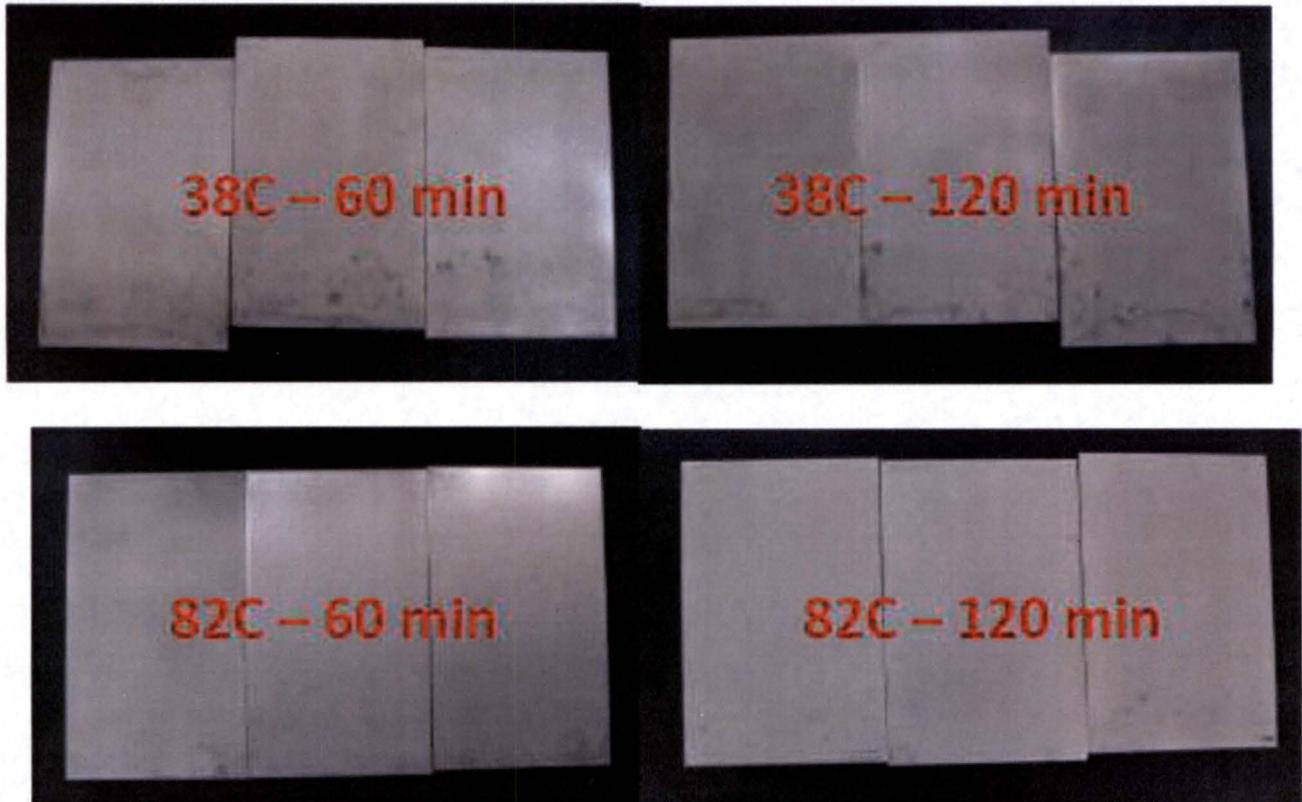


Figure 3: UNS S66286 test specimens after 504 hours ASTM B 117 exposure

Following Parameter Optimization Testing, the remainder of testing for the four (4) alloys for Stage 1 of the project was prepared using the processing parameters given in Table 3. Nitric acid processing conditions were based upon Federal Specification QQ-P-35C (et al.).

Table 3
Process Parameters Used for Current Testing

Alloy	Passivation	Concentration (%)	Bath Temperature (°C)	Dwell Time (minutes)
UNS N08367	Nitric Acid	22.5	66	20
	Citric Acid	4	38	120
UNS S66286	Nitric Acid	50	64	30
	Citric Acid	4	82	60
UNS S30400	Nitric Acid	22.5	66	20
	Citric Acid	4	49	120
UNS S17400	Nitric Acid	50	64	30
	Citric Acid	4	38	30

Tensile (Pull-off) Adhesion

Adhesion was added to the test matrix because one of the project team members from the Naval Research Laboratory noted that during their work with citric acid, the citric acid passivated panels appeared to show lower adhesion values than nitric acid passivated panels. Further research was not conducted at that time, but it was suggested that citric acid might leave an organic film on the surface that affects adhesion of subsequently applied coatings.

A variety of coating types were identified by project team members including standard liquid coatings, chemical agent resistant coating (CARC) systems, cadmium plating, and chromium plating. Thus far, only a liquid primer from the Approved Products List in NASA-STD-5008 has been tested on the four (4) alloys included in this stage of testing.

Panels of each alloy were passivated with either citric acid or nitric acid and coated with a primer from the Approved Products List in NASA-STD-5008. Adhesion values were determined using a Pneumatic Adhesion Tensile Testing Instrument (PATTI) per ASTM D 4541 using dollies that were affixed to the panel surface using a two-part epoxy. The testing instrument gave a burst pressure value that was then converted to an adhesion value (PSI).

Except for two of the nitric acid passivated panels, all pull-off values were strictly related to the epoxy adhesive, meaning that the adhesive failed prior to the coating as shown in Figure 4. There was no evidence that the citric acid passivation process is detrimental to the adhesion of a liquid coating on the alloys tested; adhesion results are shown in Table 4.



Figure 4: Typical epoxy failure while coating remained intact

**Table 4
Tensile (Pull-Off) Adhesion Testing Results**

System	BP ⁽⁷⁾	PSI ⁽⁸⁾	Average PSI	Failure Mode	Relative % Difference ⁽⁹⁾
UNS N08367 Citric	42.7	1737	2101	100% Glue	29
	58.6	2386		100% Glue	
	69.4	2827		100% Glue	
UNS N08367 Nitric	75.8	3088	2969	95% Glue – 5% Primer	10
	64.8	2639		95% Glue – 5% Primer	
	67	2729		100% Glue	
UNS S66286 Citric	73.3	2986	2731	100% Glue	19
	61.4	2501		100% Glue	
	54.5	2219		100% Glue	
UNS S66286 Nitric	36.4	1480	1671	100% Glue	27
	57	2321		100% Glue	
	50.4	2052		100% Glue	
UNS S30400 Citric	61.1	2488	2550	100% Glue	10
	71.4	2909		100% Glue	
	65.6	2672		100% Glue	
UNS S30400 Nitric	66.5	2709	2561	100% Glue	18
	50	2035		100% Glue	
	55.6	2264		100% Glue	
UNS S17400 Citric	62	2525	2550	100% Glue	4
	66	2688		100% Glue	
	63.8	2599		100% Glue	
UNS S17400 Nitric	55.5	2260	2231	100% Glue	4
	52.5	2137		100% Glue	
	53.4	2174		100% Glue	

Atmospheric Exposure Testing

This is a real-world exposure test in which treated test panels are placed at the NASA Corrosion Technology Laboratory's KSC Beachside Atmospheric Test Facility. Test specimens are located approximately 150 feet from the Atlantic Ocean high tide line. All KSC test rack procedures for fasteners and exposure angle are as stated in NASA-STD-5008.

Test panels included Passivated-only and Passivated-Coated specimens. The Passivated-Coated specimens used the same primer as that for the tensile adhesion testing and a topcoat from the Approved Products List in NASA-STD-5008.

Exposure was initiated on 10/11/2012. Both the Passivated-only and the Passivated-Coated test panels are shown in Figure 5.

⁽⁷⁾ Adhesion Tester Reading

⁽⁸⁾ Converted Adhesion Value

⁽⁹⁾ Relative % Difference = Percentage difference between values for each alloy/passivation set



Figure 5: Test panels at KSC Beachside Atmospheric Test Facility

Passivated-Coated test coupons have been evaluated per ASTM D 610 and ASTM D 714 with no signs of corrosion evident after six (6) months of coastal atmospheric exposure.

Passivated-only specimens are evaluated and photographed on a regular basis until failure. Failure is defined as a Rust Grade rating of "8" as described by Table 1, *Scale and Description of Rust Grades*, in ASTM D 610. Clearly, several of the panels failed prior to the one (1) month exposure according to this criterion. Consequently, this stage will be repeated with evaluation performed at more frequent intervals. The ratings after exposure of one (1), three (3), and six (6) months are shown in Table 5. Clearly, the citric acid passivated panels exhibited equal or better performance throughout the duration of exposure.

**Table 5
Passivated-only Test Panel Rust Grade Ratings**

Alloy	Passivation	1 Month Ranking	3 Month Ranking	6 Month Ranking
AL6XN	Citric	10	10	10
	Citric	8	7	7
	Citric	8	8	7
	Nitric	8	8	7
	Nitric	6	6	6
	Nitric	8	8	7
A286	Citric	6	5	5
	Citric	6	5	5
	Citric	6	5	5
	Nitric	3	1	1
	Nitric	6	5	4
	Nitric	6	5	4

Alloy	Passivation	1 Month Ranking	3 Month Ranking	6 Month Ranking
304	Citric	5	5	3
	Citric	5	5	3
	Citric	5	5	3
	Nitric	4	4	2
	Nitric	4	4	2
	Nitric	4	4	2
17-4PH	Citric	4	3	3
	Citric	4	3	3
	Citric	4	3	3
	Nitric	3	3	2
	Nitric	4	3	3
	Nitric	4	3	3

Stress Corrosion Cracking

Stress corrosion cracking can lead to sudden failure of normally ductile metals subjected to a tensile stress. Project team members felt that it was important to determine whether citric acid could increase the chances of stress corrosion cracking. Samples meeting ASTM G 58 were purchased, blasted, and passivated alongside flat panel specimens. The samples were stressed after passivation and placed at the KSC Beachside Atmospheric Test Facility for atmospheric exposure beginning on 10/11/2012. After six (6) months of exposure, there has been no evidence of cracking as is indicated from the six (6)-month photodocumentation of the specimens as shown in Figure 6.

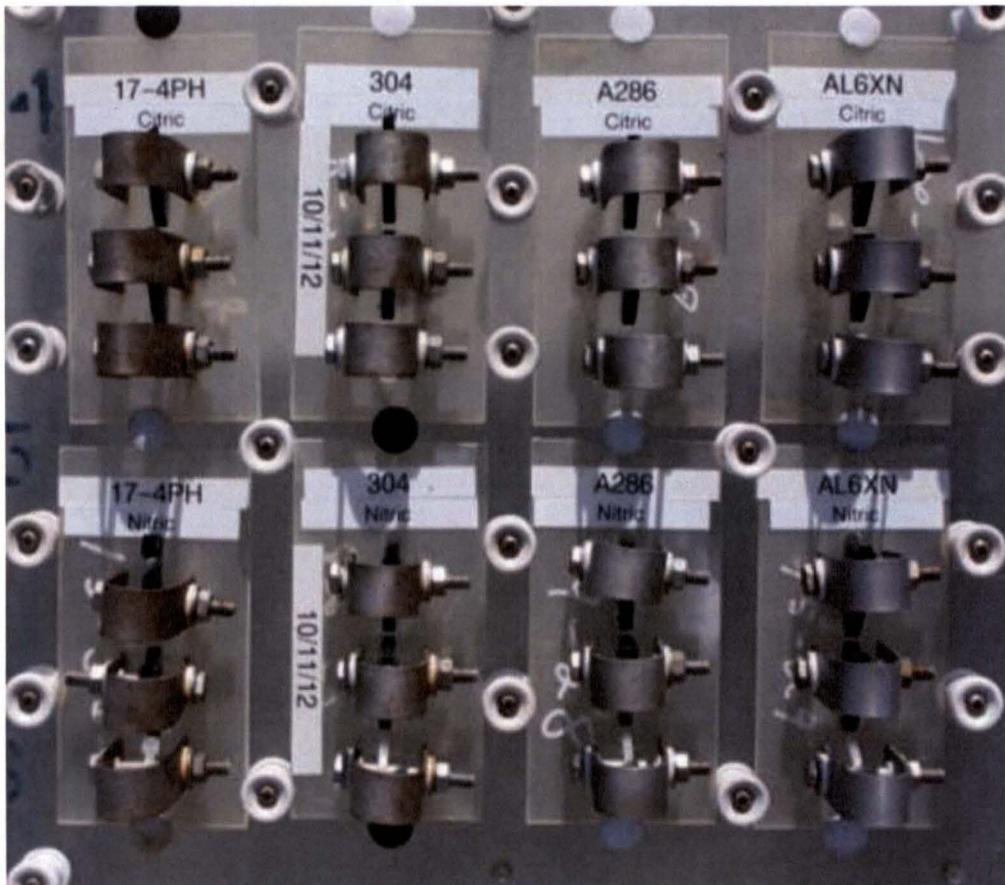


Figure 6: Stress corrosion cracking specimens after six (6) months of exposure at KSC Beachside Atmospheric Test Facility

CONCLUSIONS

The first test, Parameter Optimization Testing, was required to determine whether process parameters affected the performance of citric acid for UNS N08367 and UNS S66286. A variety of temperature and bath immersion times were included. No corrosion related staining was evident on any UNS N08367 specimen. An increase in corrosion related performance was seen for UNS S66286 as a function of temperature. Optimal processing conditions were determined.

The citric acid passivation process was tested against the nitric acid process to determine whether citric acid may affect adhesion of subsequent coatings. A liquid primer was applied to test panels and evaluated using a standard pull-off adhesion tester. It was found that the citric acid passivation process had no derogatory effect on the adhesion of the primer to the panel.

Test panels were also prepared and placed at the KSC Beachside Atmospheric Test Facility. Test panels included Passivated-only and Passivated-Coated (primer and topcoat). After six (6) months of exposure, the Passivated-only test panels showed that the citric acid panels actually had an equal or lesser degree of corrosion on the surface than the nitric acid passivated panels. There were no signs of corrosion on any of the Passivated-Coated test panels.

A set of passivated stress corrosion cracking specimens were also placed at the KSC Beachside Atmospheric Test Facility for atmospheric exposure. None of the samples cracked after six (6) months of exposure.

At this point, it appears that citric acid passivation process performs as well as, or better than, the nitric acid passivation process according to the testing employed. The citric acid process also exhibits environmental, safety, and cost benefits in preference to passivation via the traditional nitric acid procedure.

Future Work

Work is on-going in the continued testing of the alloys included in this paper, as well as the other alloys identified. Additional testing includes:

- X-Cut Adhesion
- Cyclic Corrosion Resistance
- Fatigue Testing
- Hydrogen Embrittlement

ACKNOWLEDGEMENTS

We wish to acknowledge the invaluable contributions provided by all the organizations involved in the support of this project and the creation of this document.

The following organizations provided support for this project:

- NASA Headquarters Environmental Management Division
- NASA Ground Systems Development and Operations Program

The following people provided technical support for this project:

- NASA Corrosion Technology Laboratory

Dr. Mark Kolody
Mr. Jerry Curran

Ms. Lashelle McCoy
Mr. Teddy Back

Dr. Luz Marina Calle

- NASA John F. Kennedy Space Center

Ms. Jennie Ward	Ms. Jennifer Van Den Driessche	Ms. Diana Hawkins
Mr. Jeff Sampson	Mr. James Blake	Ms. Terry Keeney
Mr. Chuck Griffin	Ms. Kai Ross	Ms. Pattie Lewis
- NASA Lyndon B. Johnson Space Center

Mr. Mike Pedley		
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- NASA George C. Marshall Space Flight Center

Mr. Cliff Watson	Mr. Eddie Davis	Mr. Ethan Chew
Mr. Dave Vermilion	Mr. Matt Frisby	Mr. Stephen Herald
Mr. Mike Watwood	Mr. Matt Segars	
- NASA White Sands Test Facility

Ms. Sandy Ruttle	Ms. Christina Piña Arpin	Mr. Clifford Madrid
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- NASA Wallops Flight Facility

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- NASA John C. Stennis Space Center

Ms. Laurie Green	Mr. Josh Craft	Mr. Anthony Jackson
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- NASA Goddard Space Flight Center

Mr. Ben White	Mr. John Wolfe	
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- United Space Alliance (USA)

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