NASA Technology Evaluation for Environmental Risk Mitigation Kennedy Space Center, FL 32899

NASA and ESA Collaboration on Hexavalent Chrome Alternatives

Pretreatments with Primers Screening FINAL Test Report May 15, 2015

NASA Contract: NNH15CM58Z Task Order: NNH15CN36D

ITB, Inc. 3700 N. Courtenay Pkwy Suite 112 Merritt Island, FL 32953



ITB, Inc. 7333 Paragon Road Suite 250 Dayton, OH 45459

TABLE OF CONTENTS

Pre	face		iii						
Acl	know	ledgements	iv						
Exe	ecutiv	ve Summary	. 5						
1	I Introduction								
	1.1 Background								
	1.2	Objective	. 7						
	1.3	Summary of Previous NASA TEERM Projects	. 7						
2	Test	Articles	. 8						
	2.1	Alloys	. 8						
	2.2	Pretreatments	. 8						
	2.3	Primers	. 8						
3	Pretr	eatment with Primer Screening	. 9						
	3.1	Method	. 9						
	3.2	Alloys	. 9						
	3.3	Pretreatment with Primers Screening Matrix	. 9						
	3.4	Salt Spray Resistance	. 9						
		3.4.1 Test Procedure	. 9						
		3.4.2 Evaluation Procedure	10						
		3.4.3 Screening Test Results	10						
	3.5	PATTI Pull Test	12						
		3.5.1 Test Procedure	12						
		3.5.2 Evaluation Procedure	13						
		3.5.3 Screening Test Results	14						
4	Conc	clusions	17						
5	Reco	ommendations	17						
Ap	pendi	x A – Salt Spray Resistance Evaluation Criteria	18						
Ap	pendi	x B – Pretreatment with Primer Screening Salt Spray Resistance Results	19						
Ap	Appendix C – Pretreatment with Primer Screening Salt Spray Resistance Test Panel Photos 21								
Ap	pendi	x D – Pretreatment with Primer Screening PATTI Pull Test	32						

LIST OF TABLES

Table 1 – Pretreatments	8
Table 2 – Primers	8
Table 3 – Pretreatment with Primer Screening	9
Table 4 – Pretreatment with Primer Screening Matrix	9
Table 5 – Salt Spray Screening Test Results: Pretreatment and Primer (2,100 hours) 1	1
Table 6 - Salt Spray Screening Test Results: Pretreatment and Primer (2,100 hours) 1	1
Table 7 – Salt Spray Screening Test Results: Pretreatment and Primer (2,100 hours) 1	2
Table 8 - Salt Spray Screening Test Results: MAPSIL® SILICo {12 to 16 µm} (555 hours) 1	2
Table 9 – PATTI Pull Screening Test Results: Pretreatment and Primer 1	5
Table 10 – PATTI Pull Screening Test Results: MAPSIL® SILICo $\{12 \text{ to } 16 \ \mu m\} \ldots 1$	6

Preface

This test report was prepared by ITB, Inc. through the National Aeronautics and Space Administration (NASA) Technology Evaluation for Environmental Risk Mitigation (TEERM) Principal Center under Contract Number NNH15CM58Z. NASA TEERM determined the structure, format, and depth of technical content of the report in response to the specific requirements of this project.

NASA TEERM has partnered with the European Space Agency (ESA) for this project. NASA and ESA share common risks related to material obsolescence associated with hexavalent chromium used in corrosion-resistant coatings.

NASA TEERM works closely with the John F. Kennedy Space Center (KSC) Corrosion Technology Laboratory to ensure the quality of the test data, including the review and analysis within this report. Similarly, ESA works with the European Space Research and Technology Centre (ESTEC).

Acknowledgements

ITB, Inc. would like to thank Mr. Thomas Rohr with ESA ESTEC for continued project support and contribution. ITB, Inc. would also like to thank Mr. Jerry Curran for coordination and oversight of the KSC Corrosion Technology Laboratory and the KSC Beachside Atmospheric Test Facility, as well as Ms. Fatema Janjali for coordination and testing support at ESA ESTEC. In addition, ITB, Inc. would like to thank Mr. Teddy Back for test panel preparation and coating application at KSC and Mr. Guillaume Sierra for coating application at MAP.

Executive Summary

Hexavalent chromium (hex chrome or Cr(VI)) is a widely used element within applied coating systems because of its self-healing and corrosion-resistant properties. The replacement of hex chrome in the processing of aluminum for aviation and aerospace applications remains a goal of great significance. Aluminum is the major manufacturing material of structures and components in the space flight arena. The National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA) are engaged in a collaborative effort to test and evaluate alternatives to hexavalent chromium containing corrosion-coating systems. NASA and ESA share common risks related to material obsolescence associated with hexavalent chromium used in corrosion-resistant coatings.

In the United States, Occupational Safety and Health Administration (OSHA) studies have concluded that hexavalent chromium is carcinogenic and poses significant risk to human health. On May 5, 2011, amendments to the Defense Federal Acquisition Regulation Supplement (DFARS) were issued in the Federal Register. Subpart 223.73 prohibits contracts from requiring hexavalent chromium in deliverables unless certain exceptions apply. Subpart 252.223-7008 provides the contract clause prohibiting contractors and subcontractors from using or delivering hexavalent chromium in a concentration greater than 0.1 percent by weight for all new contracts associated with supplies, maintenance and repair services, and construction materials.

ESA faces its own increasingly stringent regulations within European directives such as Registration, Evaluation, Authorisation and Restriction of Chemical (REACH) substances and the Restriction of Hazardous Substances Directive (RoHS) which have set a mid-2017 sunset date for hexavalent chromium.

NASA and ESA continue to search for an alternative to hexavalent chromium in coatings applications that meet their performance requirements in corrosion protection, cost, operability, and health and safety, while typically specifying that performance must be equal to or greater than existing systems.

The overall objective of the collaborative effort between NASA TEERM and ESA is to test and evaluate coating systems (pretreatments, pretreatments with primer, and pretreatments with primer and topcoat) as replacements for hexavalent chrome coatings in aerospace applications. This objective will be accomplished by testing promising coatings identified from previous NASA, ESA, Department of Defense (DOD), and other project experience. Additionally, several new materials will be analyzed according to ESA-identified specifications.

1 Introduction

1.1 Background

Hexavalent chromium (hex chrome or Cr(VI)) is a widely used element within applied coating systems because of its self-healing and corrosion-resistant properties. The replacement of hex chrome in the processing of aluminum for aviation and aerospace applications remains a goal of great significance. Aluminum is the major manufacturing material of structures and components in the space flight arena. The processing and maintenance of this material against degradation and corrosion is of prime importance to the National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA) in preserving space operation capabilities.

Key to the operability and preservation of aluminum has been the use of chromated systems (pretreatments and primers). With applied coatings, the high corrosion resistance offered by chromated films is attributed to the presence of both hexavalent and trivalent chromium in the coating. The trivalent chromium (tri chrome or Cr(III)) is present as an insoluble hydrated oxide. Hex chrome imparts a "self-healing" characteristic to the coating during an oxidative (corrosive) attack. Chromated coatings also play a critical role in supporting and enhancing the adhesion of the primer coating to the substrate.

In the United States, Occupational Safety and Health Administration (OSHA) studies have concluded that hexavalent chromium is carcinogenic and poses significant risk to human health. On May 5, 2011, amendments to the Defense Federal Acquisition Regulation Supplement (DFARS) were issued in the Federal Register. Subpart 223.73 prohibits contracts from requiring hexavalent chromium in deliverables unless certain exceptions apply. These exceptions include authorization from a level no lower than a general or flag officer or a member of the Senior Executive Service from the Program Executive Office or equivalent level, or unmodified legacy systems. Otherwise, Subpart 252.223-7008 provides the contract clause prohibiting contractors and subcontractors from using or delivering hexavalent chromium in a concentration greater than 0.1 percent by weight for all new contracts associated with supplies, maintenance and repair services, and construction materials.

ESA faces its own increasingly stringent regulations within European directives such as Registration, Evaluation, Authorisation and Restriction of Chemical (REACH) substances and the Restriction of Hazardous Substances Directive (RoHS) which have set a mid-2017 sunset date for hexavalent chromium.

NASA and ESA continue to search for an alternative to hexavalent chromium in coatings applications that meet their performance requirements in corrosion protection, cost, operability, and health and safety, while typically specifying that performance must be equal to or greater than existing systems.

Please note, this test report includes test data from NASA only. At the time of this report, testing at ESA ESTEC was in progress. ESA will author a report on their findings and a joint combined report will be created at the conclusion of the project.

1.2 Objective

The overall objective of the collaborative effort between NASA and ESA is to test and evaluate coating systems (pretreatments, pretreatments with primer, and pretreatments with primer and topcoat) as replacements for hexavalent chrome coatings in aerospace applications. This objective will be accomplished by testing promising coatings identified from "Hexavalent Chrome Free Coatings for Electronics Applications, Refinement of Coating Processes Report July 31, 2013" and "GSDO Program Hexavalent Chrome Alternatives Final Pretreatments Test Report September 01, 2013" as well as several other coating systems that have shown acceptable performance in previous Department of Defense (DOD) laboratory and atmospheric testing. Additionally, several new materials will be analyzed according to ESA-identified specifications.

1.3 Summary of Previous NASA TEERM Projects

Results from "Hexavalent Chrome Free Coatings for Electronics Applications, Refinement of Coating Processes Report July 31, 2013" and "GSDO Program Hexavalent Chrome Alternatives Final Pretreatments Test Report September 01, 2013" show that hexavalent chrome free pretreatments can perform as good as or better than hexavalent chrome containing pretreatments. In review of the data generated during these projects, hexavalent chrome free pretreatments should be considered for implementation. Based on the results of salt spray resistance testing, multiple hexavalent chrome free pretreatments met the requirements of MIL-DTL-5541 "Chemical Conversion Coatings on Aluminum and Aluminum Alloys".

Results from these previous efforts indicate there is a need to evaluate how primers and topcoats perform over hexavalent chrome free pretreatments. Unexpected interactions between hexavalent chrome free pretreatments, primers, and topcoats could produce a coating system that does not meet specifications. This collaborative effort between NASA and ESA aims to explore these unknowns.

2 Test Articles

2.1 Alloys

The aluminum alloy in this project was selected because of relatively common use in avionics and aerospace applications. All test panels were procured mill finished without mill markings. Mill finish is as supplied from the mill (raw material manufacturer), not polished, and will most likely have a dull matte appearance. The aluminum alloy selected for this project include:

• 2024-T3

2.2 Pretreatments

NASA and ESA selected the hexavalent chrome free pretreatments to be tested for this project. Two of the pretreatments, Bonderite M-NT 65000 and MAPSIL[®] SILICo (thin), were supplied by ESA. MAPSIL[®] SILICo (thin) is approximately 3 micrometers (μ m) to 5 μ m thick when applied to a panel. The selected pretreatments are listed in Table 1.

Table 1 – Pretreatments

Pretreatment	Manufacturer
Metalast TCP HF	Metalast
SurTec 650 V	SurTec
Bonderite M-NT 65000	Henkel
MAPSIL [®] SILICo (3–5 µm)	MAP

2.3 Primers

NASA and ESA selected the hexavalent chrome free primers to be tested for this project. Two of the primers, MAPSIL[®] SILICo (thick) and MAPSIL[®] SILICo AS, were supplied by ESA. MAPSIL[®] SILICo (thick) is approximately 14 μ m to 26 μ m thick when applied to a panel. The selected primers are listed in Table 2.

Table 2 – Primers

Primer	Manufacturer
Deft 02GN084	Deft, Inc.
Hentzen 16708	Hentzen Coatings, Inc.
NAVALCOAT	U.S. Navy
MAPSIL [®] SILICo (14–26 µm)	MAP

3 Pretreatment with Primer Screening

3.1 Method

Test panels with pretreatments and primers underwent screening-level testing (limited testing) to downselect the coating systems to be further tested and evaluated by NASA and ESA. A limited number of test panels were tested through these screening-level tests. Table 3 provides screening test methods, evaluation criteria, and the location of where screening tests were conducted.

\mathbf{z}										
Test	Test Method	Evaluation Criteria	Location							
Salt Spray Resistance	ASTM B 117	MIL-DTL-5541	NASA KSC and ESA ESTEC							
PATTI Pull Test	ASTM D 4541	ASTM D 4541	NASA KSC							

Table 3 –	Pretreatment	with	Primer	Screening

3.2 Alloys

Alloy 2024-T3 was selected for this round of screening. Due to this alloy's high copper content, it is typically hard to protect from corrosion, making it an ideal screening level alloy.

3.3 Pretreatment with Primers Screening Matrix

Table 4 displays the pretreatments and primers screening matrix selected by NASA and ESA.

Pretreatment	Primer					
Metalast TCP	Hentzen 16708					
Metalast TCP	Deft 02GN084					
Metalast TCP	NAVALCOAT					
SurTec 650 V	Hentzen 16708					
SurTec 650 V	Deft 02GN084					
SurTec 650 V	NAVALCOAT					
Bonderite M-NT 65000	Hentzen 16708					
Bonderite M-NT 65000	Deft 02GN084					
Bonderite M-NT 65000	NAVALCOAT					
MAPSIL [®] SILICo $(14-16 \mu m)^{(1)}$						
Note: (1) MAP indicated that MAPSIL [®] SILICo (thick) did not						
require additional pretrea	tment or primer application.					

Table 4 – Pretreatment with Primer Screening Matrix

3.4 Salt Spray Resistance

This test is used to rapidly evaluate the performance of a coating or coating system and how well it prevents corrosion. Salt spray exposure and corrosion resistance is a requirement of MIL-PRF-23377 and MIL-PRF-85582.

3.4.1 Test Procedure

Two intersecting lines shall be scribed diagonally across the coated surface of each panel, exposing the bare substrate. Test panels are then subjected to a 5 percent NaCl salt spray, pH-

NASA TEERM NASA and ESA Pretreatments and Primers Screening Final Test Report adjusted to a range of 6.5 - 7.2, in accordance with ASTM B 117 (Standard Practice for Operating a Salt Spray [Fog] Apparatus) for 2,000 hours.

Salt spray resistance testing was performed at the NASA KSC Corrosion Technology Laboratory and is in progress at ESA ESTEC. This report only provides data from NASA testing at this time.

3.4.2 Evaluation Procedure

Test panels were evaluated using a system used in past NASA TEERM and DOD projects. The evaluation system includes three categories or "digits". Digits include (1) scribe appearance, (2) undercutting/blistering at the scribe, and (3) blistering away from the scribe. All three digits are rated based on a numerical scale from 0 to 5. The second and third digit are also rated based on whether or not blistering is considered isolated. Finally, the third digit is also rated for frequency. The first digit numerical scale ranges from 0 (bright and clean) to 5 (severe corrosion product build up). The second digit numerical scale ranges 0 (no lifting of coating) to 5 (lifting or loss of adhesion beyond ½-inch). The second digit isolation identification is yes, no, or not applicable. The third digit frequency scale ranges from 0 (none) to 5 (large [greater than 13 millimeters]). The third digit frequency scale ranges from few to dense and isolation is yes, no, or not applicable. Please see Appendix A for more details of this rating system.

3.4.3 Screening Test Results

After 2,100 hours of testing, several test panels continued to perform well. Most test panels with Metalast TCP, SurTec 650 V, or Bonderite M-NT 65000 pretreatments maintained a first digit rating of 3 or less. One test panel with Metalast TCP and Hentzen 16708 received a first digit rating of 4. Similarly, all test panels with Metalast TCP, SurTec 650 V, or Bonderite M-NT 65000 pretreatments maintained a second and third digit rating of 2 or less. Table 5 through Table 8 below contain the ratings for each test panel. For complete results, including evaluation comments, see Appendix B. For test panel photographs, see Appendix C.

The remaining test panels with MAPSIL[®] SILICo (thick) began showing signs of corrosion early in testing (555 hours) and were removed from testing after 1,055 hours.

Ductucatment	Drimor	Alloy	Panel ID	1st Digit	2nd	3rd Digit			
Fietreatment	rimer			Scribe Brightness	Reading	Is olated	Size	Freq	Isolated
			20904	3	0	N/A	1	MD	Yes
Metalast TCP	Hentzen 16708	2024-Т3	20905	3	0	N/A	1	MD	Yes
			20906	4	0	N/A	1	М	Yes
Pretreatment	Primer	Alloy	Panel ID	1st Digit Scribe	2nd	Digit		3rd D	ligit
Tretreatment	1 mile i	Аноу		Brightness	Reading	Is olate d	Size	Freq	Isolated
	Deft 02GN084	2024-T3	21104	3	1	No	2	F	Yes
Metalast TCP			21005	3	0	N/A	2	F	Yes
			21006	3	1	No	0	N/A	N/A
			-				-		
Pretreatment	Primer	Alloy	Danal ID	1st Digit Scribe	2nd	Digit		3rd D	ligit
Tretreatment	1 mile i			Brightness	Reading	Is olate d	Size	Freq	Isolated
	NAVALCOAT	2024-T3	21004	3	0	N/A	1	MD	No
Metalast TCP			21105	3	0	N/A	2	MD	No
			21106	3	0	N/A	2	MD	No

Table 5 – Salt Spray Screening Test Results: Pretreatment and Primer (2,100 hours)

Table 6 – Salt S	Spray Screening	Test Results:	Pretreatment and	Primer (2	2.100 hours)
Table 0 Dale	spray screening	I Cot Itcouito.	I I CH Cathlent and		aground in the state of the sta

Protrootmont	Drimor	Alloy	Donal ID	Panel ID 1st Digit		2nd Digit			3rd Digit		
1 retreatment	1 mer	Аноу		Scribe Brightness	Reading	Isolate d	Size	Freq	Isolated		
			21304	3	2	No	0	N/A	N/A		
SurTec 650V	Hentzen 16708	2024-T3	21305	3	2	No	0	N/A	N/A		
			21306	3	2	No	0	N/A	N/A		
								-			
Ductucation	Drimor	Allow	Donal ID	1st Digit	2nd	Digit	3rd Digit				
Tretreatment	rimer	Alloy	ranei ID	Scribe Brightness	Reading	Is olated	Size	Freq	Isolated		
	Deft 02GN084	2024-T3	21404	3	2	No	0	N/A	N/A		
SurTec 650V			21405	3	1	No	0	N/A	N/A		
			21406	3	1	No	0	N/A	N/A		
Ductucation	Drimor	Allow	Donal ID	1st Digit	2nd	Digit		3rd E	ligit		
rietreatment	rimer	Апоу	raneiiD	Scribe Brightness	Reading	Is olated	Size	Freq	Isolated		
	NAVALCOAT	2024-T3	21504	3	0	N/A	1	F	Yes		
SurTec 650V			21505	3	0	N/A	1	F	Yes		
					21506	3	0	N/A	1	F	No

Due true et au est	Definition	Alloy	Panel ID	1st Digit	2nd Digit		3rd Digit		
Pretreatment	Primer			Scribe Brightness	Reading	Isolate d	Size	Freq	Isolate d
			21704	1	0	N/A	1	F	Yes
Bonderite M-NT 65000	Hentzen 16708	2024-T3	21705	1	0	N/A	1	F	Yes
			21706	1	0	N/A	1	F	Yes
Protrootmont	Primar	Alloy	Panal ID	1st Digit	2nd	Digit		3rd D	Digit
Tretreatment	1 milet	Alloy		Scribe Brightness	Reading	Isolate d	Size	Freq	Isolated
	Deft 02GN084	2024-T3	21804	3	1	Yes	0	N/A	N/A
Bonderite M-NT 65000			21805	3	1	Yes	0	N/A	N/A
			21806	3	1	Yes	0	N/A	N/A
Protroatmont	Primer	Alloy	Panal ID	1st Digit	2nd	Digit		3rd D	Digit
Tretreatment	1 mit 1	Аноу		Scribe Brightness	Reading	Is olate d	Size	Freq	Is olate d
			21904	3	0	N/A	0	N/A	N/A
Bonderite M-NT 65000	NAVALCOAT	2024-T3	21905	3	0	N/A	0	N/A	N/A
			21906	3	0	N/A	0	N/A	N/A

 Table 7 – Salt Spray Screening Test Results: Pretreatment and Primer (2,100 hours)

Table 8 – Salt S	prav Screening	Test Results:	MAPSIL® SILICo	{12 to 16 um	} (555 hours)
	pray Servening	I COU I COUICOU		(1 = 00 10 µm	j (eee nouis)

Duo tuo otmo nt	Drimon	Allow	Danal ID	1st Digit	2nd	Digit	3rd Digit			
Fretreatment	rimer	Alloy	rallerID	Scribe Brightness	Reading	Is olated	Size	Freq	Isolate d	
MADSIL	SILICo		20501	5	1	No	1	D	No	
(12 to 1	2024-T3	20502	5	1	No	1	F	No		
{12 to 16 µm}			20503	5	1	No	1	F	No	
MADEIL ®	SILICa		MAP X 1	5	2	No	2	MD	No	
MAPSIL®	SILICO	2024-T3	MAP X 2	5	1	No	1	F	No	
{12 to 1	o µm}		MAP X 3	5	1	No	1	М	No	

3.5 PATTI Pull Test

This test evaluates the pull-off strength (commonly referred to as adhesion) of a coating system from metal substrates.

3.5.1 Test Procedure

This test was conducted per ASTM D 4541 (Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers, approved February 1, 2009); Annex 3, Self-Alignment Adhesion Tester Type IV (Test Method D).

An Elcometer 110 PATTI portable pneumatic adhesion tester was used during testing in conjunction with an F-4 or F-8 piston and 0.5" pull-stub. Initial results indicated the F-4 piston was not large enough to adequately assess the pull-off tensile strength so the F-8 piston was used.

PATTI pull testing was performed at the NASA KSC Corrosion Technology Laboratory.

3.5.2 Evaluation Procedure

This is a qualitative and quantitative test performed in order to determine adhesion (both innercoat adhesion and intra-coat cohesion) of coating systems. It is a measure of the direct normal applied force required to remove one or more layers from a coating system as a measure of adhesion strength.

Qualitatively, this test evaluates the failure mode. To a limited extent, failure can be determined to be in the glue or in the coating. In the coating, failure can be classified as adhesion (attractive forces between unlike molecules) or cohesion (attractive forces of like molecules) failure. Adhesion failure occurs when the coating separates from the panel. Cohesion failure occurs when the coating separates below show the three failure modes.

Glue Failure – The glue is partially or completely pulled off the panel, leaving the coating intact.





Pull-stub

Panel

Adhesion Failure – The coating is completely pulled off the panel, leaving bare panel. In this example, glue failure is also noted (white residual).



Pull-stub



Panel

Cohesion Failure – The coating failed within itself, leaving coating residual on both the pull-stub and the panel (light yellow). In this example, glue failure is also noted (dark yellow).



Pull-stub

Panel

Quantitatively, this test evaluates the pull-off tensile strength. The Elcometer 110 PATTI portable pneumatic adhesion tester outputs burst pressure values used to determine pull-off tensile strength (pounds per square inch or psi). Based on previous experience, NASA and ESA looked for pull-off tensile strength values of 1,000 psi or above; however, pass/fail criteria is not defined in standards or requirements used by NASA or ESA.

3.5.3 Screening Test Results

Several hexavalent chromium free coating systems performed well on the 2024-T3 aluminum test panels during screening testing. Test results are summarized in Table 9 and Table 10 on the following pages.

In general, most hexavalent chromium free pretreatments with primer exceeded the 1,000 psi pull-off tensile strength that NASA and ESA were looking for. Coating systems that initially failed below 1,000 psi corresponded to a 100 percent (%) glue failure. NASA determined that these failed panels would be retested after an adjustment in test panel preparation procedure. For 100% glue failures, test panels were retested after being lightly sanded and wiped clean prior to placing the glue dolly (per ASTM D 4541). When retested, most panels exceeded the 1,000 psi pull-off tensile strength. However, the MAPSIL[®] SILICo (thick) did not exceed the 1,000 psi pull-off tensile strength during initial testing or retesting. From previous NASA and ESA project experience, silicon-containing coatings like MAPSIL[®] SILICo (thick) typically exhibit limited adhesion properties. For test panel photographs, see Appendix D.

Conversion Coat	Primer	Alloy	Panel ID	DFT (mils)	Failure Mode	Pull-Off Tensile Strength (psi)				
Motalact TCD	Hantzon 16709	2024 72	20907	1.66	60% adhesive/ 10% cohesive / 30% glue	3123				
		2024-13	20908	1.62	40% adhesive / 20% cohesive / 40% glue	3519				
Motalact TCD	Doft 02CN094	2024 72	21007	1.94	90% adhesive / 10% glue*	2690				
IVIELAIAST TCP	Dert 02GN064	2024-15	21008	1.85	85% adhesive / 15% glue*	2789				
		2024 72	21107	2.17	60% adhesive / 40% glue	2768				
Metalast TCP	NAVALCOAT	2024-13	21108	2.59	10% adhesive / 90% glue	1993				
Conversion Coat	Primer	Alloy	Panel ID	DFT (mils)	Failure Mode	Pull-Off Tensile Strength (psi)				
	1000tron 10700	2024 72	21307	2.5	30% cohesive / 70% glue	3003				
Surrec 650V	Hentzen 16708	2024-13	21308	3.31	20% cohesive / 80% glue	3288				
SurTec 650V		2024 72	21407	2.03	80% adhesive / 20% glue*	2038				
Sui lec 050V	Dent 0201004	2024-13	21408	2.01	100% glue*	2826				
		2024 72	21507	2.36	30% adhesive / 70% glue*	3077				
Surrec 650V	NAVALCOAT	2024-13	21508	1.9	100% adhesive	2970				
Conversion Coat	Primer	Alloy	Panel ID	DFT (mils)	Failure Mode	Pull-Off Tensile Strength (psi)				
Popdarita M NT 65000	Hantzon 16709	2024 72	21707	2.42	33% adhesive / 33% cohesive / 33% glue	2579				
Bondente MENT 05000		2024-13	21708	2.52	25% cohesive / 75% glue	3148				
Popdarita M NT 65000	Doft 02CN084	2024 72	21807	1.96	95% adhesive / 5% glue*	1032				
Bondente MENT 05000	Den 0201004	2024-13	21808	1.85	95% adhesive / 5% glue*	2554				
Dondorito MANT (5000		2024 72	21907	2.5	100% glue*	3416				
		2024-13	21908	2.44	100% adhesive	2805				

Table 1 – PATTI Pull Screening Test Results: Pretreatment and Primer

Conversion Coat	Primer	Alloy	Panel ID	DFT (µm)	Piston	Burst Pressure (psi)	Failure Mode	Pull-Off Tensile Strength (psi)
			20507	14	F-8	10.0	100% glue	410
MAPSIL®	SILICo	2024 72	20507	14	F-8	15.0	100% glue*	616
{12 to 16	δµm}	2024-15	20500	14	F-8	11.7	20% adhesive / 80% glue	480
			20508	14	F-8	15.4	45% adhesive / 55% glue*	632
		1	-		1			1
		2024 72		15	F-8	18.1	100% glue	744
MAPSIL®	SILICo		IVIAP A 7	15	F-8	21.3	5% adhesive / 95% glue*	876
{12 to 16 μm}	5μm}	2024-15		12	F-8	17.7	100% glue	727
				12	F-8	23.1	100% glue*	950

Tahla 7 _ PATTI P	ull Scrooning	Tost Rosults	MAPSHR	SIL ICo J	(12 to 16)	1ml
1 able 2 - 1 A I I I I	un screening	I ESI NESUIIS		SILICO	12 10 10	umz

1 Conclusions

Overall the pretreatment with primer systems performed very well during salt spray testing. The performance of the pretreatments with primers was similar regardless of pretreatment and primer combination. The best results were observed on test panels pretreated with Bonderite M-NT 65000 regardless of primer type. MAPSIL® SILICo applied at a thickness of 12 to 16 μ m did not perform well during salt spray testing. Heavy corrosion was observed in the scribe area after 555 hours of testing. After 1,055 hours of testing, the MAP coating appeared to be breaking down with corrosion forming all over the test panels. The MAP panels were removed from testing after 1,055 hours.

The pretreatment with primer systems performed very well during pull-off adhesion testing. All pretreatment with primer combinations had pull-off tensile strengths above 1,000 psi with most of the combinations approaching or exceeding 3,000 psi. The pull-off tensile strength readings for the MAPSIL® SILICo applied at a thickness of 12 to 16 μ m were much lower when compared to the other pretreatment with primer systems. The low readings are attributed to the fact that the adhesive used to secure the pull-off dolly to the test panels would not adhere to the MAP coating. This result was expected since previous testing has shown that silicon-containing coatings like MAPSIL[®] SILICo typically exhibit limited adhesion properties due to adhesive failure.

2 Recommendations

The next step would be to expand the pretreatment with primer systems testing to include; salt spray testing (ASTM B117), wet tape adhesion testing (ASTM D3359 24 hour and 96 hour), tensile adhesion testing (ASTM D4541), and atmospheric exposure testing of coated test panels at KSC. Testing could also include aluminum on aluminum galvanic corrosion coupons. Galvanic corrosion testing is widely used by NAVAIR and should be considered for future NASA-ESA test efforts. Following expanded pretreatment with primer systems testing, the best performing systems should be considered for full system testing; pretreatment with primer and topcoat.

Appendix A – Salt Spray Resistance Evaluation Criteria

1st Digit - Scribe Appearance									
0	Bright and clean								
1	Staining, minor corrosion but no build up								
2	Minor/moderate corrosion product build up								
3	Moderate corrosion product build up								
4	Major corrosion product build up								
5	Severe corrosion product build up								

2nd Digit - Undercutting / Blistering at Scribe									
0	No lifting of coating								
1	Lifting or loss of adhesion up to 1/16" (2 mm)								
2	Lifting or loss of adhesion up to 1/8" (3 mm)								
3	Lifting or loss of adhesion up to 1/4" (7 mm)								
4	Lifting or loss of adhesion up to 1/2" (13 mm)								
5	Lifting or loss of adhesion beyond 1/2" (>13 mm)								

3rd Digit - Blistering away from the Scribe								
	Size	Frequency						
0	None	F	Few					
1	Very Small up to 1/16" (2 mm)	М	Medium					
2	Small up to 1/8" (3 mm)	MD	Medium Dense					
3	Small to Medium up to 1/4" (7 mm)	D	Dense					
4	Medium to Large up to 1/2" (13 mm)							
5	Large (>13 mm)							

Protroatmont	Primer	Aller	Panel	D 1st Digit	1	nd Digi	t		3rd D	Digit	Notes Comments
retreatment	rimer	Anoy	raner	Scribe Brightnes	s Read	ling Iso	late d	Size	Freq	Isolate	d
			20904	3	0	N	I/A	1	MD	Yes	Blistering - top half of test panel; not edge effect; could be coating thickness issue
Metalast TCP	Hentzen 16708	2024-T	3 20905	3	0	N	I/A	1	MD	Yes	Blistering - top half of test panel; not edge effect; could be coating thickness issue
			20906	4	0	N	I/A	1	М	Yes	Blistering - top half of test panel; not edge effect; could be coating thickness issue
Des fair afair and	Determ	411	Denald	1st Digit Scribe	1	nd Digi	t		3rd D	Digit	Natas Commente
Pretreatment	Primer	Alloy	Panel	D Brightness	Read	ling Iso	late d	Size	Freq	Isolate	d Notes - Comments
			21104	3	1	1	No	2	F	Yes	Blistering - bottom right corner, not edge effect; could be coating thickness issue
Metalast TCP	Deft 02GN084	2024-T	3 21005	3	0	N	I/A	2	F	Yes	Blistering - very top of panel; not edge effect; could be coating thickness issue
			21006	3	1	1	No	0	N/A	N/A	
D				1st Digit Scribe	1	nd Digi	t		3rd D	Digit	
Pretreatment	Primer	Alloy	Panel	D Brightness	Read	ling Iso	late d	Size	Freq	Isolate	d Notes - Comments
											Blistering on lower half of the panel, forming near the scribe but does not appear to be undercuttering. Blisters
			21004	3	0	N	I/A	1	MD	No	formed at the top edge of the panel. Coating has been removed from some of the blisters exposing bare aluminum.
											Bistering on lower half of the namel forming near the scribe but does not annear to be undercuttering. A few
			21104	3	0	N	J/A	2	MD	No	bisters are located on the ton half of the panel. Bisters formed at the ton edge of the panel as well as along the right
Metalast TCP	NAVALCOAT	2024-1	3 2110	5	Ŭ			-		1.0	edge and bottom corners. Coating has been removed from some of the blisters exposing bare aluminum
					-						Blistering on lower half of the panel forming near the scribe but does not appear to be undercuttering. A few
			21106	3		N	Ι/ Δ	2	MD	No	bisters are located on the tan half of the panel Bisters formed at the tan edge of the panel as well as the bottom
			21100	5	Ĭ			2	MID	110	corners. Coating has been removed from some of the blisters exposing bare aluminum
											evineto. County into coort removed non-some of the cattery exposing oute analianami
Pre tre atment	Primer	Allov	Panel ID	1st Digit	2nd	Digit		3rd l	Digit		Notes - Comments
				Scribe Brightness H	Reading	Isolated	Size	Freq	Isola	ted	
G			21304	3	2	No	0	N/A	N/.	A	
Surfec 650V	Hentzen 16708	2024-13	21305	3	2	No	0	N/A	N/.	A	
			21306	3	2	NO	0	N/A	N/.	A	
-		1		1.4 D'- '4	2.1	D'- '4	1	2.11	D'- '4		
Pre tre atment	Prime r	Alloy	Panel ID	Ist Digit Sariha Brightmann I	2na .	Digit	Cine.	Sra I	Jugit	4.4	Notes - Comments
			21404		2	No	Size	FIEQ	ISOIA	A	
SurTec 650V	Deft 02GN084	2024 T3	21404	3	1	No	0	IN/A	N/.	A	
5th 100 050 V	Den 02011004	2024-15	21403	3	1	No	0	N/A	N/.	A	
			21400	5	1	NU	0	IN/A	11/.	<u> </u>	
				1st Digit	2nd	Digit	1	3rd	Digit	<u> </u>	
Pre tre atment	Prime r	Alloy	Panel ID	Scribe Brightness	Reading	Isolated	Size	Free	Isola	ted	Notes - Comments
			21504	3	0	N/A	1	F	Ve	s	1 blister found on the top half of the namel could be aggregate
SurTec 650V	NAVALCOAT	2024-T3	21505	3	0	N/A	1	F	Ye	s	1 blister found on the bottom half of the panel, could be aggregate. Blistering on lower right corner.
		-	21506	3	0	N/A	1	F	N	o 3 p	its on lower left side away from the scribe, 1 one very close to the edge. 2 blisters were found away from the scribe. Blisters

<u>Appendix B – Pretreatment with Primer Screening Salt Spray Resistance Results</u>

D rotrootmont	Drimor	Alloy	Donal ID	1st Digit	2nd	Digit		3rd E	Digit	Notes Comments
1 retreatment	1 mie i	Alloy	I allel ID	Scribe Brightness	Reading	Is olate d	Size	Freq	Isolate d	Notes - Comments
			21704	1	0	N/A	1	F	Yes	1 blisted found at the top right edge of the panel
Bonderite M-NT 65000	Hentzen 16708	2024-T3	21705	1	0	N/A	1	F	Yes	1 blister at the top center edge of the panel
			21706	1	0	N/A	1	F	Yes	2 blisters at the top right edge of the panel
Drotrootmont	Drimor	Alloy	Donal ID	1st Digit	2nd	Digit		3rd E	Digit	Notes Comments
rretreatment	1 mie i	Alloy	I allel ID	Scribe Brightness	Reading	Is olate d	Size	Freq	Isolate d	Notes - Comments
			21804	3	1	Yes	0	N/A	N/A	
Bonderite M-NT 65000	Deft 02GN084	2024-T3	21805	3	1	Yes	0	N/A	N/A	
			21806	3	1	Yes	0	N/A	N/A	
Due tree et me nt	Duimon	Allow	Danal ID	1st Digit	2nd	Digit		3rd Digit		Notas Commonts
1 retreatment	1 mie i	Anoy	1 aller ID	Scribe Brightness	Reading	Is olated	Size	Freq	Isolate d	Notes - Comments
			21904	3	0	N/A	0	N/A	N/A	Blistering / coating removal on bottom edge / corners
Bonderite M-NT 65000	NAVALCOAT	2024-T3	21905	3	0	N/A	0	N/A	N/A	Blistering / coating removal on bottom edge / corners
			21906	3	0	N/A	0	N/A	N/A	Blistering on bottom edge / corners

Conversion Coat Primer	Allow	Demolu	1st Digit	2nd	2nd Digit		3rd D	igit	Notes Commonly		
conversion coat	Finner	Alloy	Fallerid	Scribe	Reading	Isolated	Size	Freq	Isolated	Notes - Comments	
MAPSIL® SILICo			20501	5	1	No	1	D	No	PULLED FROM TESTING AFTER 1,055 HOURS	
		2024-T3	20502	5	1	No	1	Freq	No	PULLED FROM TESTING AFTER 1,055 HOURS	
{141010	{14 to 16 µm}		20503	5	1	No	1	Freq	No	PULLED FROM TESTING AFTER 1,055 HOURS	
MADELL®	MAPSIL® SILICo			MAP X 1	5	2	No	1	MD	No	PULLED FROM TESTING AFTER 1,055 HOURS
IVIAPSIL [®]		2024-T3	MAP X 2	5	1	No	1	F	No	PULLED FROM TESTING AFTER 1,055 HOURS	
{14 to 16 µm}			MAP X 3	5	1	No	1	М	No	PULLED FROM TESTING AFTER 1,055 HOURS	

Alloy: 2024-T3								
(2,100 hours)								
20905	20906							
	0000 0000 0000 0000 0000 0000 0000 0000 0000							
	(2,100 hours) 20905							

Appendix A – Pretreatment with Primer Screening Salt Spray Resistance Test Panel Photos

NASA TEERM NASA and ESA Pretreatments and Primers Screening Final Test Report



NASA TEERM NASA and ESA Pretreatments and Primers Screening Final Test Report

Alloy: 2024-T3		
Metalast TCP / NAVALCOAT	(2,100 hours)	
21004	21105	21106
000		o O O O O O O O O O O O O O O O O O O O
	00	00000
	n s	2000
R Z	Barry	



NASA TEERM NASA and ESA Pretreatments and Primers Screening Final Test Report

Alloy: 2024-T3		
SurTec 650 V / Deft 02GN084	(2,100 hours)	
21404	21405	21406
	1	
	She was	
	19. Contract (19. Contract)	
· //		15
51	\sim	<
1 30	\sim /	λ /
	λ /	40
22	\bigvee	
/*X3	A	50
/ " าเ	$/ \chi$	6. c 2.5
/ 5	Pas es	
2+3		λ
P 12	1 - N	,0 0 ⁵

Alloy: 2024-T3		
Bonderite M-NT 65000 / Hentz	en 16708 (2,100 hours)	
21704	21705	21706
		0
	N Z	N /
	$\langle \rangle$	
	$\langle \rangle$	
\sim	\sim	\sim
\wedge	\wedge	\wedge
$\sim 10^{-10}$		



NASA TEERM NASA and ESA Pretreatments and Primers Screening Final Test Report

Alloy: 2024-T3		
Bonderite M-NT 65000 / NAV	ALCOAT (2,100 hours)	
21904	21905	21906

Alloy: 2024-T3		
MAPSIL [®] SILICo (thick) (1,05	5 hours)	
20501	20502	20503
MAPSIL [®] SILICo (thick) (1,05 20501	5 hours) 20502	

Alloy: 2024-T3		
MAPSIL [®] SILICo (thick) (1,05	5 hours)	
MAP X I	MAP X 2	MAP X 3
	n /	
		R

Metalast TCP / Hen	tzen 16708 (20907)	Metalast TCP / Hen	tzen 16708 (20908)
Pull-Stub	Panel	Pull-Stub	Panel
Metalast TCP / Def	t 02GN084 (21007)	Metalast TCP / Def	t 02GN084 (21008)
Pull-Stub	Panel	Pull-Stub	Panel
Metalast TCP / NAV	VALCOAT (21107)	Metalast TCP / NA	VALCOAT (21108)
Pull-Stub	Panel	Pull-Stub	Panel
	1 11111	i un biub	1 41101

Appendix B – Pretreatment with Primer Screening PATTI Pull Test

SurTec 650V / Hent	tzen 16708 (21307)	SurTec 650V / Hen	tzen 16708 (21308)
Pull-Stub	Panel	Pull-Stub	Panel
SurTec 650V / Deft	02GN084 (21407)	SurTec 650V / Def	t 02GN084 (21408)
Pull-Stub	Panel	Pull-Stub	Panel
SurTec 650V / NAV	ALCOAT (21507)	SurTec 650V / NAV	VALCOAT (21508)
Pull-Stub	Panel	Pull-Stub	Panel

Bonderite M-NT 650 (217	000 / Hentzen 16708 707)	Bonderite M-NT 650 (212	000 / Hentzen 16708 708)
Pull-Stub	Panel	Pull-Stub	Panel
Bonderite M-NT 650 (218	000 / Deft 02GN084 807)	Bonderite M-NT 65 (218	000 / Deft 02GN084 808)
Pull-Stub	Panel	Pull-Stub	Panel
Bonderite M-NT 650 (219	000 / NAVALCOAT 007)	Bonderite M-NT 65000 / NAVALCOAT (21908)	
Pull-Stub	Panel	Pull-Stub	Panel

MAPSIL® S	ILICo (20507)	MAPSIL® SI	LICo (20508)
Pull-Stub	Panel	Pull-Stub	Panel
MAPSIL® SILICO (MAP X 7)		MAPSIL® SILICo (MAP X 8)	
MAPSIL® SIL	LICo (MAP X 7)	MAPSIL® SIL	Co (MAP X 8)
MAPSIL® SIL	JCo (MAP X 7)	MAPSIL® SIL	Co (MAP X 8)