Testing of Environmentally Preferable Aluminum Pretreatments and Coating Systems for Use on Space Shuttle Solid Rocket Boosters (SRB)

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Abstract

The SRB has historically used a chromate conversion coating prior to protective finish application. After conversion coating, an organic paint system consisting of a chromated epoxy primer and polyurethane topcoat is applied. An overall systems approach was selected to reduce waste generation from the coatings application and removal processes. While the most obvious waste reduction opportunity involved elimination of the chromate conversion coating, several other coating system configurations were explored in an attempt to reduce the total waste. This paper will briefly discuss the use of a systems view to reduce waste generation from the coating process and present the results of the qualification testing of nonchromated aluminum pretreatments and alternate coating systems configurations.

Introduction

United Space Alliance – Solid Rocket Boosters (USA-SRB) refurbishes the non-motor components of the SRB. The structural components are primarily constructed of aluminum alloy (AA) 2219 or Alclad 7075. The aluminum is chromate conversion coated with MIL-C-81706 (Alodine 1200S/1201), primed with Deft primer 44-GN-7 and then topcoated with Deft topcoat 03-W-127A.

MIL-C-81706 specifies chromate-based solutions used in the formation of a conversion coating to provide a protective layer on an aluminum surface. This conversion coating is used by industry for its corrosion protection properties and to promote adhesion of paints to the aluminum. However, the high copper content in AA 2219 negates the corrosion protection properties of the chromate conversion coating. Since much of the aluminum components of the SRBs are 2219, the pretreatment is only used to promote and stabilize paint adhesion. The waste stream for this process is one of the largest at Kennedy Space Center (KSC). Anywhere from 20 to 50% of chromate conversion coatings are chromic acid. Hexavalent chromium, which is present in these solutions, has been determined to have serious acute and chronic exposure hazards. The Occupational Safety and Health Administration (OSHA) continues to move toward stricter regulations for the use of chromium in the work place. The Environmental Protection Agency is also continuing to tighten regulations related to airborne and liquid releases of these materials.

The Deft paint system is used to coat the aluminum and Corrosion Resistant Steel (CRES) alloys. Deft was chosen as the paint system on these metal surfaces for its lower volatile organic compound (VOC) content and compatibility with the thermal protection system (TPS) materials. The Deft paint system has several processing limitations which include a short pot life for both primer and topcoat, primer curing issues and topcoat catalyst moisture sensitivity. These processing problems result in process efficiency limitations that currently have no options to avoid or mitigate them. The primer also uses a hexavalent chromium corrosion inhibitor, which is under the same regulatory pressure as the pretreatment. Finally, these coatings which cover and protect 95% of the SRB hardware are sole sourced with no alternates. A fire or other disaster at the manufacturer's plant could place space shuttle schedules in jeopardy. For these reasons the Alternate SRB Aluminum Coating Project was undertaken to identify and qualify alternatives for the current coating and pretreatment system.

Experimental Design

The Alternate SRB Aluminum Coating Project consisted of three phases: down selection, qualification, and implementation. After review of vendor information, such as data sheets, and MSDSs six pretreatments and six coating systems were tested in the down selection portion of the project. Three pretreatments and three coating systems were chosen from the down selection testing for qualification. Consult "Alternative SRB Aluminum Coating System Down Select Test Report" (8) for a discussion of how the candidates were chosen. The candidate materials chosen for qualification testing are listed in Table 1*. In addition to testing new materials for a straight drop in replacements, this qualification program has also considered a new flight configuration, which consists of primer as the only coating. The goal was to certify the pretreatment/primer configuration without topcoat in areas that are stripped every flight. All coating candidates (primer and topcoats) were evaluated on each candidate pretreatment and the baseline pretreatment (Alodine 1201). Additionally, the baseline coatings (Deft 44GN7 primer and 03W0127 topcoat) were evaluated on all pretreatments. Testing included the currently used SRB coating configuration as a baseline to assist in the performance assessment of the candidate materials. The full factorial experiment design panel matrix used for each test required 104 separate conditions to be produced which, with duplication, would mean 208 test surfaces. The alloys tested included aluminum alloys 2219, 6061, and 7075; also 304 stainless steel. Where appropriate, the number of material variables was reduced to simplify testing and data analysis.

Table 1. Candidates Selected for Qualification Testing

Pretreatments	Primers	Topcoats
Alodine 1201	Deft 44GN7	Deft 03W127A
Alodine 5700	PRC-DeSoto EEAE152A/B	PRC-DeSoto EUAW098A/ EUAC082B
Chemidize 727A	Hentzen Corp. 05510WEP-X/ 05511CEH-X	Hentzen Corp. 4636WUX-3/ 4600CHA-SG
Okemcoat 4500	Lord Corp. 9929A/B	Lord Corp. A276

When a coating was found to be unsatisfactory for qualification at any point, testing of that pretreatment and/or coating system was halted. One pretreatment and one coating were dropped during the qualification testing, Okemcoat 4500 pretreatment and the PRC-DeSoto coating system.

Testing was conducted in accordance with "Alternative SRB Aluminum Coating System Qualification Test Plan," CPC-054-98MP (8) which gathered corrosion protection, bond strength, compatibility with other SRB materials, batch-to-batch consistency, and thermal environments data. A complete copy of the test data along with pertinent digital photographs has been placed on computer disk and is available upon request.

Methods

Testing conducted to qualify the candidate pretreatments and coating systems consisted of analysis in these areas: Corrosion, bond strength, compatibility with other SRB materials, batch-to-batch consistency, and thermal environments.

The corrosion protection offered by a coating system relies upon the adhesion of the coating to the metal surface, corrosion inhibition properties of the pigments within the coating and the resistance of the coating to moisture absorption/permeation. It is very difficult to formulate a single product to encompass all of these considerations. To maximize the corrosion protection, several classes of products are used. These include adhesion promoters (pretreatments), primers (incorporating corrosion inhibiting pigments) and topcoats (to minimize moisture permeation). The level of corrosion protection required for a particular component/system should be based upon the use environment and service (refurbishment cycle) requirements.

Corrosion testing of the candidate pretreatments and coating systems was conducted to determine the effectiveness of the corrosion preventative properties of the candidate materials. The evaluations included beach exposure, seawater immersion, and Electrochemical Impedance Spectroscopy (EIS). These test regimes were selected to provide an aggressive real life performance evaluation of the candidates combined with a practical laboratory evaluation technique. While the SRB uses several aluminum alloys, AA 2219 was selected for all corrosion testing due to its inherent poor corrosion resistance.

Seawater immersion testing was conducted to reflect the actual use environment of the SRB hardware. After launch, the SRB lands in the ocean and is towed back to port. The SRB normally spends 24 to 48 hours in seawater but has been immersed more than twice that long when problems were encountered in recovery. The coupons used in the seawater immersion test were then used for the beach exposure test. The coupons were placed at the Kennedy Space Center (KSC) Atmospheric Corrosion Test site within 48 hours of removal from the seawater. These coupons went through both tests scribed. The exposure times were six and twelve months.

The final corrosion test conducted was Electrical Impedance Spectroscopy (EIS). With EIS, a small amplitude AC voltage is applied to the test coupon and the coating response is measured. This process is repeated over a range of frequencies to generate a broad response spectrum. EIS technique is considered to be nondestructive to the coating, this allows the change in coating performance to be measured over time. Each electrolytic cell over the test coupon contained 200 ml of filtered seawater. Measurements were taken at 4, 168, and 672 hours.

The most critical physical property for SRB coatings is the ability to act as an adhesive base for the thermal protection system materials. Bond strength testing was conducted to ensure that the coating systems adhere to the pertinent SRB substrates and to themselves. Next the testing was expanded to look at the effects of different environments, thermal cycling, water immersion, and damage resistance or flexibility. Adhesion testing was conducted per ASTM D 4541-95 “Pull-Off Strength of Coatings Using Portable Adhesion Testers” (6). Adhesion of the coatings in high temperature environments was tested per ASTM D 1623-78 “Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics” (7). A failure during a water immersion test could be an indication of a deficiency in the coating itself or inadequate surface preparation provided by the candidate pretreatment. Coatings used on the SRB need to be able to adhere under these conditions. Water resistance is not a corrosion test and there is no correlation with immersion into the Atlantic Ocean. Testing was conducted per ASTM D 870-97 “Testing Water Resistance of Coatings Using Water Immersion” (3). Flexibility testing was conducted to determine the resistances of the candidate coatings to cracking on metal substrates as those substrates are deformed. This is a measure of damage resistance. The testing was conducted per ASTM D 522-93a “Mandrel Bend Test of Attached Organic Coatings” (5).

Any coating system used on SRB surfaces must be compatible with the chemicals and materials that will come in contact with the coating system. This includes thermal protection system, sealants, cleaners and possible incidental contact with various chemical compounds. Materials applied to the coating system must be able to adhere to the coating system at flight temperatures as well as at room temperature. The testing for material adherence and chemical compatibility was conducted per ASTM D 1623 “Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics” (7), ASTM D 4541-95 “Pull-Off Strength of Coatings Using Portable Adhesion Testers” (6), and ASTM F 502-93 “effect of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces” (4).

The candidate coatings and pretreatments must be able to withstand the thermal environments of space flight. The candidate coatings were subjected, bare and with the thermal protection system in place, to convective testing performed in the National Aeronautic Space Administration / Marshall Space Flight Center (NASA/MSFC) Improved Hot Gas Facility (IHGF). The IHGF is a nominal MACH 4.1 aerothermal tunnel that burns a lean mixture of hydrogen (GH_2) and air to produce temperatures up to 2200ºF at total pressures up to 200 psia (pounds per square inch atmospheric). The coatings and pretreatments used on SRB flight hardware must not be able to support combustion. Testing for flammability of the pretreatments and coatings was conducted per. 4.1 of NHB 8060.1B "Flammability, Odor, and Off-gassing Requirements and Test Procedures for Materials in Environments that Support Combustion" (2).

To ensure that the vendors of the candidate coatings and pretreatments could supply a consistent product from lot to lot, repeatability testing was conducted. This testing consisted of EIS testing, adhesion testing, and compatibility testing to Spirit 126 (cleaner used on coated surfaces).

Results

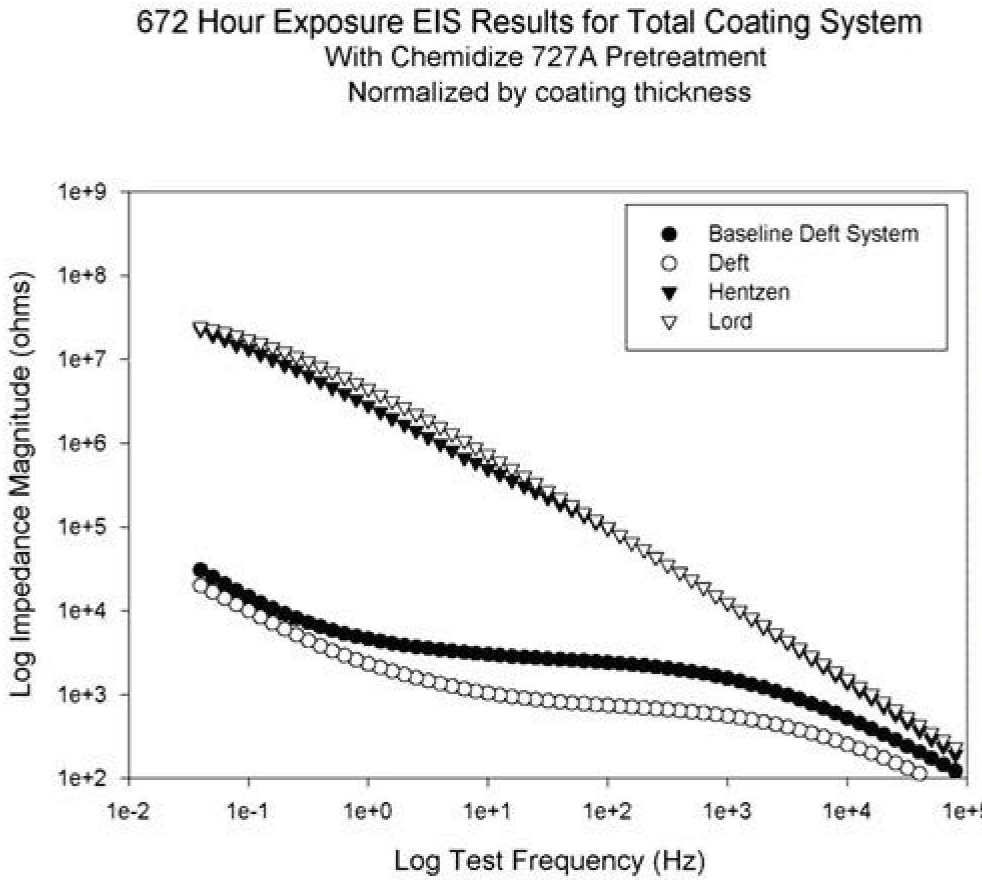
The amount of data produced from the tests discussed above was substantial. Aluminum alloy 2219 was selected for all corrosion testing. The exposure tests conducted were more severe than tests used in past coating qualifications. The sea water immersion results showed that the Chemidize 727/Hentzen primer coating configuration was significantly worse at the end of the exposure period than the baseline coating/pretreatment system. All other coatings were considered nominal. The coupons used for the sea water immersion were then used for the beach exposure testing. After exposure, no coupons were found to have either filiform corrosion or any biological growth. The Alodine 5700 pretreatment with Lord primer and the Alodine 5700 with the Lord primer and topcoat had the least amount of corrosion other than the baseline configuration. The PRC-DeSoto primer was the poorest performing primer regardless of the pretreatment configuration. At the time of the corrosion testing the PRC-DeSoto announced that they would no longer produce the topcoat that was in the test program. Testing continued with the primer for further evaluation of the product. The results of the corrosion data showed that the PRC-DeSoto primer was not an acceptable coating for SRB use

The EIS testing showed no significant difference between the pretreatments of the primed and topcoated coupons. However, the performance of the primer-only coupons did show some differences for the Deft and Hentzen coatings. Both of these primers over Chemidize 727 pretreatment showed a decrease in performance, see Figure 1*.

The bond strength testing includes data of the pretreatments and coatings on 2219 T87 aluminum, 7075 Alclad aluminum, and 304 stainless steel. The coatings were tested on the 304 stainless steel without pretreatment. The coatings adhered to the stainless steel better than to the aluminum alloys. There was no significant difference in the coating material adhesion attributable to the pretreatment material. After the alloy and pretreatment factor is removed from the results, the Deft topcoat performance appeared to be better than the candidates by about 12 %. Still the adhesion values of the candidates are close to double the minimum requirement of 700 ponds per square inch (psi). The averages range from 1266 psi for Lord primer over Chemidize 727 on 7075 Alclad to 2469 psi for Deft topcoat on 304 stainless steel.

For repair purposes, testing was conducted to determine the strength of the candidates over each other. This included all variations of primers and topcoats. The values of the adhesion results were within expected population of the nominal values obtained from the previous adhesion tests with a majority of the values above 1400 psi. The range was 1259 psi to 2647 psi. Some high performance coatings develop very inert surface layers due to side reactions or level of cure and cross-linking. For this reason adhesion testing was conducted on double-coated coupons. The first coat was fully cured prior to application of the second coat. The results of the tests were nominal as compared with the previous adhesion test results with the average of the values being 1500 psi.

Figure 1. Magnitude for Chemidize 727 With Primer Candidates



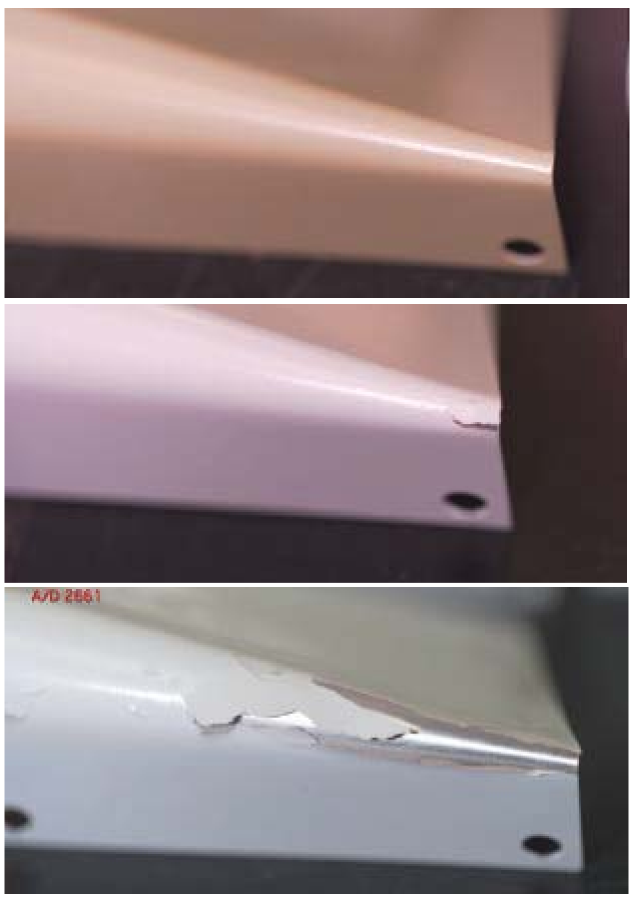
To ensure the coatings could withstand multiple flights before having to be removed, Cyclic high temperature testing was conducted. The coupons were cycled to 350ºF from room temperature ten times with adhesion testing being conducted after five and ten cycles. The Hentzen and Lord primers did not show any significant change in strength throughout the cycling. This shows a stability that was expected for these materials. The baseline Deft primer showed a decrease in strength throughout the cycling. One possible explanation is that the Deft primer remains more reactive after it has dried. This would cause it to oxidize readily when exposed to a more reactive (hotter) environment. The pretreatments had no influence on the results of this test.

Flatwise-tensile testing was conducted to determine how well the pretreatments and coating systems adhere at 350ºF, the worst case SRB temperature environment. One pattern emerged from the testing. The Lord coatings had a marked tendency to fail at the or near the pretreatment at the elevated temperature. This was even more pronounced when Lord coatings were over Chemidize 727 pretreatment. The lowest value of the Lord coating with the primer/pretreatment failure was 181 psi. The average of all the tests was 259 psi. This shows a drop in strength, however there is no minimum strength requirement for a paint to adhere at 350ºF. Our criteria is that the coatings exceed TPS strength in the same environment. Therefore, all the paints are considered acceptable for program use.

Water immersion testing was conducted on scribed panels of the candidate coating systems per ASTM D870-97 "Testing Water Resistance of Coatings Using Water Immersion" (3). A failure during this test could be an indication of a deficiency in the coating itself or inadequate surface preparation. The principal criteria were to have no lifting of the coating and no active corrosion under the coating. Every panel, regardless of alloy, which was pretreated with Okemcoat 4500 and then coated with PRC-DeSoto failed due to blistering and flaking of the coating, both at the scribe and in the general acreage. With other pretreatments the PRC-DeSoto coatings would develop small blisters on about 46% of the panels which "healed" when the panels dried. Panels pretreated with Okemcoat 4500 developed blisters on 65% of the panel area. If the Chemidize 727 pretreatment was not completely rinsed from the panels, the edges could flake. Some level of blistering was observed on all panels coated with Hentzen over Chemidize 727. The Alodine 1201 and Alodine 5700 had minor blistering observed at the edges. This underscored the importance of thorough rinsing and draining during the application process

The flexibility test was used to determine the resistances of the candidate coatings to cracking on metal substrates as those substrates are deformed. The results of this test showed one pretreatment, Okemcoat 4500, had significantly poorer properties. The Okemcoat 4500 did not cause the paint to crack, the pretreatment failed in shear loading along the bend leading to delamination of the coating with cracking and peeling. There are no SRB program acceptance or rejection criteria for elongation, but the failure in shear is felt to be not acceptable due to environments that the coating would see. For this reason and the results of the water immersion, Okemcoat 4500 was dropped from the qualification program. The other pretreatments did not influence the results. The coatings were all comparable with the exception of the Hentzen topcoat. It was significantly more rigid than the other coatings but still acceptable. Figure 2* shows examples of coupons that passed versus a failure of the Mandrel Bend Test.

Figure 2. Mandrel Bend Coating Failure



Acceptable >32% elongation

Acceptable ~20% elongation

Failure – Coating Delamination

Material compatibility testing was conducted to ensure that the materials applied over to the SRB coated surface would adhere to the candidate coating systems. The following materials were tested with the candidates:

MCC-1	PR-1422	CORK	P/S 870	BTA	RT455
Hypalon	PR-1770	Insta-Foam	RTV 133	K5NA	EA-934
VCI 368	LPS Hardcoat	DeSoto repair Primer		DeSoto Repair topcoat	

Some of the materials have minimum strength requirements. All the tensile strength requirements were met. The values were nominal as compared to the baseline and past tensile studies. The Hentzen topcoat did show a marked decrease in bond strength at room temperature with RTV 133. The drop in bond strength was about 53%. There is no minimum strength requirement for RTV 133. All failures were between the RTV 133 and the Hentzen topcoat. A change in surface preparation prior to bonding silicone sealants may be needed. The drop in bond strength for RTV 133 over Hentzen did not follow through to the high temperature testing. The values for RTV 133 over Hentzen at 350ºF were nominal as compared to the other candidates and the baseline. The DeSoto repair topcoat showed a great affinity to the Hentzen and Lord primers. The average strength of the DeSoto repair compatibility testing was 1469 psi, whereas the average of the DeSoto topcoat over Hentzen and Lord primers was 2655 psi.

In addition to room temperature testing, the thermal protection system (TPS) and sealants were also tested at elevated temperature to ensure that they could adhere to the candidate coatings during flight. There is no minimum tensile strength requirement for elevated temperature adhesion, only that the material demonstrate adherence. MCC-1, K5NA, Cork, RT455, BTA, and RTV 133 were tested at 350ºF. PR-1422 was tested at 275ºF, PR-1770 was tested at 280ºF, and P/S 870 was tested at 235ºF. The polysulfide sealants were tested at a lower temperature due to their respective softening temperatures. As was seen in earlier flatwise tensile testing at elevated temperatures the Lord coatings over Chemidize 727 failed between the primer and the pretreatment. The PR-1422 with Lord primer also had failures between the primer and Okemcoat 4500, Alodine 1201, and Alodine 5700. There was significant variation within the material of the PR-1770 sealant, influencing the results of that particular test data. The rest of the test results were nominal as compared to the baseline configuration.

Chemical exposure tests were conducted to determine if contact to cleaning fluids and incidental contact to pertinent chemicals would affect the candidate coatings. A drop of 2 pencil grade is considered a failure in this test. The candidate coatings were exposed to following compounds at 95°F:

Spirit 126	Prime	PF Degreaser
14% Citric Acid	13% Sodium Hydroxide	

The exposure temperature for the following compounds was room temperature due to their flammability:

Isopropyl Alcohol (IPA)	Acetone	DS-104
Hydraulic Fluid	Toluene	Methyl Ethyl Ketone (MEK)

Spirit 126, Prime, PF Degreaser, hydraulic fluid, 13% sodium hydroxide, and 14% citric acid did not affect any of the candidate coatings. Okemcoat 4500 had been withdrawn from the test program prior to compatibility testing. IPA did affect PRC DeSoto topcoat. The other solvents, MEK, DS-104, toluene, and acetone are aggressive solvents that would affect or strip a coating. These solvents are used to remove tenacious contaminates. A drop in 2 pencil grade is not considered a failure for these solvents. Acetone did have a definite affect on PRC DeSoto and Deft topcoats. DS-104 affected Deft, PRC DeSoto, and Hentzen topcoats. DS-104 also had some effect on Hentzen primer. A second test was conducted with these solvents to more clearly determine their effect on the candidate coatings in a handwipe situation. These solvents are only used on SRB coatings in handwipe processes. A hand wipe rub test was conducted per MIL-C-85285B "Coating: Polyurethane, High Solids" (1). All candidate cleaners passed the rub test for all solvents. Okemcoat 4500 and PRC-DeSoto had been withdrawn from the test program prior to the rub test. The candidate coatings were also exposed to hydrazine. All of the candidate coating systems passed the hydrazine compatibility test.

To ensure that the candidate coatings systems are able to withstand the thermal environments of space flight, thermal testing at MSFC IHGF was conducted. All the candidates passed thermal testing with TPS applied to the surface and without TPS applied to the surface. Panels without TPS were tested at 5 BTU/ft^2 and TPS panels were tested at 7 BTU/ft^2. Okemcoat 4500 and PRC-DeSoto were dropped during the thermal testing and were not included in the results.

The coatings and pretreatments were tested to ensure that they would not be able to support combustion. All pretreatments and coatings passed the flammability tests. The burn lengths were less than 6 inches with a burn time of less than 10 minutes. There was no sparking, sputtering, or dripping. The panels that had Okemcoat 4500 pretreatment had the longest burn length with an average of 0.8 inches. The average of all the other panels was 0.25 inches.

To ensure that the vendors of the candidate coatings and pretreatments could supply a consistent product from lot to lot, repeatability testing was conducted. This testing consisted of EIS testing, adhesion testing, and compatibility testing to Spirit 126 (cleaner used on coated surfaces). The EIS testing showed very little performance difference for the candidate configurations. One notable exception is the performance of Deft primer. The spectrum for lot 2 of Deft primer was more typical of a spectrum for a coating performing as a barrier. Lots 1 and 3 were more indicative of a damaged coating. Visual examination of the Deft primer coupons showed blister on the Deft primer over Chemidize 727 lot 1 coupon and on all of the lot 3 Deft primer coupons. Despite the variation in the Deft primer performance, the second and third lots still indicate performance equal to or better than the first lot tests.

The adhesion repeatability testing showed no significant difference from the first to second lot for all the candidate configurations. Lot number three however, is significantly lower in adhesion than the previous two lots for all materials. Looking at the three charts it is possible to deduce that the drop is independent of any of the variables that were being tested. The following variables were then reviewed for a possible answer to the drop in strength: adhesive, temperature, humidity, test procedures, and personnel. Adhesive, temperature, humidity, and test procedure all turn out to be negative. However, the variation in bonding the test stubs and preparing the bond site is suspected to have been great enough from one analyst to another to cause the drop in bond strength. The results of the repeatability testing of chemical compatibility of Spirit 126 to three lots of the candidate coatings showed no variation from lot to lot.

Conclusion

The coating configurations acceptable for use on the SRB aluminum components are list in the Table 2*. Note that the pretreatment and primer only configuration is acceptable for surfaces which have the coating removed after each flight cycle. The primers and topcoats listed are also qualified for stainless steel surfaces. For further information about this test program, please refer to CPC-025-00MP "Alternate SRB Aluminum Coating System Qualification Test Report" (10).

Table 2. Coating Configurations Recommended for Use on SRB Aluminum Components

Pretreatment	Primer	Topcoat
Alodine 1201	Deft 44GN7	Deft 03W127
Alodine 1201	Hentzen 05510WEP-X/05511CEH-X	Hentzen 4636WUX-3/4600CHA-SG
Alodine 1201	Lord 9929A/B	Lord A276
Alodine 5700	Deft 44GN7	Deft 03W127
Alodine 5700	Hentzen 05510WEP-X/05511CEH-X	Hentzen 4636WUX-3/4600CHA-SG
Alodine 5700	Lord 9929A/B	Lord A276
Chemidize 727	Deft 44GN7	Deft 03W127
Chemidize 727	Lord 9929A/B	Lord A276

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