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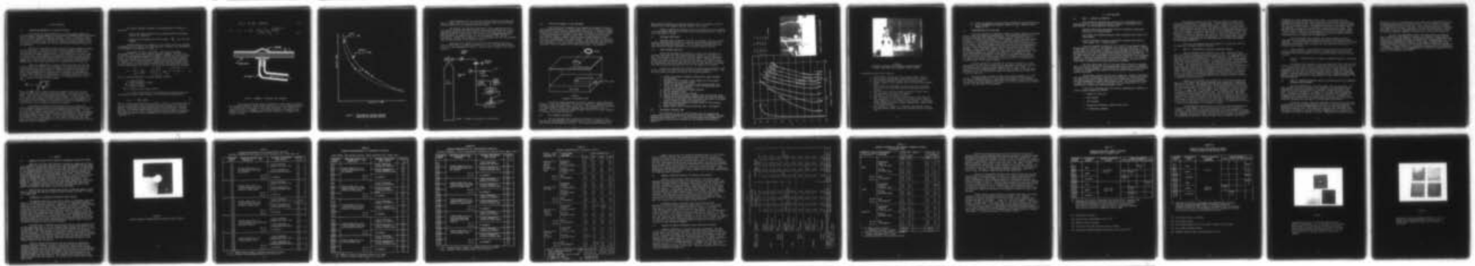

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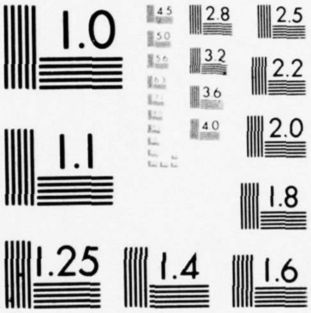

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This report describes the results obtained in a study program to optimize surface preparation and application of organic coatings on non-metallic composites and to develop adhesion data on additional aluminum alloys following an optimized anodizing process. This study also describes development of adhesion data on additional aluminum alloys following an optimized chemical conversion coating processes. The Blister Test Method was utilized for

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generation of all adhesion data obtained in this study. The concluding segment of this study describes optimization of chemical conversion coating processing parameters to obtain reliable adhesion characteristics within practical processing tolerances and investigation of more rapid test methods of measuring resistance of aluminum alloys to corrosion.

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OPTIMIZATION OF PROCESSING VARIABLES WHICH AFFECT ADHESION OF ORGANIC COATINGS

FINAL TECHNICAL REPORT

Submitted to
Naval Air Systems Command
Material Acquisition Group
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FOREWORD

This program was designed to study the effects of surface preparation and application techniques of applying organic coatings on non-metallic composites and to develop adhesion data via the Blister Test Method. The program was also intended to optimize chemical conversion coating processes and to develop a rapid means of corrosion testing of chemically treated aluminum alloys. This program was sponsored by the Naval Air Systems Command under Contract N00019-76-C-0358 and was a follow-on effort to Contract N00019-75-C-0087. Technical administration of this program was the responsibility of Mr. David P. Hornick of the Engineering Division of the Materials Acquisition Group of the Naval Systems Command. ?

The program was initiated in April of 1976 and completed in a one year time period.

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1.0 INTRODUCTION AND SUMMARY

This program was completed in order to investigate adhesion characteristics of organic coatings on non-metallic composites and on aluminum alloys utilizing the Blister Test Method to evaluate surface preparation techniques and processing. The program provided additional adhesion data obtained from anodized and chemically treated aluminum alloys. Processing parameters were investigated to obtain improved corrosion resistance of chemically treated aluminum alloys and an improved method of rapidly measuring the corrosion resistance of chemically treated aluminum alloy substrates was sought.

This investigation was designed to develop adhesion data of the standard Navy paint system (MIL-C-81773 polyurethane topcoat over MIL-P-23377 epoxy-polyamide primer) over various non-metallic composite materials utilizing the Blister Test Method to quantify adhesion surface energy density between this surface finish and the non-metallic composite substrates. Composite laminates tested were as follows:

- Rigidite 5208, Thornel 300
- AVCO 5505/4
- Hercules 3501/AS1-6

Specimens of each of the cured laminates were prepared for coating by five methods and then contaminated with MIL-H-5606 hydraulic fluid. Specimens were cleaned and then coated with the standard Navy paint system. The adhesion of each specimen was determined with the Blister Test Method after exposure to condensing humidity at 165±5°F, boiling deionized water and as prepared. Good results of adhesion were obtained with values of greater than the accepted minimum of 1.0 in.-lb./in.^{2} for all specimens tested.

The following four aluminum alloys were evaluated in anodizing optimization studies:

- 2219
- 6061
- 7075
- 7050

The 6061 and 7075 alloys were obtained from Vought Shop Stores in the bare condition. The 7050 alloy was supplied by the NAVAIR in the clad condition, and this alloy was processed to remove the clad. The 2219 was obtained from commercial supplier.

In order to investigate more rapid methods to measure corrosion resistance of anodized aluminum alloys, it was necessary to produce coatings that will not pass the presently required salt spray test. A literature survey indicated two possible methods to produce the desired results, improper heat treatment of the aluminum substrate or interrupted current during anodizing. Both methods were reported to reduce the corrosion resistance of the anodized aluminum alloys; however, corrosion tests of these improperly processed specimens were found to equal specification values. Adhesion of the standard

Navy paint system was also determined for the improperly treated specimens and found to be adequate, although variable. The "as prepared" control specimens failed at lower values than did some of the specimens exposed to hostile environments. This was attributed to a slow curing primer, and the control specimens failed to have the benefit of the elevated temperature to which the exposed specimens were subjected, thus completing the cure of the exposed specimens and increasing the adhesion values in this case.

Chemical conversion coatings on aluminum alloy substrates were evaluated to meet requirements of MIL-C-5541, "Chemical Films and Chemical Film Materials for Aluminum and Aluminum Alloys." Two commonly used aluminum alloys and two widely used conversion coatings were used in these evaluations; these were 7075 and 2024 aluminum and a Class 1A and Class 3 conversion coatings. These application techniques were used with each coating-alloy combination as follows:

- Tank application following cleaning and deoxidation
- Spray application following cleaning, but not deoxidized
- Abrasion scrubbing application with a Scotch Brite abrasive pad wetted with the chemical conversion solution following cleaning, but not deoxidized

Chemically treated specimens were prepared and coated with the standard Navy paint system. Coated specimens were cured at room temperature and the adhesion of these specimens was determined by the Blister Test Method after exposure to condensing humidity and boiling deionized water environments. Again, variation was observed between the adhesion values for the exposed specimens and controls. This variation was also considered to be a state of cure variation in primer due to post cure resulting during exposure of the specimens to the elevated temperatures during test.

A rapid means of determining corrosion resistance of aluminum alloys protected with chemical conversion films was evaluated which involved determination of the weight per square foot of the conversion coating depositied, coating loss or change during accelerated corrosion testing and development of a spot test to determine if the corrosion coating had been penetrated in the accelerated tests. Ease of stripping of the conversion coatings was determined. The Class 1A and Class 3 conversion coatings, 7075 and 2024 aluminum alloys, were utilized in generation of this data. Rapid corrosion of the chemically treated aluminum alloy specimens was effected by exposing these specimens to a boiling 5% NaCl solution at approximately 15 psia with added gaseous oxygen being introduced into the pressure vessel during test. This test method produced more severe corrosion than the standard 7 day salt fog tests. Standardization of variables of heat up rate, cool down rate and oxygen input are needed to utilize this test for production acceptance testing.

2.0 TEST PROCEDURES

2.1 ADHESION MEASUREMENTS BY BLISTER TEST METHOD

The Blister Test Method application to coating adhesion which was developed under a previous program (1) was utilized to determine adhesion characteristics of organic coatings to anodized surfaces. This is a relatively simple test in which the adhesion is measured by applying fluid under pressure through a hole in the substrate under the coating. From the pressure required to lift the coating from the substrate and the blister-height, the adhesion strength can be determined.

The Blister Test Method had been utilized to measure adhesive stengths of other materials. A similarity between certain problems of adhesion and fracture was discussed by Williams (2,3). It was noted that in both cases, if one considers the elastic stress analysis in the neighborhood of a sharp crack (or slight region of non-adhesion), a singularity in stress is found to exist. In the case of a central finite length crack in an infinite sheet subjected to tension, the classic Griffith problem gives a local stress variation which is proportional to the inverse square root of the distance from the crack tip.

Since this (mathematical) infinite stress exists here for even the smallest loading, it appears that instantaneous fracture would occur and that stress analysis would not be useful for predicting a finite stress which the sheet could withstand before fracture. However, Griffith (4) developed an overall energy balance, which incorporated the integrable stress singularity, by equating the reduction in strain energy to the energy required to create new surfaces. The result was the prediction of a finite applied tensile stress, σ_{cr} needed to initiate fracture, namely:

$$\sigma_{cr} = \sqrt{\frac{2E\ \gamma_c}{\pi\ a}}$$

where E and γ_c are the Young's modulus and energy to create new fracture surface, respectively, and 2a is the finite length of the crack in the thin sheet. Thus, the use of the integrated energy balance circumvented the question of how infinite the stress need become before fracture. It also suggests the way in which other problems in stress analysis having stress singularities can be attacked in order to predict a finite stress at failure notwithstanding an infinite stress at the origin of the fracture initiation.

The character of elastic stress singularities to be expected for various geometric discontinuities was investigated by Williams and later applied to the specific situation of the interface between dissimilar media. In this case, too, when a crack existed along a line of demarcation of the two materials, the stress singularity was likewise singular and the similarity between cohesion and adhesive failure becomes clear. In the Griffith problem the finite length of the central crack 2a lies along the x axis, with the upper and lower half planes occupied by the same material; in the second case, the materials above and below the x axis are different.

The adhesive mechanics approach is straightforward and consists of two parts:

- Conduct the stress analysis for the bonded materials including a flaw at the interface.
- Express the incremental new surface energy (γ_a) as the crack extends.

Williams developed the treatment for the blister test, first proposed by Dannenberg (5) but without the fracture mechanic statement, for determination of the strength of an adhesive (i.e. γ_a).

The samples are easily constructed. The pressure uniformly distributes itself in the flaw, reducing alignment problems. The tests can be conducted with apparatus generally available in research and testing laboratories. To determine the strength of an adhesive only the critical pressure for failure, the flaw size, the system geometry, and the material properties are required. For a circular plate of incompressible elastic material bonded to a rigid plate, with air injected through a hole in the rigid member into a circular unbonded area (see Figure 1) the following relationship was developed:

$$P_c = \left[\frac{32}{3\,(1-\gamma^2)} \left(\frac{h}{a} \right)^3 \right]^{1/2} \sqrt{\frac{E\,\gamma_a}{a}} \qquad (1)$$

where P_c= pressure necessary to initiate adhesive fracture

E = Young's Modulus
h = Plate thickness (coating)
γ = Poisson's Ratio
a = Radius of unbond
γ_a = Adhesive surface energy density

H. B. Jones, and Williams (6) did additional work which showed that the equation could be written in terms of the center plate deflection as:

$$P_c W_0 = 2\,\gamma_a \text{ (plate)} \qquad (2)$$

where W_o = center plate deflection (see Figure 1) which is a convenient description for an experimental test since both the pressure necessary to initiate adhesive fracture and the center deflection at that pressure can be measured directly. The adhesion surface energy density is then calculable. For the same plane form, if the plate is thin and deflections are large, the mid-plane stretching or membrane stresses predominate and criticality can be defined (reference 7).

$$P_c W_o = 2.4\, \gamma_a \quad \text{(membrane)} \tag{3}$$

or

$$P_c = 1.532 \left(\frac{h}{a^{(3/4)}} \right) \sqrt[4]{\frac{E\, \gamma a}{a}} \tag{4}$$

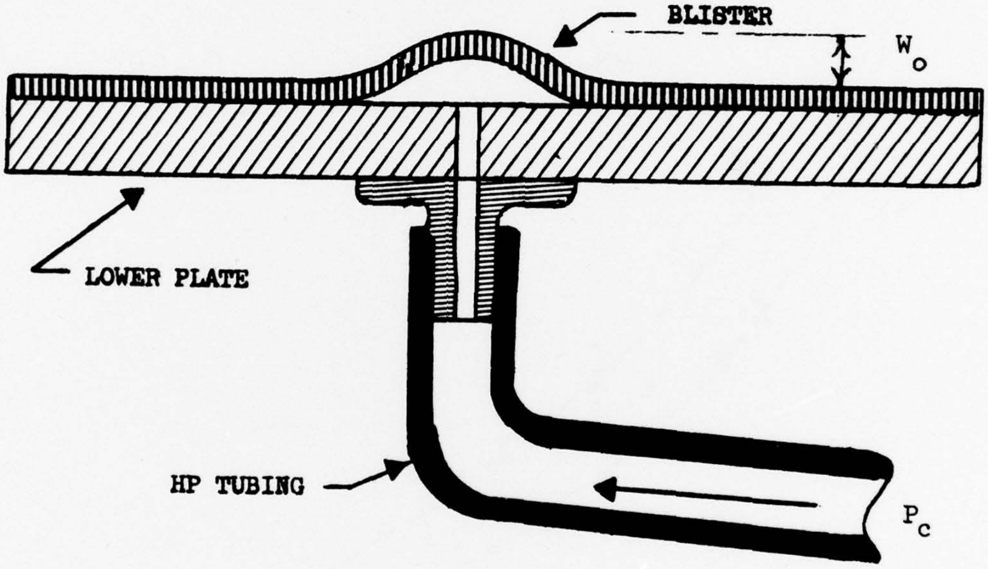


FIGURE 1 SCHEMATIC OF ADHESIVE TEST SPECIMEN

Tests using filled and unfilled elastomers indicate that the response of a specimen undergoes a rather smooth transition from plate to membrane behavior over a relatively narrow range of increasing deflections. This transition is indicated by the x's on Figure 2. This orderly transition in behavior, then, appears to present no insurmountable difficulty relative to the analysis of blister test data.

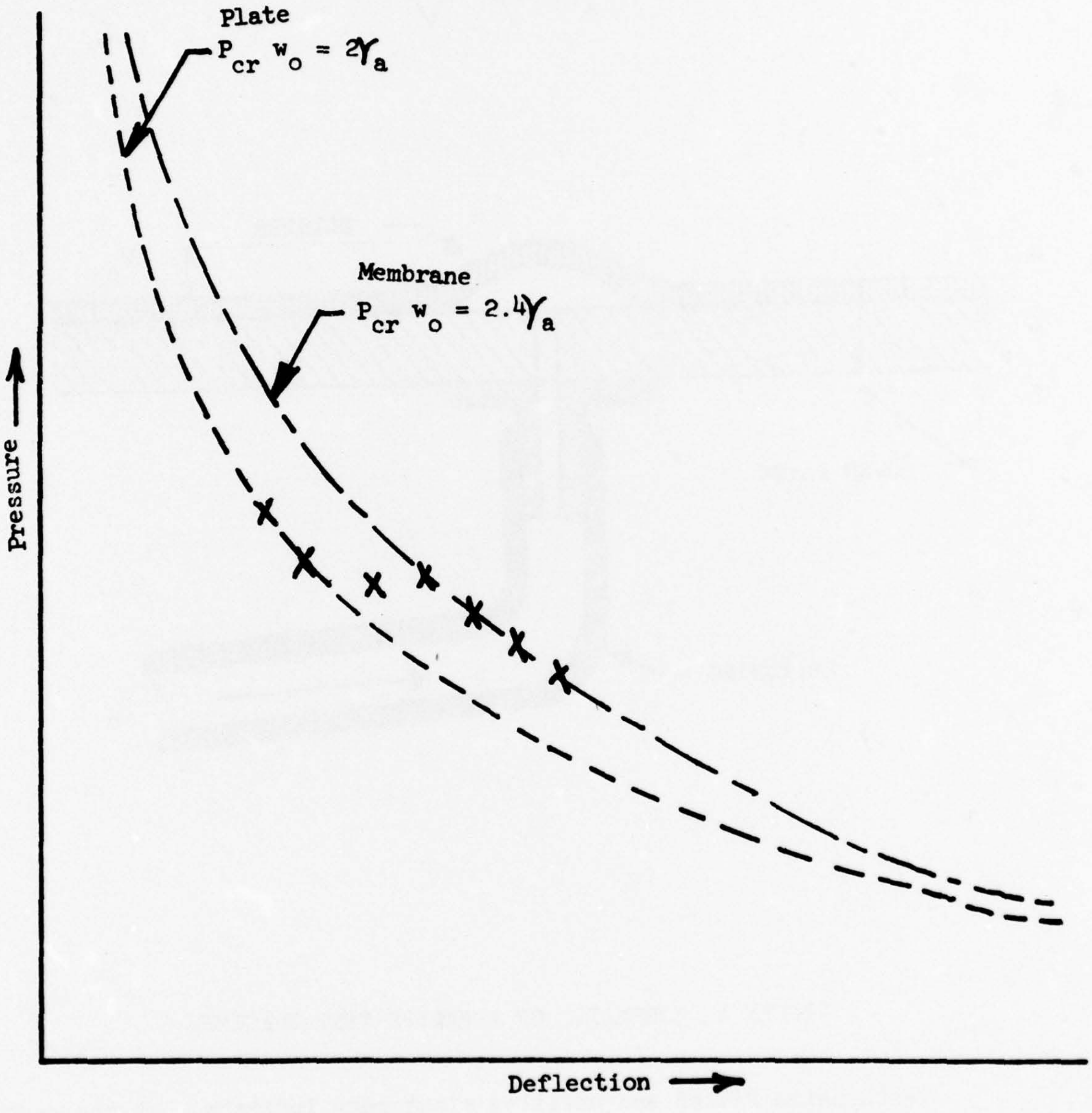


FIGURE 2 CHARACTERISTIC SPECIMEN PRESSURE-DEFLECTION AND FRACTURE BEHAVIOR

During repeated tests on a specimen, where deflections may range from small to large, there will, of course, be intermediate values for which neither the plate solution nor the membrane solution is valid.

The analysis has also been developed for other geometries such as a bond between two disks of different elastic properties (double blister), for two rigid plates bonded together by an adhesive and for multiple layers of elastic materials bonded to a rigid plate. All of these can be handled mathematically and according to Williams (8) since the value determined is γ_a and the relationship includes this thickness, it is not necessary that the test specimens have the same thickness as the practical coating.

Depending on the system to be studied the test apparatus may be as simple as that shown in a paper by Williams, et al (9), or a more sophisticated one such as that diagrammed in Figure 3. This was used at Vought in the previous study (1).

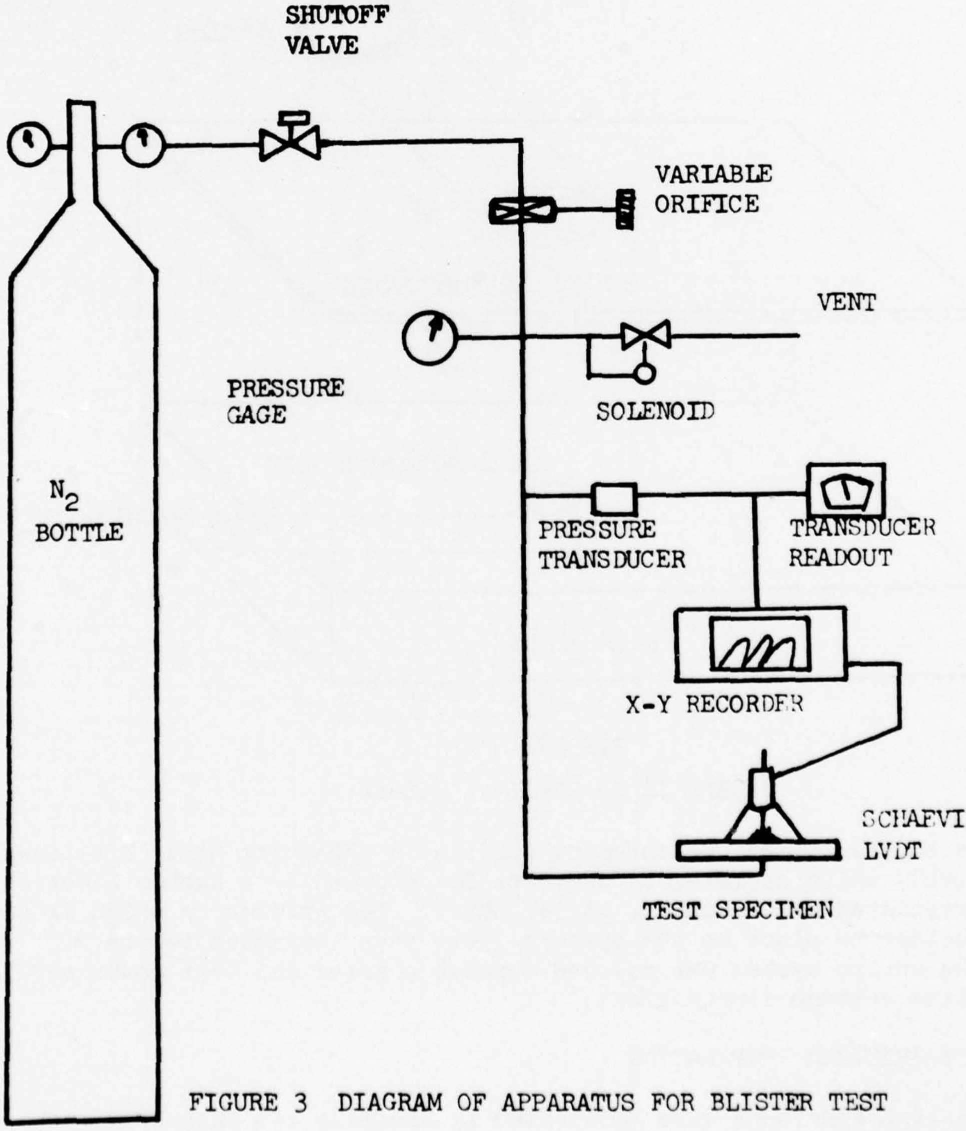


FIGURE 3 DIAGRAM OF APPARATUS FOR BLISTER TEST

2.2 DESIGN AND ASSEMBLY OF TEST EQUIPMENT

The "Blister Test" apparatus was assembled as diagrammed in Figure 3 using a Statham Instrument Inc. strain gauge, model UC3 in a body shell adapter, model U6P4-B in which selected diaphrams may be installed. A Statham Universal Readout, model SC1001 was attached to the pressure transducer and then relayed to a Hewlett Packard X-Y recorder, model 7004B. The system was pressurized with cylinder nitrogen through a cylinder pressure gauge/regulator, cutoff valve and a micrometering valve, model 1B22RS4. System pressure was also monitored, and calibrated with a Ashcroft 0-60 psig. test guage which had previously been calibrated. A 110 volt solenoid operated pressure release valve and specimen holder, Figure 4 completed the pressurization side of the apparatus.

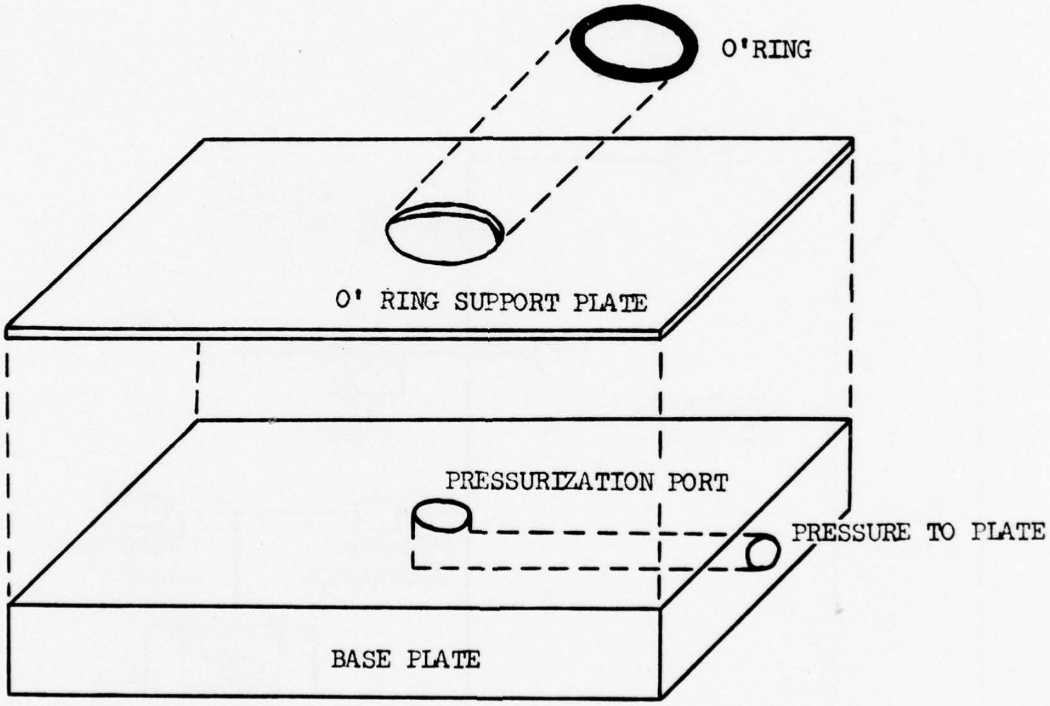


FIGURE 4

SCHEMATIC OF SPECIMEN HOLDER

The blister height measurement utilized a Schaevitz Liner Displacement Transducer (LVDT) which operated on 32 volts DC supplied by a Lambda Electronics Corporation regulated power supply, series LCS-4. The transducer which is mounted in a tripod holder to place on the specimen, was then connected to the X-Y recorder. The entire system was powered through a Freed 115 volt power regulator to minimize line voltage fluctuations.

2.3 TEST SPECIMEN PREPARATION

The test specimens were fabricated by shearing a 3 inch by 3 inch specimen from approximately 1/4 inch sheet stock. A 1/4 inch diameter hole was then punched in approximately the center. The simplified specimen design was made possible by using an "o-ring" and retainer plate with clamps to hold the specimen to the test assembly specimen holder to form a seal.

Prior to coating the specimens after all processing had been completed a 3/4 inch masking tape disk was placed over the pressurization port to serve as the initial flaw or blister.

2.4 SPECIMEN CONDITIONING

Specimens were evaluated in both the "as prepared" condition, as well as after being subjected to salt fog corrosion tests, condensing humidity at 165°F and in boiling deionized water. The "as prepared" specimens were held under laboratory storage conditions with no special humidity controls.

2.5 TEST SPECIMEN EVALUATION

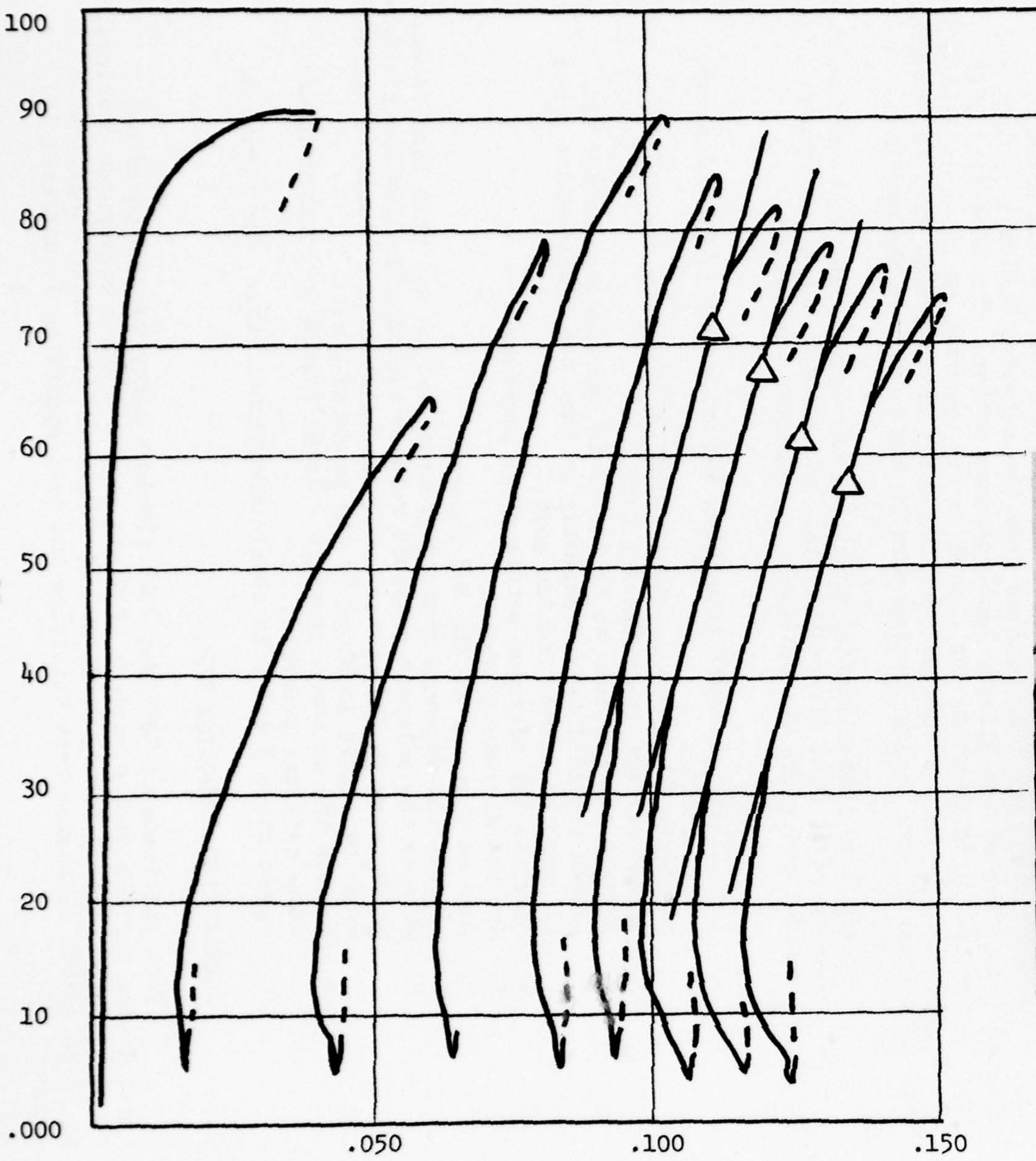
Before the specimens were subjected to Blister Test evaluation all specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days. The specimens were then removed from the relative humidity cabinet and clamped into place on the Blister Test specimen holder. The Schaevitz LVDT tripod, previously calibrated, was placed on the specimen as shown in Figure 5. The displacement probe was located over the built in flaw made with the masking tape disk covering the pressurization port in the center of the specimen.

Previous to installing the specimen, the system was calibrated so that the X-Y recorder would read directly in psig and blister height in inches. The following general procedure was followed for specimen evaluation.

1. Install the pressure transducer assembly with proper diaphram and calibrate.
2. Calibrate the X-Y recorder to plot signals from the pressure transducer and the Schaevitz LVDT.
3. Install the specimen to be tested in the specimen holder and locate the LVDT tripod assembly so that the displacement probe is over the pressurization port.
4. Regulate the nitrogen bottle gauge pressure.
5. Set the micrometer valve.
6. Turn on thepower to the X-Y recorder.
7. Depress the solenoid pressure release switch and open hand valve.
8. Release the solenoid pressure release switch and allow the pressure to increase in the system until the blister size starts to increase noted by change in slope of the produced curve.
9. Depress the solenoid pressure release switch to lower pressure below critical pressure.
10. Repeat steps 8 and 9 to obtain additional data. See Figure 6.

2.6 ACCELERATED CORROSION TEST

Test specimens of 7075 and 2024 aluminum alloy were prepared and treated with various concentrations of Class 1A and Class 3 conversion coatings. Specimens were treated for 1, 3, 5, and 10 minutes before being subjected to the accelerated corrosion test utilizing the test apparatus illustrated in Figure 7.



	P_{cr}	W_o	a	
1	70.0	.109	3.2	
2	66.2	.118	3.3	
3	60.3	.125	3.1	
4	56.3	.133	3.1	
			3.2	Average

FIGURE 6 EXAMPLE OF BLISTER TEST DATA

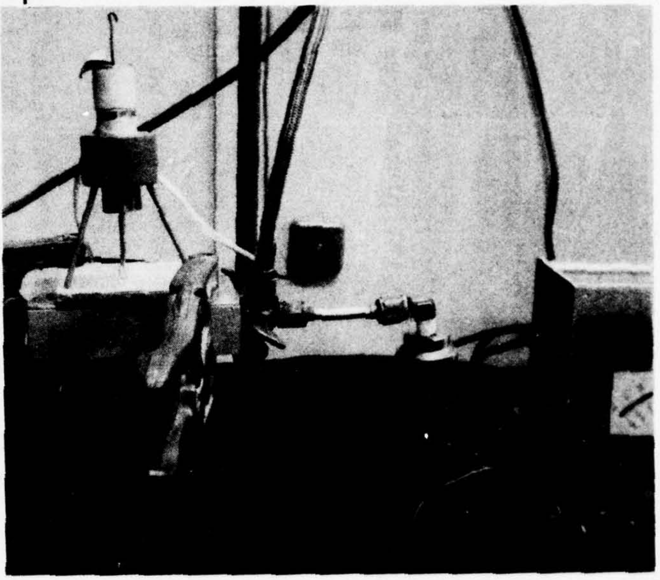


FIGURE 5 SCHAEVITZ LVDT ON SPECIMEN

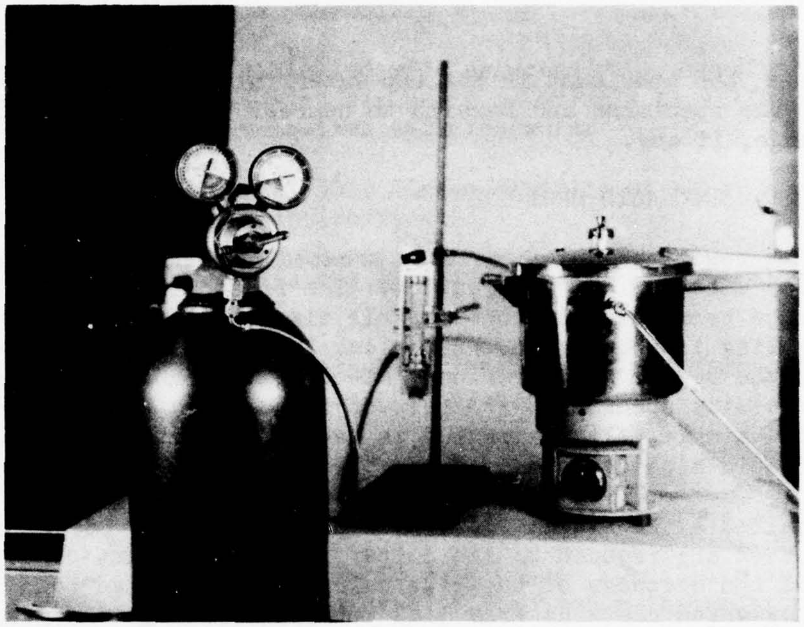


FIGURE 7
ACCELERATED CORROSION TEST APPARATUS INCLUDING PRESSURE
COOKER, FLOW METER, AND COMPRESSED OXYGEN CYLINDER

Accelerated Corrosion Test Procedure

1. Turn hot plate to high heat with pressure vessel in place.
2. Add 2500 ml of 5% by weight sodium chloride (NaCl) solution.
3. Place weighed specimens in rack inside pressure vessel.
4. Regulate oxygen to fast bubbling rate at 35 psig with micrometer valve.
5. When the salt solution begins to boil, close top of pressure vessel and close top putting pressure regulating popoff valve in place.
6. Time test start from first pressure release of popoff valve. Boil specimens at 15 psia for 30 minutes.
7. Turn heat to low for duration of test.
8. Upon termination of the test, remove pressure vessel from hot plate and place on a cool table top or large plate of metal to reduce temperature of the pressure vessel quickly. Leave oxygen flowing during cool down to prevent siphoning salt solution back to the metering valve.
9. When pressure is reduced to 1 atmosphere approximately, remove lid from pressure vessel, empty salt solution and turn off oxygen flow.

10. Rinse the specimens in hot tap water followed by cool distilled water.
11. Dry the specimens and reweigh to nearest 0.1 mg. Determine weight change, if any.

2.7 PHOSPHOMOLYBDIC ACID SPOT TEST

Corrosion resistance of chemically treated or anodized surface is of prime interest along with adhesion characteristics. The presently accepted salt fog corrosion resistance test requires considerable time to complete in order to determine if a coating is acceptable. After completion, some cases are difficult to classify as to the degree of corrosion. Electrical conductivity tests and visual tests to classify specimens with respect to severity of corrosion are also time consuming, and a more reliable method is needed to classify a satisfactory coating. A more rapid method would be desirable and Boeing Vertol Company (14) developed a quick test which showed merit. The test is a basic spot test for copper as detailed by F. Fiegel (15) which produces a "molybdenum blue" when the phosphomolybdates are reduced by the copper ion. The amount of copper present determines the deepness of the blue color. The work had been done on 2024 aluminum alloy which contains from 3.8% to 4.9% copper so the measurement of copper on the surface of the anodize surface was felt to be indicative of the effectiveness of the anodize coating.

Boeing's evaluations indicated that a positive reaction; i.e., the development of a blue color, occurred on all specimens of chromic acid anodized 2024 aluminum alloy whose salt spray endurance was less than 12 days. No reaction occurred on specimens which lasted 14 days or longer in the salt spray. Specimens in the 12-14 day endurance range were marginal and could either fail or pass the spot test.

The test method consisted of dipping an anodized specimen into a 10% solution of phosphomolybdic acid containing a small amount of wetting agent. The specimen was then placed on a paper towel and allowed to stand for 1.5 minutes. The specimens were then examined for evidence of a blue color which would indicate failure. Any edge effect was disregarded.

3.0 TEST PROCEDURE

3.1 TASK I - COATING OF COMPOSITES

Three composites evaluated were selected with coordination of the technical monitor. The selection was based on present or anticipated use on Navy aircraft. The three composites selected were:

Rigidite 5208 Thornel 300; Narmco Division of Whittaker Corporation; Costa Mesa, CA. (Graphite/Epoxy)

AVCO 5505/4; AVCO Systems Division; Lowell Industrial Park; Lowell, MA. (Boron/Epoxy)

Hercules 3501/AS1-6; Hercules, Incorporated, Systems Group; Bacchus Works; Magna, Utah. (Graphite/Epoxy)

A four ply laminate (oriented 90°, 0°, 0°, 90°) of each graphite/epoxy prepreg bonded to a sheet of .250 aluminum alloy (2024) was fabricated. A portion of each panel was laid up with a peel ply. On the remainder of the panel, TX 1040 parting material, as specified in Vought specification 208-8-401/2, was used. The autoclave cure cycle was in accordance with Vought specification 208-8-8C for the Narmco material and in accordance with the vendor instructions for the Hercules prepreg. A two ply boron/epoxy panel was prepared and bonded to a .250 aluminum alloy plate utilizing a peel ply on a portion of the layup. The panel was autoclave cured in accordance with Vought specification 208-8-8C.

The three panels were then cut into three inch by three inch specimens and a 1/4 inch hole was drilled in the approximate center to serve as a pressurization port during evaluation. The specimens were then marked for identification. A total of 144 specimens was prepared for coating adhesion evaluation by the Blister Test Method.

The specimens prepared from the three composites (Rigidite 5208-Thornel 300, AVCO 5505/4, and Hercules 3501/AS1-6) were processed for coating and coated. Five methods were used to prepare the surface for coating after the specimens had been contaminated with MIL-H-5606 hydraulic fluid. All specimens were wiped to remove the hydraulic fluid and then wiped with a rag wetted with 1,1,1 trichloroethane to remove any remaining hydraulic fluid.

A set of nine specimens each will be given supplementary treatments in order to improve adhesion as follows:

- Removal of a peel ply
- Hand sanding
- Grit blasting
- Wiping with a methylene chloride wetted cloth
- No additional treatment

The prepared specimens were then placed on sheets of cardboard with double-back masking tape in a random order. A paint masking disk (3/4 inch diameter) was placed over the pressurization port. A light coat of MIL-P-23377 epoxy polyamide primer was applied to the surface followed by a normal primer coat. The specimens were then coated with MIL-C-81773 polyurethane coating using a cross coat pattern. Additional applications of the polyurethane coating were made until a coating thickness of .035 inches to .040 inches was obtained. This thickness of coating is necessary when using the Blister Test Method to prevent cohesive failure at the edge of the masking disk. The specimens were allowed to cure at room temperature prior to subjecting these to hostile environments before adhesion evaluation.

A total of 135 specimens was thus prepared for coating and coated at the same time to minimize coating variables.

3.2 TASK II - CONTINUATION OF ANODIZING OPTIMIZING STUDIES

Adhesion specimens were included in this task. Specimens of 2219, 6061, 7050 and 7075 aluminum alloys were sheared to 3" x 3" x approximately 1/4". The 7050 specimens were stripped of the clad by abrasion. The remainder of the materials were in the unclad condition. A 1/4" hole was punched in the approximate center of each specimen to serve as a pressurization port. Specimens were marked for identification and anodized. Four anodizing variations were evaluated as follows: All specimens were anodized in production anodizing facilities. The sulfuric acid anodizing was in accordance with CVA 9-14 "Process Specification, Anodic Treatment, Sulfuric Acid, of Aluminum and Aluminum Alloys." One set of sulfuric acid anodized specimens was sealed in 100 ppm chromic acid solution at 190°F ± 10°F instead of 5% sodium dichromate solution required by the specification. Two chomic acid anodizing processes were utilized, Type A and Type B in accordance with CVA 9-4 "Process Specification, Chomic Acid Anodic Treatment of Aluminum and Aluminum Alloys."

Coating of the specimens was accomplished in two batches because the Type B, 22 volt chromic acid anodize specimens were delayed due to operational problems in the plant. The specimens were mounted on cardboard in random order with double back pressure sensitive tape. The pressurization ports of the specimens were covered with a 3/4 inch diameter paint masking disk in preparation for coating. A light coat of MIL-P-23377 epoxy polyamide primer was applied by spraying followed by a normal primer coat. The specimens were then coated with MIL-C-81773 polyurethane coating using a cross coat pattern. Additional applications of polyurethane coating were made until a coating thickness of .035 inches to .040 inches was obtained. This thickness of coating has been found to be necessary to prevent cohesive failure at the edge of the paint masking disk which serves as the built-in flaw for Blister Testing. The specimens were allowed to cure at room temperature for a minimum of 10 days.

The specimens prepared from each alloy and each anodize variation were divided into three groups. One of the groups was held in the "as prepared" condition. Another group of specimens was placed in a glass container on a rack over deionized water for condensing humidity bath held at 165°F ± 5°F approximately half submerged and held for 7 days. The third group of specimens was conditioned by submerging the specimens in deionized water which was heated to boiling and maintained at boiling temperature for four hours. This conditioning was accomplished the same day the 7-day conditioning was completed. Both groups of specimens were wiped dry after conditioning and placed in a 50% relative humidity with the "as prepared" groups. All specimens were held at 50% relative humidity for a minimum of 7 days prior to evaluation by the Blister Test Method. The specimens were evaluated for adhesion surface energy density soon after removal from the humidity chamber with the pressure versus the blister height recorded on an X-Y recorder. The data was then reduced to obtain the reported values in inch-pounds force per square inch of surface area.

The 7075 aluminum alloy specimens were reheat-treated to produce a bad heat treat and sulfuric acid anodized along with 7075-T6 aluminum alloy specimens. In addition 7075-T6 aluminum alloy specimens were sulfuric acid anodized interrupting the current after 10 minutes and then continuing the process to simulate a power failure.

Three specimens of each anodize batch were placed in the salt spray cabinet to measure the salt spray endurance. Specimens were salt fog tested per Federal Test Method Standard No. 141, method 6061 using a 5% sodium chloride solution.

3.3 TASK III - ADHESION STUDIES OF CHEMICAL CONVERSION COATING OF ADDITIONAL ALLOYS

The adhesion test specimens were prepared from 2219, 6061, 7050, and 7075 alloys and conversion coated using two commercial materials as described for Task III. Specimens were coated with the standard Navy paint system to a dry film thickness of 30 to 40 mils as described in Task I above. Again, specimens were conditioned for four hours in boiling distilled water and 7 days in a condensing humidity. Controls were cured at room temperature only. Adhesion surface energy density values were obtained for these specimens via the Blister Test Method. The data was tabulated.

3.4 TASK IV - CHEMICAL CONVERSION COATING ON ALUMINUM ALLOYS AND ACCELERATED CORROSION TEST PROCEDURE

The 7075 specimens were .070 thick without a hole in the center. The 2024 specimens were .25" thick with a 1/4" diameter in the approximate center. Specimens were cleaned per CVA9-18 and chemically treated with Class 1A and Class 3 MIL-C-5541 treatments at 2 oz./gal. and 4 oz./gal. solution concentration. Specimens were weighed to the nearest 0.1 milligram after treatment and stripped per MIL-C-5541 using a 50% solution of nitric acid. Specimens were reweighed after stripping and the coating weight per square foot was calculated. Sixty-four specimens were processed for coating weight determination.

Accelerated corrosion tests were performed utilizing a set of specimens of all alloys and solutions/concentrations described in the above paragraph. All specimens were given the phosphomolybdic acid spot test before and after the accelerated corrosion test. Each specimen set was given the accelerated corrosion test as described in the test procedure (paragraph 2.2) for a duration of 90 minutes (30 minutes at boiling at 15 psia). Approximately 45 minutes time was required for the set of specimens and the pressure vessel containing 2500 ml of 5% sodium chloride solution to come to a boil. About 15 minutes were required for depressurization and cool down time. After corrosion testing, specimens were washed in hot tap water, followed by cold deionized water rinse. Specimens were dried and given the phosphomolybdic acid spot test and inspected visually for pitting. Data was compiled in tabular form for these tests in Table IX.

The use of phosphomolybdic acid on the panels prior to testing was done to establish if any copper was present on the surface on these specific panels prior to corrosion testing. The question arises as to whether this would influence the subsequent accelerated corrosion test. Phosphomolybdic acid is a strong oxidizer and would act similarly to the chromate reagent used in the chemical conversion coating so that no adverse effect on corrosion resistance is anticipated. However, this was not specifically demonstrated in this program so that the prior exposure to the phosphomolybic acid should be considered in evaluation of the accelerated test results.

4.0 RESULTS

4.1 ADHESION OF THE NAVY PAINT SYSTEM TO NON-METALLIC COMPOSITES RESULTS

Adhesion of the Navy Paint System to non-metallic composites was found to be adequate. The test data obtained showed that any reasonable surface preparation will yield satisfactory adhesion to the epoxy composites. One additional bit of information gained during the evaluation was that the failure mode was not on the substrate surface as has been the case with most metallic specimens but was a cohesive failure in the epoxy-polyamide primer layer. Figure (8) shows a typical specimen which was dissected after failure. It will be noted that the center of the specimen was covered with a paint masking disk and received no primer or coating. The gray area marked (1) was primed and painted. Area marked (2), a bright yellow, is the top coat and primer removed from the surface marked (1). This area is yellow from the primer, although some black shows through. The adhesion failure was for the most part within the primer since primer was retained on both faces of the failure interface.

Adhesion data for the Standard Navy System to 5208-T300 graphite epoxy, 5505/4 boron epoxy and 3501/AS1-6 graphite epoxy are shown in Tables I, II, and III, respectively.

4.2 CONTINUATION OF ANODIZING OPTIMIZATION STUDY RESULTS

The adhesion evaluation of the Type B chromic acid specimens which were coated in a different batch of specimens yielded low to unacceptable adhesion values in the "as prepared" condition. Examination of a number of these specimens showed the failure to be cohesive within the primer layer.time of painting. It was also noted that the values on the specimens which had received 4 hours boiling water conditioning were much higher and within the range of values obtained on the other anodizing methods evaluated. The data obtained on the condensing humidity specimens were very erratic and in some instances blisters were noted on the specimens prior to Blister Test evaluation. Examination of these specimens showed that the failure was cohesive in the primer layer except at the blister locations caused by the condensing humidity condition where the failure was adhesive between the substrate and the primer.

From this data it appeared that the primer had not cured satisfactorily at room temperature cure in the time elapsed between coating and evaluation. but the additional temperature, 4 hours in boiling water advanced the cure to a satisfactory level. This also explains erratic values obtained on the condensing humidity specimens. Where blisters or beginning of blisters occurred in the test area of the specimens, low adhesion would be recorded, but where cure was advanced in non-blister areas by the elevated temperature the values were satisfactory. Thus, osmotic pressure from water or water vapor decreased the amount of test pressure needed to lift the coating.

Adhesion surface energy density data was compiled for additional aluminum alloys and presented in Table IV according to anodize type and conditioning treatment. The "as prepared" specimens were found to have adhesion surface energy densities that were generally lower than those for the conditioned specimens; although these values were somewhat lower than those obtained for the exposed specimens, in most cases, acceptable to good adhesion values (1 inch pound per square inch) were obtained. Mean adhesion values were high for all types of anodize evaluated except the low voltage, Type B Chromic which yielded good values.

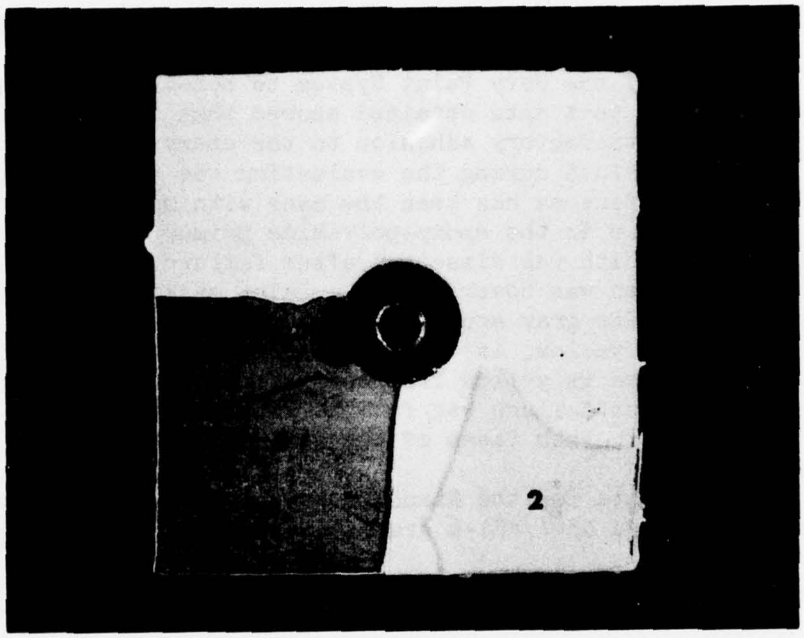


FIGURE 8

TYPICAL COMPOSITE SPECIMEN SHOWING ADHESION FAILURE LOCATION

TABLE I

ADHESION CHARACTERISTICS OF A GRAPHITE/EPOXY COMPOSITE
(Rigidite 5208-Thornel 300, Narmco Division, Whittaker Corp., Costa Mesa, CA)

SPECIMEN NUMBER	PRECOATING SURFACE PREPARATION	M(1) S	SPECIMEN CONDITIONING AFTER COATING	ADHESION γ_a(2)
A-1	Surface wiped with 1,1.1 trichloroethane then peel ply removed		7 days condensing humidity at 165°F ± 5°F	1.7
A-2				1.7
A-3				1.5
A-4			4 hours submerged in boiling deionized water	2.5
A-5				1.8
A-6				3.1
A-7		M=1.96	As prepared	2.3
A-8		S=0.24		2.2
A-9				1.8
C-1	Surface wiped with 1,1.1 tricholoroethane then hand sanded to remove glaze		7 days condensing humidity at 165°F ± 5°F	1.4
C-2				1.5
C-3				1.8
C-4			4 hours submerged in boiling deionized water	2.0
C-5				2.0
C-6				1.9
C-7		M=1.96	As prepared	2.1
C-8		S=0.24		2.1
C-9				2.3
C-10	Surface wiped with 1,1,1 trichloroethane then grit blasted to remove glaze		7 days condensing humidity at 165°F ± 5°F	2.3
C-11				No results
C-12				1.4
C-13			4 hours submerged in boiling deionized water	1.7
C-14				1.9
C-15				1.8
C-16		M=1.97	As prepared	2.5
C-17		S=0.38		2.3
C-18				2.2
C-19	Surface wiped with 1,1,1 trichloroethane then wiped with methylene chloride		7 days condensing humidity at 165°F ± 5°F	1.8
C-20				1.6
C-21				1.5
C-22			4 hours submerged in boiling deionized water	2.4
C-23				1.5
C-24				1.8
C-25		M=1.91	As prepared	2.0
C-26		S=0.39		2.5
C-27				2.0
C-28	Surface wiped with 1,1,1 trichloroethane only		7 days condensing humidity at 165°F ± 5°F	1.6
C-29				1.6
C-30				1.5
C-31			4 hours submerged in boiling deionized water	1.5
C-32				1.9
C-33				2.0
C-34		M=1.95	As prepared	2.1
C-35		S=0.41		2.6
C-36				2.4

1. M/S: M=Mean Value of sample, S=Standard deviation of sample.
2. γ_a = Adhesion Surface Energy Density in in.-lbs./in.^{2}.

TABLE II

ADHESION CHARACTERISTICS OF A BORON/EPOXY COMPOSITE

(AVCO 5505/4, AVCO Systems Division, Lowell Industrial Park, Lowell, Ma.)

SPECIMEN NUMBER	PRECOATING SURFACE PREPARATION	M(1) / S	SPECIMEN CONDITIONING AFTER COATING	ADHESION γ_a (2)
X-1	Surface wiped with 1,1,1 trichloroethane then peel ply removed		7 days condensing humidity at 165°F±5°F	2.0
X-2				1.8
X-3				1.6
X-4			4 hours submerged in boiling deionized water	2.5
X-5				1.9
X-6				2.1
X-7		M=2.03	As prepared	2.1
X-8				2.1
X-9		S=0.27		2.1
B-1	Surface wiped with 1,1,1 trichloroethane then hand sanded to remove glaze		7 days condensing humidity at 165°F±5°F	1.9
B-2				1.7
B-3				1.6
B-4			4 hours submerged in boiling deionized water	1.6
B-5				1.5
B-6				1.6
B-7		M=1.83	As prepared	2.1
B-8				2.2
B-9		S=0.32		2.3
B-10	Surface wiped with 1,1,1 trichloroethane then grit blasted to remove glaze		7 days condensing humidity at 165°F±5°F	1.8
B-11				1.8
B-12				1.8
B-13			4 hours submerged in boiling deionized water	1.7
B-14				1.3
B-15				1.7
B-16		M=1.93	As prepared	2.3
B-17				2.1
B-18		S=0.43		2.7
B-19	Surface wiped with 1,1,1 trichloroethane then wiped with methylene chloride		7 days condensing humidity at 165°F±5°F	1.2
B-20				2.0
B-21				1.1
B-22			4 hours submerged in boiling deionized water	1.6
B-23				1.4
B-24				1.9
B-25		M=1.79	As prepared	2.0
B-26				2.3
B-27		S=0.39		2.0
B-28	Surface wiped with 1,1,1 trichloroethane only		7 days condensing humidity at 165°F±5°F	1.4
B-29				1.8
B-30				1.7
B-31			4 hours submerged in boiling deionized water	2.1
B-32				1.9
B-33				1.9
B-34		M=1.94	As prepared	2.0
B-35				2.1
B-36		S=0.14		2.0

1. M/S: M=Mean for sample, S=Standard deviation for sample
2. γ_a = Adhesion Surface Energy Density in in.-lbs./in.^{2}

TABLE III

ADHESION CHARACTERISTICS OF A GRAPHITE/EPOXY COMPOSITE

(Hercules 3501/AS1-6, Hercules Incorporated, Systems Group, Bacchus Works, Hagna, Utah)

SPECIMEN NUMBER	PRECOATING SURFACE TREATMENT $\frac{M^{(1)}}{S}$	SPECIMEN CONDITIONING AFTER COATING	ADHESION $\gamma_a^{(2)}$
S-1	Surface wiped with 1,1,1 trichloroethane then peel ply removed M=1.96 S=0.18	7 days condensing humidity at 165°F±5°F	2.7
S-2			1.7
S-3			1.9
S-4		4 hours submerged in boiling deionized water	1.8
S-5			2.2
S-6			2.2
S-7		As prepared	2.0
S-8			1.9
S-9			2.0
D-1	Surface wiped with 1,1,1 trichloroethane then hand sanded to remove glaze M=1.86 S=0.24	7 days condensing humidity at 165°F±5°F	2.1
D-2			2.3
D-3			1.9
D-4		4 hours submerged in boiling deionized water	1.8
D-5			1.7
D-6			1.5
D-7		As prepared	1.7
D-8			2.0
D-9			2.0
D-10	Surface wiped with 1,1,1 trichloroethane then grit blasted to remove glaze M=1.60 S=0.32	7 days condensing humidity at 165°F±5°F	1.0
D-11			1.4
D-12			1.2
D-13		4 hours submerged in boiling deionized water	2.1
D-14			1.8
D-15			1.7
D-16		As prepared	1.4
D-17			1.8
D-18			2.0
D-19	Surface wiped with 1,1,1 trichloroethane then wiped with methylene chloride M=1.70 S=0.29	7 days condensing humidity at 165°F±5°F	1.2
D-20			1.5
D-21			1.8
D-22		4 hours submerged in boiling deionized water	2.0
D-23			1.3
D-24			1.7
D-25		As prepared	1.6
D-26			2.2
D-27			1.5
D-28	Surface wiped with 1,1,1 trichloroethane only M=2.13 S=0.29	7 days condensing humidity at 165°F±5°F	1.8
D-29			1.9
D-30			1.7
D-31		4 hours submerged in boiling deionized water	1.8
D-32			2.3
D-33			2.2
D-34		As prepared	2.2
D-35			2.5
D-36			2.4

1. M/S: M= Mean value of sample, S= Standard deviation of sample
2. γ_a = Adhesion Surface Energy Density, in in.-lbs./in.^{2}

TABLE IV

ADHESION CHARACTERISTICS OF ANODIZED ALUMINUM

ANODIZE TREATMENT	M(5) S	CONDITIONING TREATMENT	Y_a(1) BY ALUMINUM ALLOY			
			2219	6061	7050	7075
Sulfuric Acid CVA 9-14 Except 100 ppm Chromic Acid Seal		Condensing Humidity 7 days at 165°F	2.9	2.2	2.2	2.6
			2.7	2.9	2.2	2.8
			2.7	2.9	2.1	3.0
		Boiling Deionized Water 4 Hours	2.8	2.0	3.1	2.9
			2.4	2.1	1.7	2.7
			2.3	2.7	2.5	2.8
	M=2.35 S=0.32	None "as prepared"	2.2	2.2	1.7	2.1
			2.4	1.7	2.1	2.0
			2.1	2.0	1.9	2.1
Sulfuric Acid CVA 9-14		Condensing Humidity 7 days at 165°F	2.9	2.5	2.3	2.6
			2.7	3.0	2.1	2.4
			2.6	2.3	2.5	2.8
		Boiling Deionized Water 4 Hours	2.1	2.9	2.4	2.7
			2.3	2.5	2.7	2.3
			2.5	2.7	2.6	2.1
	M=2.38 S=0.24	None "as prepared"	2.6	2.1	1.7	2.3
			2.2	2.0	2.0	2.4
			2.4	1.9	2.2	2.4
Chromic Acid CVA 9-4 Type B 22 Volts		Condensing Humidity 7 days at 165°F	2.0	0.3 (3)	0.6(4)	1.7
			1.6	1.9	1.1(4)	1.3
			1.6	1.6	2.6	1.6
		Boiling Deionized Water	2.7	3.1	3.1	2.9
			2.0	3.1	3.0	1.5
			2.8	1.4	1.4	2.5
	M=1.83 S=0.68	None "as prepared"	1.4	1.1	0.9	1.1
			1.3	1.2	0.9	1.1
			1.2	1.1	0.7	1.2
Chromic Acid CVA 9-4 Type A 40 Volts		Condensing Humidity 7 days at 165°F	2.8	2.5	1.9	2.8
			3.1	2.1	2.6	2.4
			3.2	2.4	1.9	2.3
		Boiling Deionized Water	2.8	2.4	2.5	2.9
			2.2(2)	3.1	2.5	2.4
			3.3	2.6	2.2	2.4
	M=2.43 S=0.27	None "as prepared"	2.5	2.5	2.2	2.7
			1.8	1.9(2)	2.2	2.5
			2.2	2.3	2.0	2.0
			M=2.37	M=2.20	M=2.06	M=2.28
			S=0.52	S=.65	S=0.64	S=0.55

1. Adhesion surface energy density in.-lbs. / in.^{2}
2. Flaw in specimen coating.
3. Adhesive failure
4. Blister under paint prior to test
5. M/S: M=Mean of sample, S=Std. dev. of sample

Average M=2.18
Average S=0.59

Results from the salt fog corrosion resistance tests of 7075 aluminum alloy produced by different heat treatment procedures failed to produce a variance in endurance time as had been anticipated. The 7075 aluminum alloy specimens which were anodized after improper heat treating to the T6 condition were still satisfactory after 60 days in the salt spray. Also the 7075 aluminum alloy specimens which were anodized by interrupting the current during processing passed the 60 day salt spray endurance testing just as satisfactcrily as the properly heat teeated and processed 7075-T6 aluminum alloy specimens. Additional investigation and inquiries failed to find a method which will yield unsatisfactory salt spray corrosion resistance.

4.3 CHEMICAL CONVERSION COATINGS ON ALUMINUM ALLOYS RESULTS

Adhesion of the standard Navy paint system to variously treated aluminum alloy specimens was found to be satisfactory after conditioning. The "exposed" specimens yielded considerably higher adhesion surface energy density than the "as prepared" control specimens. Specimens treated with Class 1A generally yielded higher adhesion surface energy densities than those treated with the Class 3. Tank applied coatings yielded adhesion energies higher than coatings that were applied by abrasion during coating. The spray applied coatings produced the lowest adhesion results of the three application methods evaluated. The mean value for all specimens processed with Class 3 was 1.66 in.-lbs./in.^{2} adhesion surface energy density with an average standard deviation of 0.56 while respective values for samples processed with Class 1A were 1.76 in-lbs/in.^{2} and a 0.56 average standard deviation.

Specimens treated with Class 3 and Class 1A via the tank application method had a mean adhesion surface energy of 1.95 in.-lbs./in.^{2} and 1.91 in.-lbs./in.^{2} and standard deviation of .61 and .50, respectively. Spray applied Class 3 and Class 1A yielded mean values of 1.99 and 1.68 with standard deviation of .56 and .61, respectively. The abrasion applied Class 3 and Class 1A had adhesion surface energy density values of 1.80 and 1.92 in.-lbs./in.^{2} with standard deviation of .64 and .59, respectively. This data was calculated from results presented in Table V and in interest to simplify this data table, some M/S values were deleted from the table, however included in this text. All values, although aberrant as denoted by footnotes, were included in these statistical samples.

4.4 RESULTS OF CHEMICAL CONVERSION COATING CORROSION EVALUATION

Adhesion comparison of Class 1A and Class 3 conversion coatings on 2024 and 7075 aluminum indicated again, that satisfactory adhesion surface energy density can be obtained with these conversion coatings and the Navy paint system. The exposed specimens yielded considerably higher adhesion values than the "as prepared" control specimens. Specimens treated with Class 3 had an adhesion surface energy density of 1.94 in.-lbs./in.^{2} and a standard deviation of 0.60 and the specimens treated with Class 1A resulted in values of 1.87 in.-lbs./in.^{2} and a 0.59 standard deviation. Average of all specimens produced adhesion energies of 1.94, 1.86 and 1.89 in.-lbs./ft.^{2} for tank, spray and abrasion application methods, respectively. Adhesion comparison data for these tests were condensed in Table VI.

TABLE V

ADHESION CHARACTERISTICS OF TWO CHEMICAL CONVERSION COATINGS ON FOUR ALUMINUM ALLOYS

METHOD OF APPLICATION	$\frac{M(4)}{S}$	CONDITIONING TREATMENT	MIL-C-5541 Class 3 $Y_a^{(1)}$ By Alloy				MIL-C-5541 Class 1A $Y_a^{(1)}$ By Alloy			
			2219	6061	7050	7075	2219	6061	7050	7075
		Condensing Humidity 7 days at 165°F	2.1 2.4 2.2	2.0 1.8 2.9	1.9 2.5 2.2	2.5 1.6 1. 1.1	1.7 1.9 1.6	1.8 1.3 1.9	1.8 2.0 1.8	2.1 1. 2.1 2.9
TANK		Boiling Deionized Water	1.7 2.6 2.1	1.6 2.0 2.5	2.9 2.9 2.1	1.7 2.2 1.9	2.0 1.7 2.1	2.9 1.8 1.4	2.6 2.5 2.6	2.8 2. 2.0 2.2
	M=1.84 S=0.57	None "as prepared"	1.3 1.2 1.3	1.6 1.2 1.2	1.1 1.2 1.0	1.3 1.4 1.4	1.1 1.1 1.0	1.3 1.8 1.6	1.1 1.1 1.0	1.6 1.2 1.4
		Condensing Humidity 7 days at 165°F	2.2 0.6(2) 1.1	2.1 2.5 0.2(2)	2.2 1.1(2) 0.9(2)	2.2 1.2 0.9(2)	2.1(2) 2.4 2.6	2.6 2.5 2.5	1.9 2.1 2.0	0.2(2),(3) 1.6 0.3(3)
SPRAY		Boiling Deionized Water	1.5 2.3 1.9	2.0 1.7 2.0	1.8 2.5 2.0	2.0 1.9 2.6	2.2 2.3 2.0	2.7 1.6 1.5	2.6 1.9 2.4	2.2 1.2 1.4
	M=1.54 S=0.58	None "as prepared"	1.0 1.7 1.2	1.1 1.2 1.3	1.2 1.0 1.2	1.3 1.1 1.4	1.0 1.2 1.6	1.6 1.1 1.5	1.4 1.0 1.1	1.2 0.9 1.1
		Condensing Humidity 7 days at 165°F	2.6 1.5 1.8	2.5 1.6 0.9	1.7 1.8 1.7	1.5 2.0 1.9	2.0 2.1 1.8	1.4 1.9(2) 1.7	1.4 2.7 1.8	2.1 2.3 2.9
ABRASION		Boiling Deionized Water	2.3 2.2 2.2	1.9 1.6 2.5	2.1 2.1 1.8	1.2 1.4 2.1	2.7 2.4 2.3	2.3 2.3 2.0	2.1 2.2 1.6	2.3 1.3 3.4
	M=1.63 S=0.45	None "as prepared"	1.0 1.3 1.2	1.0 1.1 1.6	1.4 1.2 1.0	1.3 1.4 1.1	1.0 1.3 1.2	1.2 1.4 1.2	0.9 1.2 1.1	1.1 1.2 1.0
			M=1.71	M=1.68	M=1.72	M=1.58	M=1.80	M=1.81	M=1.77	M=1.69
			S=0.56	S=0.62	S=0.61	S=0.43	S=0.54	S=0.52	S=0.59	S=0.80

1. Adhesion surface Energy density $\frac{in.lb}{in.^2}$
2. Blister under paint prior to test
3. Adhesive failure
4. $\frac{M}{S}$: M= Mean value of sample
   S= Std. dev. of sample

TABLE VI

ADHESION COMPARISON OF TWO CHEMICAL CONVERSION COATINGS ON ALUMINUM ALLOYS

METHOD OF APPLICATION	M(1) S	CONDITIONING TREATMENT	MIL-C-5541 Class 3 Y_a(2) By Alloy		MIL-C-5541 Class 1A Y_a(2) By Alloy	
			2024	7075	2024	7075
TANK		Condensing Humidity 7 days at 165°F	3.1	2.3	2.2	1.2(3)
			2.4	2.8	2.9	1.7
			2.9	2.1	2.0	2.2
		Boiling Deionized Water	1.8	2.4	2.0	2.2
			2.3	2.5	2.2	2.6
			2.0	1.8	2.4	1.8
	M=1.94	None "as prepared"	1.0	1.9	1.1	1.5
	S=0.54		1.1	1.6	1.6	2.1
			0.9	1.3	1.2	1.8
SPRAY		Condensing Humidity 7 days at 165°F	2.3	3.0	2.8	1.3(4)
			2.3	2.4	2.7	0.6
			2.3	2.3	2.5	1.7
		Boiling Deionized Water	2.3	2.1	2.4	2.3
			2.2	2.5	1.8	1.3
			2.1	2.0	2.4	1.4
	M=1.86	None "as prepared"	1.1	2.1	1.7	1.4
	S=0.61		1.2	1.2	0.7	1.5
			1.0	1.7	1.2	1.6
ABRASION		Condensing Humidity 7 days at 165°F	2.9	1.3	2.7	2.6
			2.9	1.6	2.8	2.3
			0.7(3)	3.0	2.5	2.7
		Boiling Deionized Water	2.4	2.2	2.2	2.3
			1.9	2.2	1.9	1.8
			1.8	2.3	2.0	1.7
	M=1.89	None "as prepared"	1.2	1.6	1.1	1.7
	S=0.62		1.3	1.3	0.8	1.8
			1.1	1.8	1.0	1.5
			M=1.94		M=1.87	
			S=0.60		S=0.59	

1. M: M=Mean value of sample;
   S S=Standard deviation of sample
2. Adhesion surface energy density in.-lbs/in^2
3. Blister under paint prior to test.
4. Specimen ruptured in test, results invalid.

Corrosion resistance data for salt fog testing of 2024 and 7075 aluminum alloys treated with MIL-C-5541 Class 1A and Class 3 by the tank, spray and abrasion methods were included in Tables VII and VIII, respectively. The tank method of application produced the best corrosion resistance; all specimens exceeded 7 days without a failure in the salt fog tests. Both spray and abrasion applied specimens failed within 7 days time period. The Class 3 coated 2024 specimens all failed after 21 days exposure to the salt fog, and the 7075 specimens coated with the Class 3 had all passed 28 days salt fog exposure without failure. The Class 1A coated 7075 specimens failed spray application within 7 days, but passed 7 days exposure on 2024 substrates.

Results for accelerated corrosion tests indicated that all specimens tested failed the phosphomolybdic acid spot test. Minor pitting was noted on some specimens with a small number of specimens showing severe pitting. A visual examination of all specimens exposed to the accelerated corrosion was made and recorded in Table IX. Figure 9 depicts accelerated corrosion specimens in various stages of process. The accelerated corrosion test was severe enough to cause some penetration of the chemical films on all specimens; this is illustrated in Figures 10 and 11. All specimens were discolored severely and the conversion coating was hardened (i.e., difficult to strip in a nitric acid solution).

Average coating weight deposited with Class 1A and Class 3 conversion coatings on 7075 and 2024 aluminum alloys was variable. The Class 1A produced the heaviest coatings on 7075 alloy and the Class 3 produced the heaviest coatings on 2024 alloy. Class 1A failed to produce a coating of 40 mg/ft^2 on 2024 alloy except in one case of 43.6 being produced. The Class 3 coating produced more consistent results in the double concentration (i.e. 4.0 oz/gal) than the Class 1A. Specimens of 7075 alloy treated with Class 1A in the double concentration produced variable results with coatings produced that were highly inconsistent in film thickness and that tended to strip in rinsing before drying. Coating weight data is included in Table X.

TABLE VII

RESULTS OF SALT SPRAY TESING OF CHEMICAL CONVERSION 2024T3 ALUMINUM ALLOY

SPECIMEN NUMBER	APPLICATION METHOD	CHEMICAL CONVERSION COATING	TIME TO FAILURE(1)			
			7 days	14 days	21 days	28 days
2A-3 2A-4 2A-5	Tank	MIL-C-5541 Class 3			Failed (2)	
2A-8 2A-9 2A-10	Spray		Failed (3)	Failed (3)		
2A-13 2A-14 2A-15	Abrasion		Failed (4)			
2A-18 2A-19 2A-20	Tank	MIL-C-5541 Class 1A				Failed (5)
2A-23 2A-24 2A-25	Spray			Failed (6)		
2A-28 2A-29 2A-30	Abrasion			Failed (6)		

(1) Specimens were tested in accordance with ASTM-B-117-64, inspected each week removing specimens which had developed corrosion. Specimens successfully withstanding 7 days exposure meet requirements of MIL-C-5541 for the Class II coating; however, testing was continued until failure occurred or terminated after 28 days.

(2) Very slight corrosion.

(3) Very fine pits approximately 240 per in^2.

(4) Moderate stain no pitting.

(5) Only 10-12 very small pits per panel, no stains.

(6) Pitting and stains moderately severe 160 to 200 pits per in^2.

TABLE VIII

RESULTS OF SALT SPRAY TESTING OF CHEMICAL CONVERSION COATED 7075T6 ALUMINUM ALLOY

SPECIMEN NUMBER	APPLICATION METHOD	CHEMICAL CONVERSION COATING MIL-C-5541	TIME TO FAILURE (1)			
			7 days	14 days	21 days	28 days
7A-3 7A-4 7A-5	Tank	Class 3				Passed (2)
7A-8 7A-9 7A-10	Spray					Passed (3)
7A-13 7A-14 7A-15	Abrasion					Passed (4)
7A-18 7A-19 7A-20	Tank	Chromate Class 1A				Passed (4)
7A-23 7A-24 7A-25	Spray		Failed (5)			
7A-28 7A-29 7A-30	Abrasion			Failed (6)		

(1) Specimens were tested in accordance with ASTM-B-117-64, inspected each week removing specimens which had developed corrosion, Specimens successfully withstanding 7 days exposure meet requirements of MIL-C-5541 for the Class II coating; however, testing was continued until failure occurred or terminated after 28 days.

(2) Only slight stains, no pitting.

(3) No corrosion stains.

(4) Only slight stains and 5-10 very minute isolated pits per panel.

(5) Very slight corrosion stains.

(6) Moderate stains, slight pitting beginning to form.

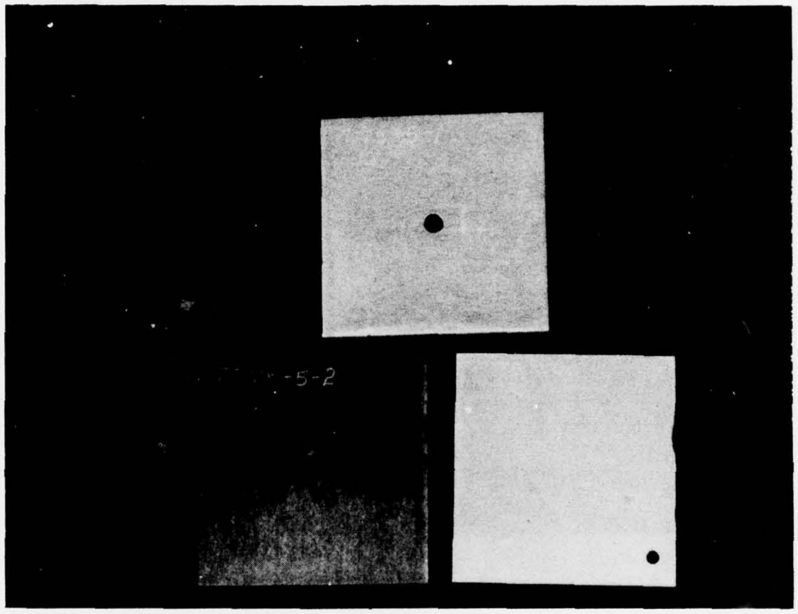


FIGURE 9

ACCELERATED CORROSION TEST SPECIMENS SHOWING VARIOUS STAGES OF CONDITIONING. THE TOP SPECIMEN HAS BEEN CLEANED AND IS READY TO BE ALODIZED, THE SPECIMEN ON THE LOWER LEFT HAS BEEN ALODIZED IN ALODINE 1200S, AND THE SPECIMEN ON THE BOTTOM RIGHT HAS BEEN CORROSION TESTED. NOTE THE LOSS OF COLOR OF THE ALODINE ALTHOUGH NO PITTING WAS OBSERVED.

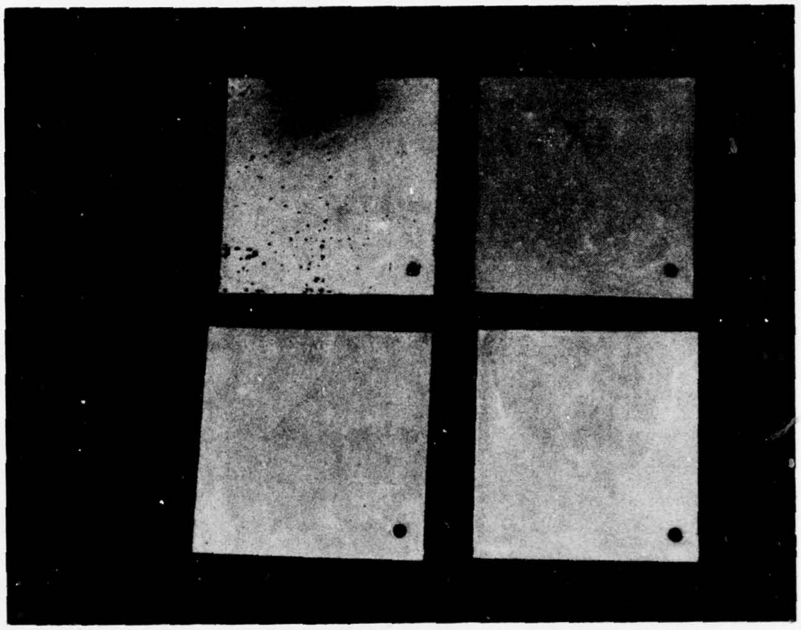


FIGURE 10

ACCELERATED CORROSION SPECIMENS AFTER TEST. 7075 ALLOY TREATED WITH ALODINE 1200S SHOWING, FROM TOP LEFT: SEVERE PITTING, MODERATE PITTING, SLIGHT PITTING, AND NO PITTING.

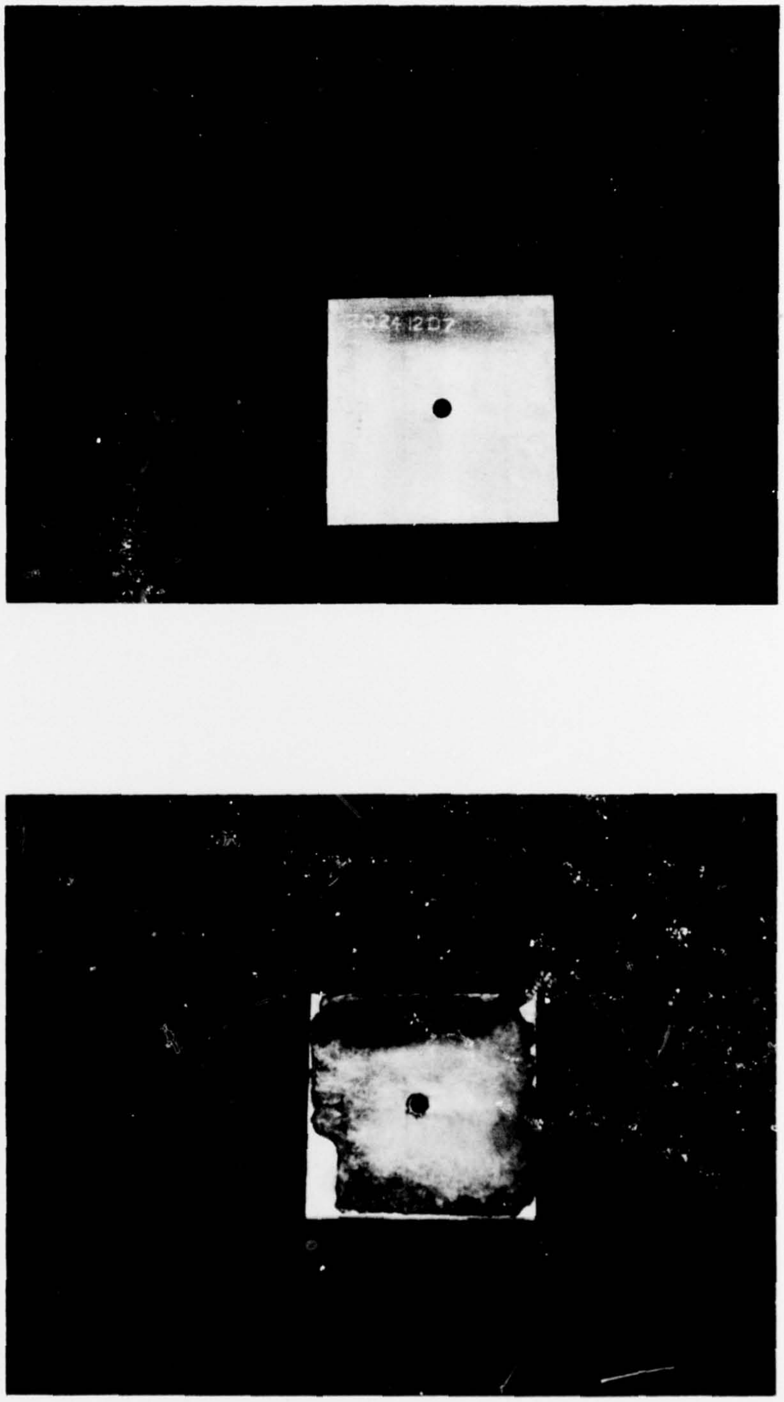


FIGURE 11

TOP: SPECIMEN AFTER ACCELERATED CORROSION TEST WITH VERY LITTLE VISIBLE PITTING, BUT FAILING SPOT TEST.
BOTTOM: SAME SPECIMEN TESTED WITH PHOSPHOMOLYBDIC ACID SPOT TEST SHOWING THE POSITIVE TEST FOR CORROSION

TABLE IX

ACCELERATED CORROSION TEST RESULTS - VISUAL INSPECTIONS (1)

APPLICATION TIME MINUTES	7075 SUBSTRATE-SOLUTION CONC. 2.0 OZ/GAL.				2024 SUBSTRATE-SOLUTION CONC. 2.0 OZ/GAL.			
	MIL-C-5541 Class 1A		MIL-C-5541 Class 3		MIL-C-5541 Class 1A		MIL-C-5541 Class 3	
1	NO PITTING	NO PITTING	NO PITTING	NO PITTING			NO PITTING	NO PITTING
3	FEW SMALL PITS	NO PITTING	NO PITTING	NO PITTING	--		NO PITTING	MANY VERY SMALL PITS
5	NO PITTING	NO PITTING	NO PITTING	NO PITTING			FEW SMALL PITS	VERY FEW SMALL PITS
10	MANY DEEP PITS	NO PITTING	NO PITTING	NO PITTING			MANY SMALL PITS	NO PITTING
	7075 SUBSTRATE-SOLUTION CONC. 4.0 OZ/GAL.				2024 SUBSTRATE-SOLUTION CONC. 4.0 OZ/GAL.			
1	NO PITTING	NO PITTING	NO PITTING	NO PITTING	MANY SMALL PITS	3 OR 4 VERY SMALL PITS	FEW SMALL PITS	3 OR 4 VERY SMALL PITS
3	PITTING	MANY SHALLOW PITS	NO PITTING	NO PITTING	NO PITTING	NO PITTING	NO PITTING	NO PITTING
5	NO PITTING	FEW SMALL PITS	NO PITTING	NO PITTING	NO PITTING	NO PITTING	3 OR 4 SMALL PITS	NO PITTING
10	NO PITTING	MANY SMALL PITS	NO PITTING	3 OR 4 VERY SMALL PITS	NO PITTING	NO PITTING	NO PITTING	MANY SHALLOW PITS

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(1) All specimens showed to have broken conversion coatings by the phosphomolybdic acid spot test after corrosion testing.

TABLE X

AVERAGE COATING WEIGHT DETERMINATIONS FOR CHEMICAL CONVERSION COATINGS (1)

APPLICATION TIME MINUTES	7075 SUBSTRATE-SOLUTION CONC. 2.0 OZ/GAL.		2024 SUBSTRATE-SOLUTION CONC. 2.0 OZ/GAL.	
	MIL-C-5541 Class 1A	MIL-C-5541 Class 3	MIL-C-5541 Class 1A	MIL-C-5541 Class 3
1	30.2	24.0	39.8	19.2
3	36.8	24.0	38.0	15.2
5	32.9	16.8	43.6	19.6
10	40.9	26.0	30.5	25.2
	7075 SUBSTRATE-SOLUTION CONC. 4.0 OZ/GAL.		2024 SUBSTRATE-SOLUTION CONC. 4.0 OZ/GAL.	
1	38.8	25.6	29.9	46.4
3	35.3	21.6	30.2	42.4
5	49.7	26.8	34.7	35.2
10	48.7	44.3	30.2	55.8

(1) Coating weight determinations performed in tank application with solution concentrations noted at 100 ± 10°F. Substrates were alkaline cleaned, acid rinsed per CVA-9-64 prior to conversion coating applications. Coating weights are stated in mg/ft^2.

5.0 DISCUSSION OF RESULTS

5.1 HOW MUCH ADHESION DO WE NEED

The development and utilization of the fracture mechanics approach to absolute adhesion testing has been utilized in three programs. The adhesion surface energy densities have been measured for coatings and sealants over a wide variety of substrates. The interface which has been tested frequently in these studies and for which we have widespread service experience is the MIL-P-23377 epoxy primer to anodized aluminum. In these tests it was seen that the tensile strength of an .030 inch thickness film would just exceed the adhesion of a 1.0 inch pound per square inch of area. For values less than 1.0 this adhesion could be overcome and for values above 1.0 it could not be overcome. From the examination of these specimens, it appears that this is the lowest ($\gamma\alpha$) adhesion surface energy density which provides the probability of good service performance for the exterior of aircraft.

5.2 ADHESION OF THE NAVY PAINT SYSTEM TO NON-METALLIC COMPOSITES

Test results indicated that the Navy Paint System will adhere well to all of the non-metallic composite materials evaluated. Adequate adhesion (1.0 inch pound per square inch) was obtained for all precoating surface preparations utilized; however, it should be noted that the values obtained utilizing a solvent wipe plus a peel ply on the outer surfaces of the composites yielded average adhesion surface energy densities of 2.03 in.-lbs./in.^{2}. Values obtained for specimens receiving only a wipe before coating yield average surface energy densities of 2.01 in.-lbs./in.^{2}. These results are very close to the same value and the degree of precision of the test method will not differentiate between these. However, considering the values equal, it seems the expensive peel ply would not be warranted on the exterior side (i.e., side to receive the exterior paint system) of the part to improve the adhesion of the paint system.

5.3 CONTINUATION OF ANODIZING OPTIMIZATION STUDY

Adhesion of the Navy Paint System to anodized aluminum alloys was found to be higher than that to the non-metallic substrates. In order to obtain a larger sample by which to evaluate a particular anodize, one should consider all specimens tested. In so doing the "as prepared" specimens, which yielded low adhesion results as compared to the exposed specimens, lowered the average adhesion surface energy density obtained for each type anodize evaluated. All systems yielded values which were within relatively narrow limits, except for the Type B, 22 volt Chromic acid anodized specimens. Adhesion values for the Type B Chromic acid anodized specimens were found to be approximately 25% below those of the other anodized specimens.

In consideration of the "as prepared" specimens yielding lower adhesion surface energy densities than those specimens being exposed to test conditions, one must realize that the "as prepared" specimens did not have the benefit of an elevated temperature post cure. The exposed specimens were all subjected to elevated temperature which served to post cure the coating, thus most likely increased the degree of cure of the coatings and improved the mechanical properties of the coatings. It should be noted that the failure of the "as prepared" specimens failed cohesively within the primer as illustrated in Figure 8. Subjection of all specimens to an elevated post cure before beginning tests would have probably reduced the data scatter within the "as prepared" specimens and produced a higher adhesion value for the "as prepared" specimens rather than for the exposed.

This is consistent with actual service experience of low adhesion found by 24 hour water soaks prior to wet tape testing causing blistering. This has been shown to be a result of osmotic pressure of the soluble chromates through the semipermeable paint film membrane generating sufficient pressure to overcome the cohesion of the film at that stage of curing. Aircraft exhibiting this condition have almost always improved with additional curing time and generally to the extent of no blistering after several additional days or weeks, depending on ambient conditions. In the case of these specimens in which an abnormal thickness of primer is applied, solvent retention and thus lower than normal cohesive strength is a high probability for long time periods. This fact must be considered for the design of future tests utilizing solvent applied finish systems in blister adhesion testing.

5.4 ADHESION COMPARISON OF TWO CHEMICAL CONVERSION COATINGS TO FOUR ALUMINUM ALLOYS (REF. TABLE V)

Test results were variable due to differences in the "exposed" and "as prepared" specimens; however, compiling all data indicates a positive trend. The specimens prepared with MIL-C-5541 Class 1A produced average adhesion values slightly higher than those produced with the MIL-C-5541 Class 3. It should be noted that the average standard deviation for adhesion values for all alloys processed is equal to that of specimens prepared with MIL-C-5541 Class 3, indicating the same data scatter or variation in the specimens produced with both materials on all four alloys tested. If one considers only the 7075-Class 1A processed specimens, a standard deviation of 0.80 is considerably higher than the 0.59 for the total average value. This probably is due to the fact that the Class 1A material reacts very rapidly on the 7075 alloy, and excessive buildup is the result if specimens are alodized for longer than optimum time. Non-adhering coatings can also result due to cleaning variations prior to alodizing. Some aluminum alloys tend to smut heavily when cleaned in alkaline cleaners, and incomplete smut removal can affect alodize buildup and properties of the conversion coating. Coating data obtained with the Class 3 on the 7075 is a good case in point. This combination produced adhesion results with slightly lower adhesion values considerably smaller than those for the 7075-Class 1A combination indicating less data scatter in these Class 3 processed specimens; it seems this should be given consideration when selecting a specific conversion coating for more reproducible results.

The tank applied conversion coatings produced adhesion values that were higher than those produced with the spray or the abrasion method, and the tank applied coatings were found to produce a slightly smaller data spread than for those data produced for the spray application method. The abrasion application method produced adhesion values higher than those for the spray application method, and values obtained for the abrasion method showed the least amount data scatter for any application method evaluated. The lower adhesion values produced by the spray applied coatings should be considered when selection of application methods for conversion coatings as this method has some disadvantages. Failure of the spray application to completely contact the surface to be coated for sufficient time to produce a satisfactory coating is one. Maintaining solution temperature is another problem in spray applications. Requirements to respray coatings repeatedly over an area to be coated, excessive loss of solution, and attend problems of contamination of solutions in a recovery system are some negative aspects of spray application of chemical conversion coatings. Failure of spray applied solutions to contact surfaces uniformly is the most probable reason for the lower adhesion values obtained with the spray application.

5.5 ACCELERATED CORROSION EVALUATION OF CHEMICAL CONVERSION COATINGS

Average adhesion values obtained with different application methods agree well with those obtained using different alloy substrates. The tank application method yielded the highest adhesion values, followed by the abrasion method and poorest adhesion was obtained with the spray method of application. Average adhesion values produced by each of the two conversion coatings changed in comparison to those obtained with other alloys. It should be expected that the adhesion values obtained with only two alloys would be somewhat different results than those obtained with four alloys. Also, the four alloy test and the two alloy test had had only one common alloy -7075. Considering only those values for the 7075 alloy, the Class 3 still produced higher values than those produced for the Class 1A in this test. Values for the two alloy test were approximately 4% higher for the 7075-Class 1A combination and 29% higher for the 7075-Class 3 combination. This is a large difference and it seems, should be considered somewhat variable.

5.6 EFFECTS OF ACCELERATED CORROSION TESTS

The accelerated corrosion results indicated that this method of corrosion testing offers considerable advantage in rapid segregation of alodized parts with respect to corrosion resistance. Pitting was shown to occur after 30 minutes boiling of some specimens. This is a tremendous time saving advantage over the standard 168 hour salt fog test. Variation of the length of time of the boiling test can also be utilized for specific requirements. An exact description of the degree of corrosion is a difficult thing to accomplish. Attempts to quantify corrosion as a function of weight loss of the conversion coating of specimens exhibiting no pitting failed because the elevated temperature of the boiling corrosion test fixed the conversion coating, making uniform stripping of the coating impossible. Stripping of the fixed coating resulted in only a partial strip of the coating and etching of the substrate occurred in those areas stripped, while the tenacious area of the coating resisted stripping. This prevented development of a more quantitative method of describing the degree of corrosion produced in the accelerated corrosion test. The phosphomolybdic acid spot test produced good results and readily identifies those areas of the coating that are broken. This test, however, is limited to those alloys containing copper.

Results of the coating weight study for conversion coatings on aluminum alloy substrates indicated a definite advantage in selection of a specific conversion treatment for a given alloy. Consulting Table X data, one can readily see that the Class 1A produces heavier coating buildup on 7075 aluminum alloys and the Class 3 produces a heavier coating on the 2024 alloy. Processing time is not as great a factor in these cases in which the coatings produce the heavy films readily, as in the case of the Class 1A material on the 7075 alloy, additional processing time increases coating build somewhat, but in most cases, coating build was not greatly increased appreciably after 5 minutes processing time. This was more evident with the Class 1A than with the Class 3 materials. Higher solution concentration and longer processing times produced increasing coating buildup with the Class 3 material on the 2024 alloy, but was less consistent on the 7075 alloy. Heavy smut buildup during alkaline cleaning of the 7075 alloy may be responsible for these variations; however, repeat tests with longer desmut rinse times failed to change these results.

Potentially, the most important finding of this program is the relationship of coating weights shown in Table X for various concentrations and alloys and the results of the oxygen enriched accelerated corrosion test. It is generally believed that higher chromate coating weights give better corrosion protection. But under the conditions of this accelerated test, coating weights below 35 milligrams per square foot did not pit. Above this coating weight mixed results but a heavy preponderance of pitting was observed. This result, because of the widespread use of MIL-C-5541 Class 1A chemical film on 7000 series aluminum, and the initiation of stress corrosion cracking and fatigue failures from pits, is cause for concern.

TABLE XI

CORRELATION OF OXYGENATED BOILING 5% SALT TEST WITH PITTING OF VARIOUS FILM WEIGHTS OF MIL-C-5541 TYPE 1A AND 3 CONVERSION COATINGS ON 7075-T6 ALUMINUM

	Type 3				Type 1A			
	2 ounce/gallon		4 ounce/gallon		2 ounce/gallon		4 ounce/gallon	
Treatment Time	Pitting*	Film Weight**	Pitting	Film Weight	Pitting	Film Weight	Pitting	Film Weight
1	0/2	24.0	0/2	25.6	0/2	30.2	0/2	38.8
3	0/2	24.0	0/2	21.6	1/2	36.8	2/2	35.3
5	0/2	16.8	0/2	26.8	0/2	32.9	1/2	49.7
10	0/2	26.0	1/2	44.3	1/2	40.9	1/2	48.7

* 0/2 No pitting on either of 2 specimens

** Film weight in milligrams/square foot

6.0 CONCLUSIONS

- Adhesion of the Navy Paint System to non-metallic composite materials is more than adequate. Adhesion surface energy densities were found to be approximately 2.0 in.-lbs./in.^{2}; values of 1.0 in.-lb./in.^{2} are considered minimum for good results.

- Hand sanding, grit blasting or mechanical abrasion of the non-metallic composite surface to be coated is not necessary in order to obtain good adhesion of organic coatings to these substrates.

- A peel ply on the surface to be coated with an organic coating is not necessary in order to obtain good adhesion of the coating to the non-metallic substrates.

- Only a solvent wipe before coating produces good adhesion of the Navy Paint System to these non-metallic composite substrates.

- Standard degreaser solvent MIL-T-81533 (1,1,1 trichloroethane) was found to be effective in removing surface soils from non-metallic composites prior to coating.

- Adhesion surface energy density of the Navy Paint System to anodized aluminum alloys was higher than that for the same paint system to the non-metallic composite substrates, except for the Type B, low voltage, Chromic acid anodized substrates.

- The Type B, low voltage, Chromic acid anodized substrates yielded adhesion values approximately 20% lower than those for other types of anodize.

- Scatter or data spread in adhesion values obtained for the "as prepared" control specimens and exposed specimens resulted due to an incomplete cure of the coatings on the control specimens.

- Flaws in coatings such as bubbles, blisters and voids produced some poor results in a small number of individual data points. This serves to emphasize the importance of proper specimen fabrication, allowing all solvents time in which to diffuse out of the partially cured coating film before subsequent coats are applied.

- The heavy coating film of 30 to 35 mils dry film thickness required in making the blister test specimens is one disadvantage of the Blister Test Method.

- Lack of a post cure for all specimens before exposure and test by the Blister Test Method increased the data scatter in these investigations.

- The Type A, 40 volt, Chromic acid anodized aluminum alloy substrates produced the highest average adhesion values of any anodized substrates evaluated. These average values were approximately 3.3% higher than those obtained from the best sulfuric acid anodize.

- o Tank applied conversion coatings produced the highest and most reproducible adhesion results. Spray applied conversion coatings produced slightly lower adhesion values than those applied with abrasion.

- o Adhesive surface energy density of the Navy Paint System to treated aluminum alloy substrates varied according to the conversion coating and alloy substrate combination.

- o Improper heat treatment of the 7075 aluminum alloy failed to produce a noticeable difference in salt fog corrosion resistance compared to that of the standard 7075-T6 condition.

- o The boiling 5% salt solution accelerated corrosion test method produced more severe corrosion than the standard 7 day salt fog tests. Standardization of variables of heat up rate, cool down rate, and oxygen input are needed to utilize this test as a quality control procedure.

- o The phosphomolybdic acid spot test is a reliable indicator of broken conversion coating films on copper bearing substrates.

- o MIL-C-5541 Class 3 conversion coating materials produced the most desirable results with 2024 aluminum alloy and MIL-C-5541 Class 1A produced the best results with 7075 alloy substrates.

- o MIL-C-5541 Class 1A conversion coatings produced slightly higher values for adhesion surface energy density by the Blister Test Method, in most cases, than did the MIL-C-5541 Class 3 materials.

- o MIL-C-5541 Class 1A conversion coating with weights of less than 35 milligram per square foot did not result in pitting in oxygen enriched boiling five percent salt solution corrosion test.

- o Data spread was generally less for adhesion values on MIL-C-5541 Class 3 prepared specimens as compared to specimens prepared with MIL-C-5541 Class 1A.

- o Exposure of aluminum alloys coated with chemical conversion coatings to the accelerated corrosion test "fixes" the coating to the degree that makes uniform stripping impossible without some etching of the substrate. This prevents a quantitative determination of the coating loss as a result of the accelerated exposure.

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