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THE SIGNIFICANCE OF PROCESSING VARIABLES ON THE ADHESION OF SEALANTS AND ORGANIC COATINGS TO METALLIC AIRCRAFT SURFACES

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes the results obtained in a study program to improve adhesion between aircraft type metals and organic coatings currently used. The blister test method was used which utilizes fluid pressure under the coating through a hole in the substrate to cause adhesive failure. Specimens were evaluated in the "as prepared" condition as well as after being subjected to hostile environments. Variations of standard surface

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treatments, application techniques and adhesion promoting agents were evaluated. Surface treatments consisted of sulfuric acid anodize on aluminum alloy, 7075, chromic acid anodize on aluminum alloy, 2024, abrasion chemical conversion treatments on titanium alloy, Ti6Al4V, phosphate chemical conversion treatments on steel, 4340, and chromate and phosphate chemical conversion treatments on cadmium plated steel, 4340. A total of seven organic coatings and five silanes were utilized in the evaluations.

FOREWORD

The program designed to study the effects of variations in surface preparation, application techniques and adhesion promoting additives on the adhesion of organic coatings to aircraft type metals was initiated in February 1973 under Naval Air Systems Command contract N00019-73-C-0286. The project has been administered by Tom Johnston of the Material Acquisition Group of the Engineering Division of Naval Air Systems Command. The object of this program was to study the effect of variations in surface preparation, application techniques and adhesion promoting additives by measuring the adhesion surface energy density of combinations by the "Blister Test Method". By this evaluation the optimum surface preparation and application may be selected for each substrate/coating system combination.

The program, as most present-day research programs, has been dependent on the cooperative efforts of individuals from a number of areas. The cooperation and guidance from Mr. Tom Johnston has greatly assisted the program. Technical personnel within and outside of Vought Systems Division have been vital to the accomplishment of this program. Dr. M. L. Williams, Professor of Engineering, University of Pittsburg was very helpful in furnishing copies of previous work using the blister test method and in giving personnel encouragement. Dr. W. B. Jones, Rocketdyne, McGregor, Texas, is acknowledged for his time, encouragement and direction in the modification of the Blister Test Apparatus to determine meaningful data from nontranslucent materials. Although the number at Vought Systems Division precludes individual recognition, their efforts as well as those mentioned above are recognized and appreciated.

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1.0 INTRODUCTION AND SUMMARY

This report describes a study and test program in which various combinations of surface preparations, application techniques and adhesion promoting additives were evaluated. The "Blister Test Method" was utilized in the evaluation so that the adhesive surface energy density could be determined between the substrate and the coating. The method utilizes fluid pressure under the coating through a hole in the substrate to cause adhesive failure. The coating must be thick enough to prevent a cohesive failure and allow a blister to form around the built in flaw in the system.

The adhesion characteristics of currently used organic coatings were evaluated on aircraft type metals. The metals selected were titanium alloy (Ti6Al4V), aluminum alloy (7075), aluminum alloy 2024, steel (4340) and cadmium plated steel (4340). The coating materials used in the evaluation were:

Epoxy polyamide primer	Material Code 1
Polyurethane Enamel	Material Code 2
Acrylic Nitrocellulose Lacquer	Material Code 3
Sealant Polysulfide (Chromate cure)	Material Code 4
Sealant Polysulfide (Manganese cure)	Material Code 5
Neoprene Rain Erosion Coating	Material Code 6
Polyurethane Elastomer	Material Code 7

The object of the program was to improve the adhesion characteristics of the various coating/substrate combinations and to determine what processing variables detrimentally affected the adhesion.

To accomplish this goal variations of the presently used process were selected to evaluate the processing parameters. These variations along with application techniques and adhesion promoting additives would make it possible to select the best surface preparation system for each coating/substrate combination.

The first problem was to select a suitable specimen preparation technique. Various port sealing techniques were investigated which could be utilized with the coatings and obtain sufficient strength to prevent cohesive failure. The next step was to prepare sufficient specimen to evaluate the processing parameters and since the end use would affect aircraft it was necessary to subject some specimens to hostile environments. Specimens were prepared in sufficient numbers to evaluate all parameters in the "as prepared" condition and selected parameters which had been subjected to 30 day intermittent salt water exposure. On the titanium alloy some specimens were subjected to a 24 hour soak in distilled water and some to 24 hours at 250°F.

A number of approaches were explored and a study made to select the type of adhesion promoting agents to evaluate. The silanes appeared to offer promise and vendors were contacted for information and data on adhesion promoting additives or couplings agents that could be applied to the surface of the processed metals prior to application of the epoxy polyamide primer and/or mixed with the epoxy polyamide primer prior to application. From this study five silanes were selected for evaluation.

Several of the evaluation parameters selected for evaluation produced equally good adhesion characteristics so that the data precluded selection of an optimum processing variation. These evaluations only proved that the substrate processing/coating application adhered equally well within the processing variations selected for evaluation.

In other instances the results obtained were very pronounced. The processing of titanium to improve adherence of coating materials showed that the fluosilicic acid/aluminum oxide slurry abrasion treatment was far superior to the chromate solution/abrasive nylon pad abrasion treatment.

The evaluation of adhesion promoting agents or coupling agents selected failed to produce significant increase in adhesion. In fact, in some instances the materials significantly lowered the adhesion.

The processing variation of sulfuric acid anodizing on the 7075 aluminum alloy showed equally good adhesion with the epoxy polyamide primer. No parameters of the anodizing process were identified which would explain the random failure of coatings or adhesives to adhere to anodized surfaces in production applications.

The adhesion was found to be much higher on the specimens which were coated with the polyurethane elastomer over the epoxy polyamide primer than with the epoxy polyamide primer only. The reverse occurred when the polyurethane enamel was applied over the epoxy polyamide primer as the adhesion was lowered. This would indicate that the material applied over the primer affects the adhesion characteristics of the substrate/coating system, either by a reaction with the primer or a solvency effect.

The chromic acid anodize variations on the aluminum alloy (2024) showed equally good adhesion with the epoxy polyamide primer. Similar results were obtained when the polyurethane elastomer was applied over the epoxy polyamide primer as was found with the sulfuric acid anodized specimens and an increase in adhesion was observed. Unlike the results obtained on the sulfuric acid anodized specimens the chromic acid anodized specimens did not show a reduction adhesion when polyurethane enamel was applied over epoxy polyamide enamel.

The steel and cadmium plated steel specimens when evaluated verified that a brush chemical conversion treatment was equally effective as an immersion chemical conversion treatment.

2.0 TEST PROCEDURES

2.1 BLISTER TEST APPARATUS

2.1.1 Introduction - The most promising method for obtaining values for the adhesive strength in absolute units appeared to be the blister test. This is a relatively simple test in which the adhesion is measured by applying a fluid under pressure through a hole in the substrate under the coating. From the pressure required to lift the coating from the substrate the adhesive strength can be determined.

A similarity between certain problems of adhesion and fracture was discussed by Williams (1,2). 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 (mathematically) 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 (3) 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:

$$\sigma_{cr} = 2E\,\gamma_c/\pi^{a} \qquad (1)$$

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 infinite 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:

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

Williams developed the threatment for the blister test, first proposed by Dannenberg (4) but without the fracture mechanic treatment, for determination of the strength of an adhesive (i.e. γ_a).

The samples are easily constructured. 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:

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

where P_c = pressure necessary to initiate adhesive fracture
E = Young's Modulus
h = Plate thickness (coating)
ν = Poisson's Ratio
a = Radius of unbond
γ_a = Adhesive surface energy density

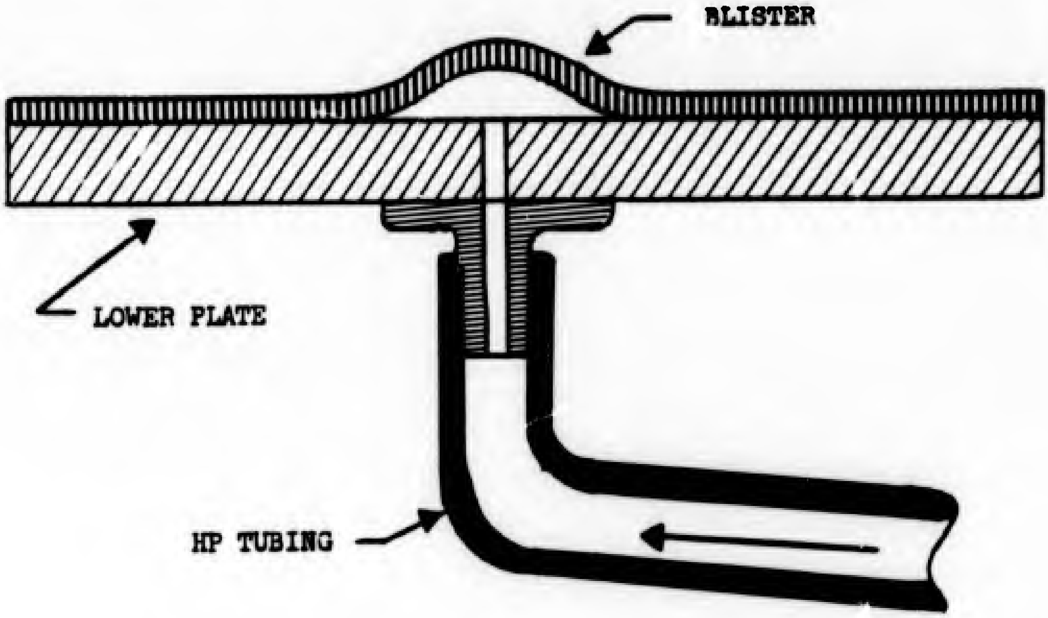


Figure 1. Schematic of Adhesive Testing Apparatus

2.1.2 Coating Materials Physical Properties - An attempt was made to use ultrasonic measurements to determine Poisson's Ratio and Young's Modulus. These data were to be obtained by means of ultrasonic wave velocity measurements and calculated based upon the following equations:

$$\sigma = \frac{1-2\left(\frac{V_T}{V_L}\right)^2}{2-2\left(\frac{V_T}{V_L}\right)^2} \qquad (3)$$

$$Y = \frac{\rho V_L^2(1-\sigma)(1-2\sigma}{1-\sigma} \qquad (4)$$

where σ = Poisson's Ratio

Y = Young's Modulus

P = Density

V_T = Transverse wave velocity

V_L = Longitudinal wave velocity

The density measurement was straight forward and the longitudinal wave velocity measurement was accomplished without too much difficulty using a specially-designed transducer. The transducer had an extremely short pulse length and was placed perpendicular to the coating surface sending a longitudinal wave through the coating and substrate and receive the reflective waves. By amplifying the resultant signals, the two signals indicating reflection from the coating surface and the coating/substrate interface were displaced along the time base of an oscilloscope. This gave a measurement of the time for the longitudinal wave to pass through the coating twice. The coating thickness was measured and the wave velocity (V_L) calculated from the equation.

$$V_L = \frac{d}{t} \qquad (5)$$

where d = thickness of coating

t = time for the longitudinal wave to pass through the coating.

Two techniques were tried for the measurement of transverse wave velocity. A plastic wedge with attached transducer and aluminum block were made such that mode conversion from longitudinal to transverse would occur. When the transducer was pulsed at 1000 volts, a strong signal was received from the bottom of the block, indicating that a transverse signal was being generated and striking the bottom of the block perpendicularly. This assembly was placed against the coated side of a specimen and pressure was applied to transfer the transverse wave across the aluminum/coating interface. This approach did not prove successful so one half of the aluminum block was coated. The plastic wedge with attached transducer was scanned back and forth while the oscilloscope trace was observed for a shift corresponding to the step from a coated to an uncoated surface. The shift was not observed. Other than an apparent inability to transfer a transverse wave across the aluminum/coating interface, the primary fault with this approach was the inability to pulse the state-of-the-art transducer with a sufficiently high voltage to get a strong transverse signal while maintaining a short pulse length which would be detectable. It was concluded that a signicantly more sophisticated approach was required.

Due to the additional work and expense for the development of a new approach, the original equations were re-examined for an alternate solution. It was found that the expected accuracy of wave velocity measurement was such that a large inaccuracy could be expected in the calculated values for Poisson's Ratio and Young's Modulus for these materials. This is true for all materials, such as these coatings, which have a low modulus and high Poisson's Ratio but not true for metals which have a high modulus and low Poisson's ratio.

2.1.3 Design and Assembly of Test Equipment - The "Blister Test" apparatus was assembled as diagrammed in Figure 2 using a Statham Instrument Inc. strain gauge, model UC3 in a body shell adapter, model U6P4-B in which various pressure diaphrams can be installed. A Statham universal readout, model SC 1001 was attached to the pressure transducer and then relayed to a Hewlett Packard X-Y recorder, model 7004B. The system was pressurized with bottled nitrogen through a tank pressure guage regulator, cut off valve and micro metering valve model 1B22RS4. System pressure was also monitored, and calibrated with an Ashcroft, 0 to 60 psig, test guage which had previously been calibrated. A solenoid operated valve and specimen holder, Figure 3, completed the assembly. The specimen holder was designed for ease of specimen installation by using an o'ring seal with clamps to hold the specimen in place.

During checkout of the apparatus it was found that measuring the blister diameter was very difficult with the non-transparent material being evaluated, epoxy-polyamide primer. To determine the adhesive surface energy density the critical pressure for failure, the flaw size, the system geometry and the material properties are required.

A review of the literature found that Jones, H. B. and Williams (reference 5) had done additional work which showed that the equation (2) could be written in terms of the center plate deflection as

$$P_c W_o = 2\gamma_a \tag{6}$$

where W_o = centerplate deflection, see Figure 4, 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-plan stretching or membrane stresses predominate and criticality can be defined, (reference 6).

$$P_c w_c = 2.4\gamma_a \tag{7}$$

$$\text{or } P_c = 1.532\left(\frac{h}{a^{3/4}}\right)\sqrt[4]{\frac{E\gamma_a}{a}} \tag{8}$$

A telecon with Dr. W. B. Jones verified the application of this technique and the increased accuracy which could be obtained by this method. A one day trip was made to visit Dr. Jones at Rocketdyne, McGregor, Texas, to discuss the "Blister Test" method they were using and examine their apparatus.

FIGURE 2

DIAGRAM OF APPARATUS TO MEASURE ADHESIVE SURFACE ENERGY DENSITY

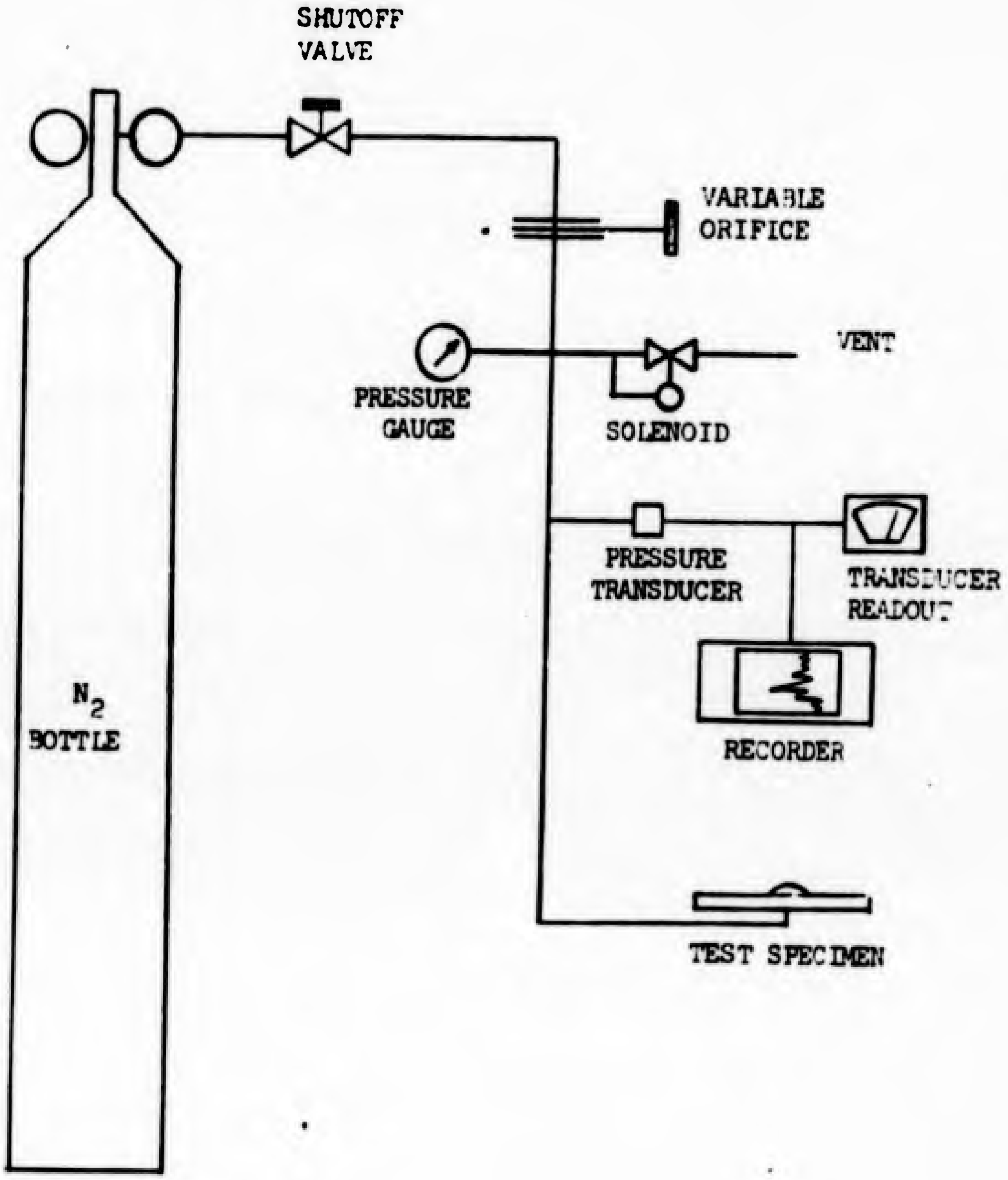
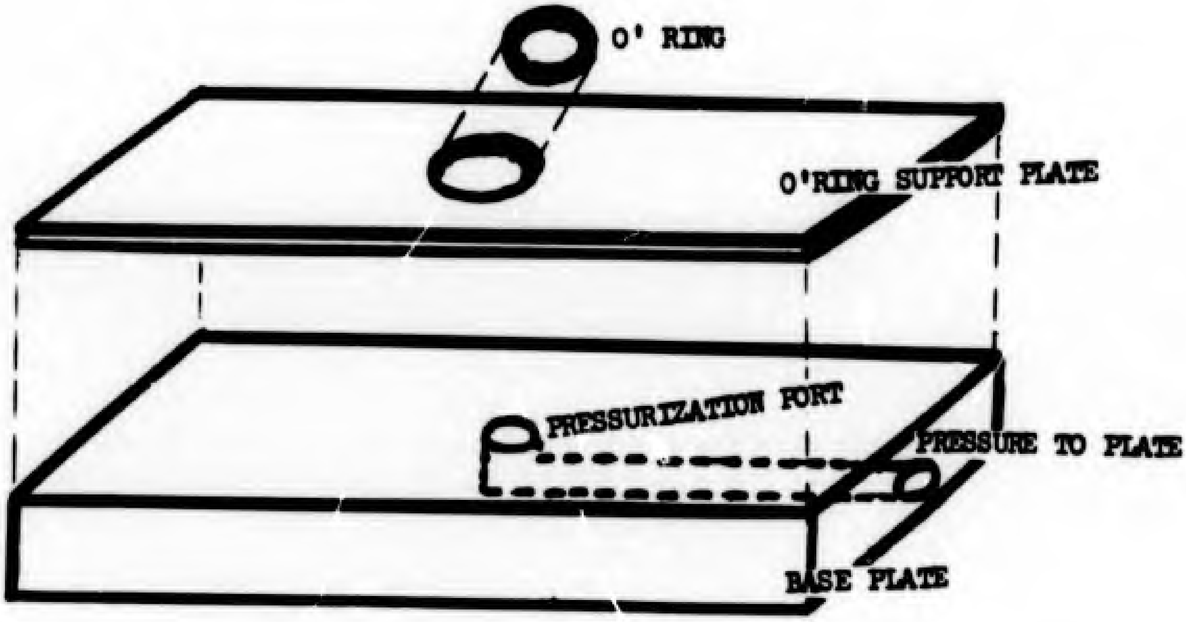


FIGURE 3

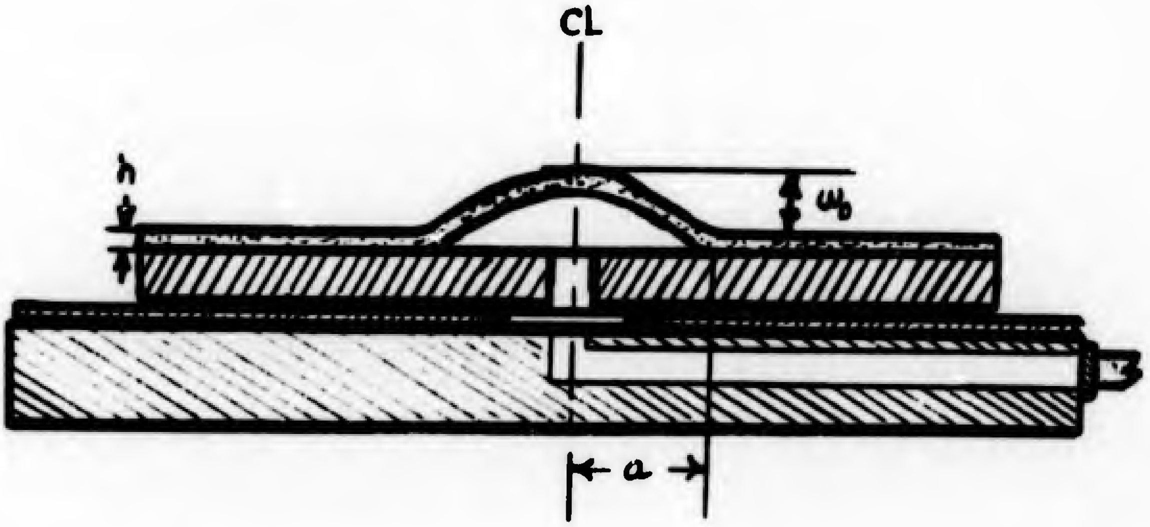
SCHEMATIC OF SPECIMEN HOLDER



As a result of the visit the system was modified to measure the blister height using a Schaevitz Liner Displacement Transducer (LVDT). The output from the LVDT was connected to the X-Y recorder so that a direct plot of critical pressure (P_c) versus W_o was obtained. From this plot the adhesive surface energy density (γ_a) may then be calculated from Equation 7. With this arrangement several plots may be obtained from each specimen and results averaged as shown on a typical plot in Figure 5.

FIGURE 4

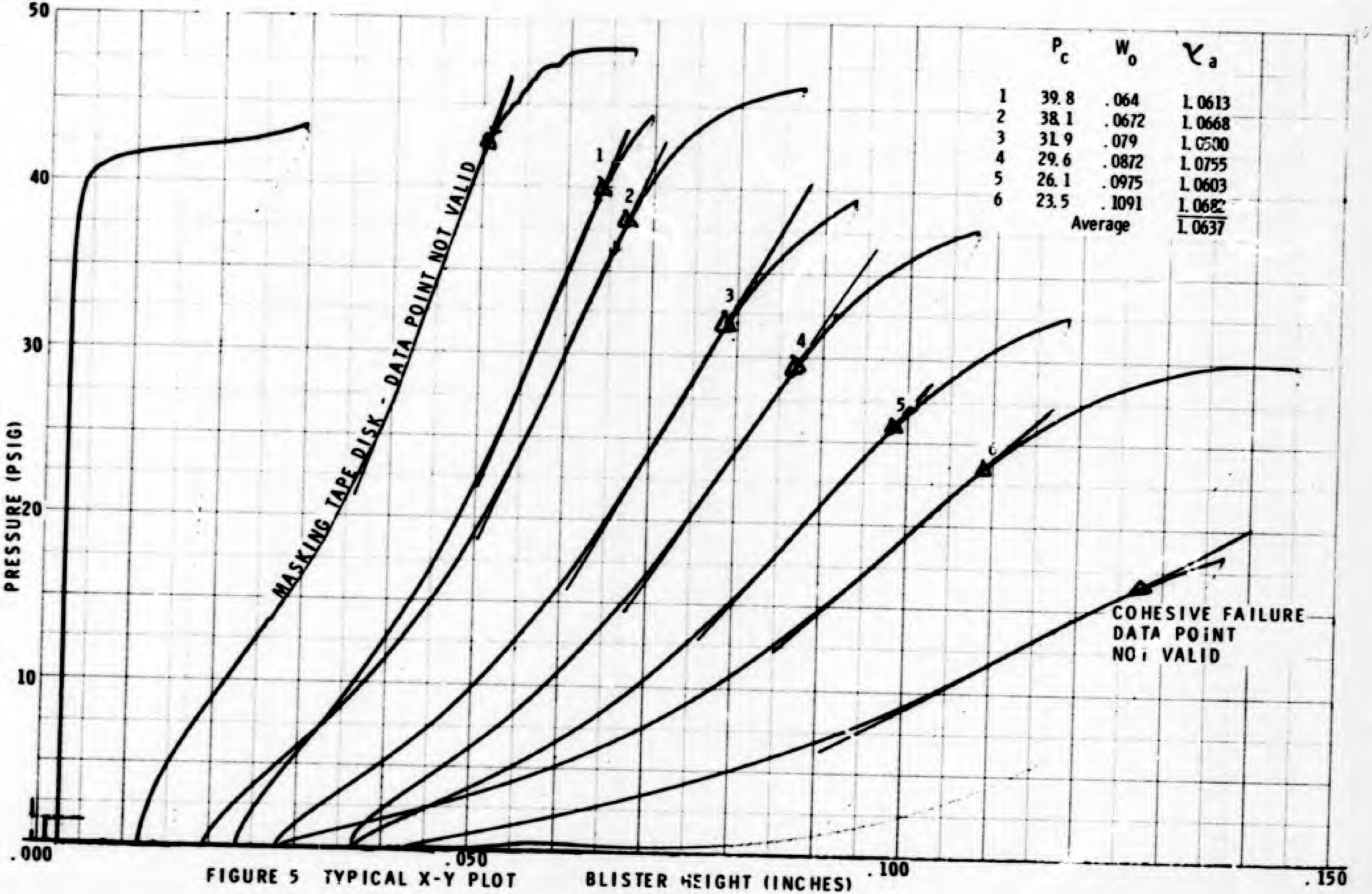
SCHEMATIC OF ADHESION TEST SPECIMEN



In view of the change in test method no additional attempts were made to obtain Poissons Ratio or Young's Modulus as they were not required in the calculation.

2.1.4 Test Specimen Preparation - Many methods were evaluated of pressurization port sealing. These included wax filling, filling with low temperature melting alloy, teflon plugs, and covering the port on the side to be coated with aluminum foil, paper and polyethylene film. In all cases it was possible to obtain satisfactory coverage but in some instances the results during subsequent testing were not satisfactory.

1. Teflon plugs were evaluated both coated with silicone grease and uncoated. In each case the coating covered nicely but the fit between the hole in the specimen and the plug left a filet



	P_c	W_o	γ_a
1	39.8	.064	1.0613
2	38.1	.0672	1.0668
3	31.9	.079	1.0500
4	29.6	.0872	1.0755
5	26.1	.0975	1.0603
6	23.5	.1091	1.0682
	Average		1.0637

FIGURE 5 TYPICAL X-Y PLOT

which was difficult to break loose. Out of one series of ten specimens only three were satisfactory for adhesion testing.

2. Wax filling of the pressurization was very simple but contaminated the surface of the specimen to be coated. Several attempts to mask surface was made before this method was eliminated as it had been used successfully by Williams previously.

3. A low melt point alloy (158°F) was used to fill the pressurization ports and did a fairly good job when the specimen was preheated to prevent rapid cooling of the fluidized alloy. This method produced good specimens but was eliminated when it was found that the specimens had to be heated to about 170°F to 180°F to obtain satisfactory removal of the alloy.

4. Covering of ports on the surface to be coated proved to be the most successful, as well as the simplest method of sample preparation. This method had been used by Dannenberg, reference 4, which consisted of gluing a small aluminum foil patch over the pressurization port with weak rubber cement. This method as well as paper disks, and polyethylene patches were tried. All seemed to work well and additional specimens were evaluated comparing disks to square patches with no apparent differences. It was felt that a contamination problem might be encountered as the disks would have to be applied after final surface treatment. Pressure sensitive paint masking disks currently used at LTV-VSD; (Material Code 10) were tried and found to be very satisfactory without the possible contamination problems.

A coating thickness of approximately .040 inches is required to prevent cohesive failure of the coatings and to prevent excessive stretching during testing of the coating which results in non-uniform or increasing Ya on the same specimen instead of agreement as shown in Figure 5 . This coating is obtained by repeated applications until a coating thickness of approximately .040 inches is obtained.

Final specimen configuration was a 3 inch by 3 inch square sheared from approximately 1/4 inch stock and a 1/4 inch diameter drilled or punched in the approximate center.

The application of polysulfide sealants required a special fixture so that uniform coating thickness could be obtained. Figure 6 is a sketch of the Sealant Coating Fixture. Around the edges of each specimen a narrow strip of .050 inch aluminum was bonded with double back tape to form a box on the surface to be coated. A cover plate was fabricated to fit over the specimen and rest on the aluminum straps. Holes were drilled in each corner of the cover plate just inside the box area. A base plate was fabricated to fit under the specimen with a plug in the center to fill the specimen pressurization port. The whole system is clamped together after applying release agent to the cover plate. The sealant is then injected into one of the corner holes of the cover plate until the cavity is filled and sealant is extruded from the other three holes.

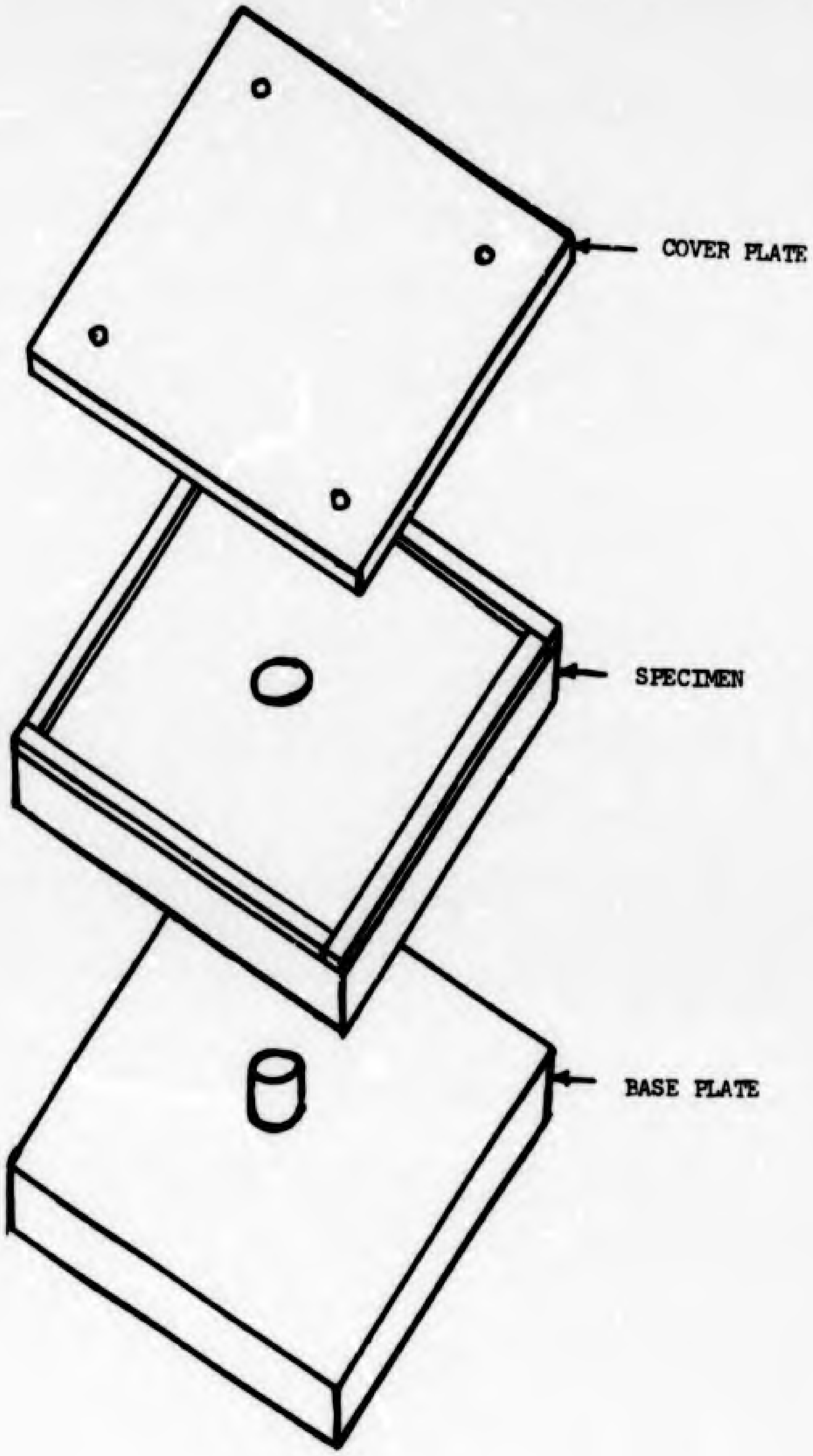


FIGURE 6

SEALANT COATING FIXTURE

2.1.5 Specimen Conditioning - Specimen were evaluated in both the "as prepared" condition as well as after being subjected to hostile environments simulating use conditions prior to evaluation for adhesion surface energy density.

Preselected specimens were subjected to the following conditioning:

Specimens were submerged in distilled water at room temperature for 24 hours, removed, wiped off and air dried.

Specimens were placed in an oven at 250°F in air for 24 hours, removed and allowed to come to room temperature in air.

Specimens were placed in an alternate immersion apparatus (Figure 7) and subjected to 30 day intermittent salt water exposure. The cycle consisted of immersing the specimen in a 3.5% salt water solution maintained between 6.6 pH and 6.8 pH for 10 minutes followed by air drying for 50 minutes. The test was continuous 24 hours a day for 30 days at which the specimens were removed rinsed with distilled water, wiped off and allowed to air dry.

All the conditioned specimens as well as the "as prepared" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to evaluation on the Blister Test Apparatus.

2.1.6 Specimen Evaluation - The "Blister Test" apparatus used for specimen evaluation is shown in the photograph, Figure 8 . Figure 9 is a close-up photograph of the specimen mounted on the specimen holder. The Schaevitz LVDT tripod holder is in place on the specimen and to the left may be seen the pressure transducer assembly. Another photograph, Figure 10, is a close-up of the specimen showing the displacement probe of the LVDT located above the pressurization port.

The following general procedure was followed:

1. Install 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 specimen to be tested in specimen holder and locate the LVDT tripod assembly so that the displacement probe is over the pressurization port.
4. Regulate nitrogen bottle gauge pressure
5. Set micrometer valve.
6. Turn on power to X-Y recorder.
7. Depress solenoid pressure release switch and open hand valve.

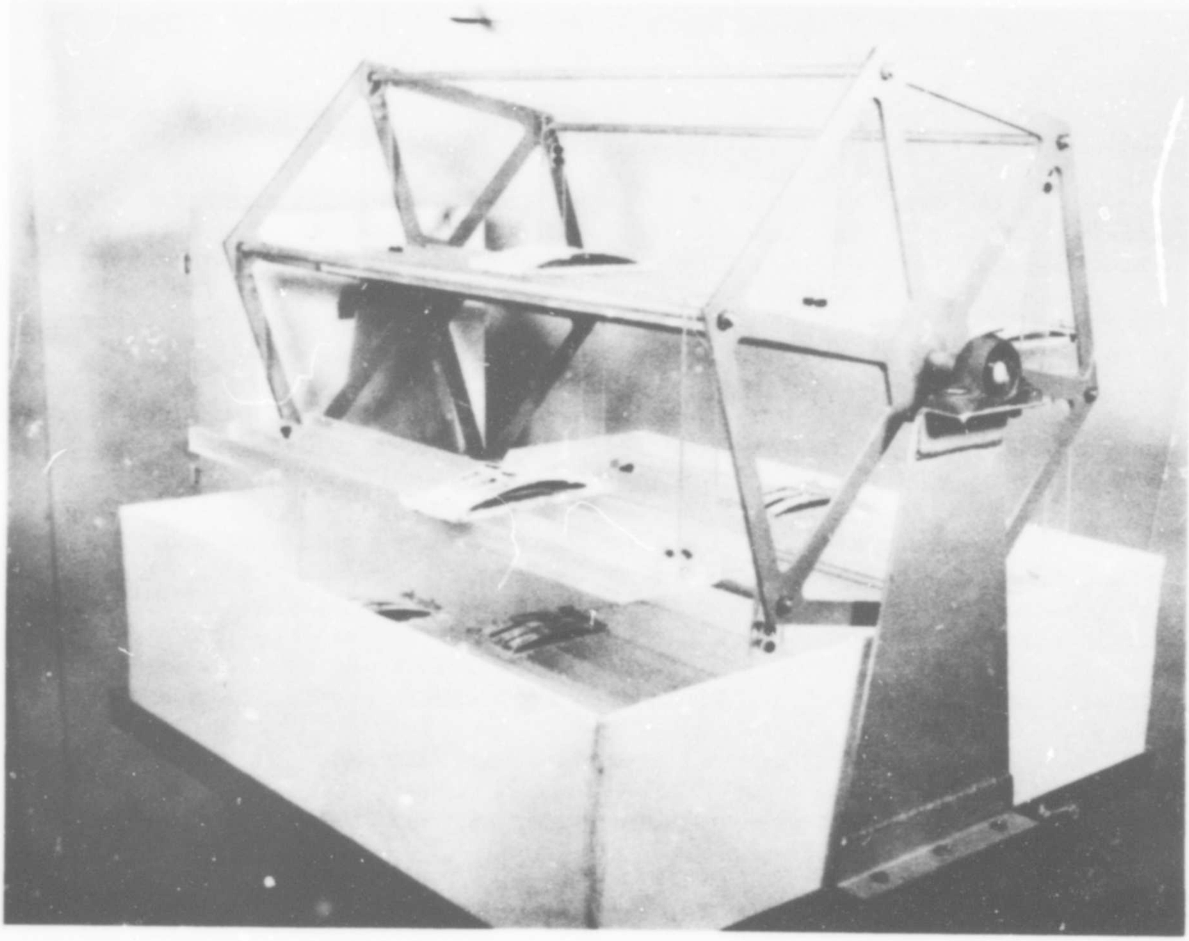


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FIGURE 7

ALTERNATE IMMERSION APPARATUS

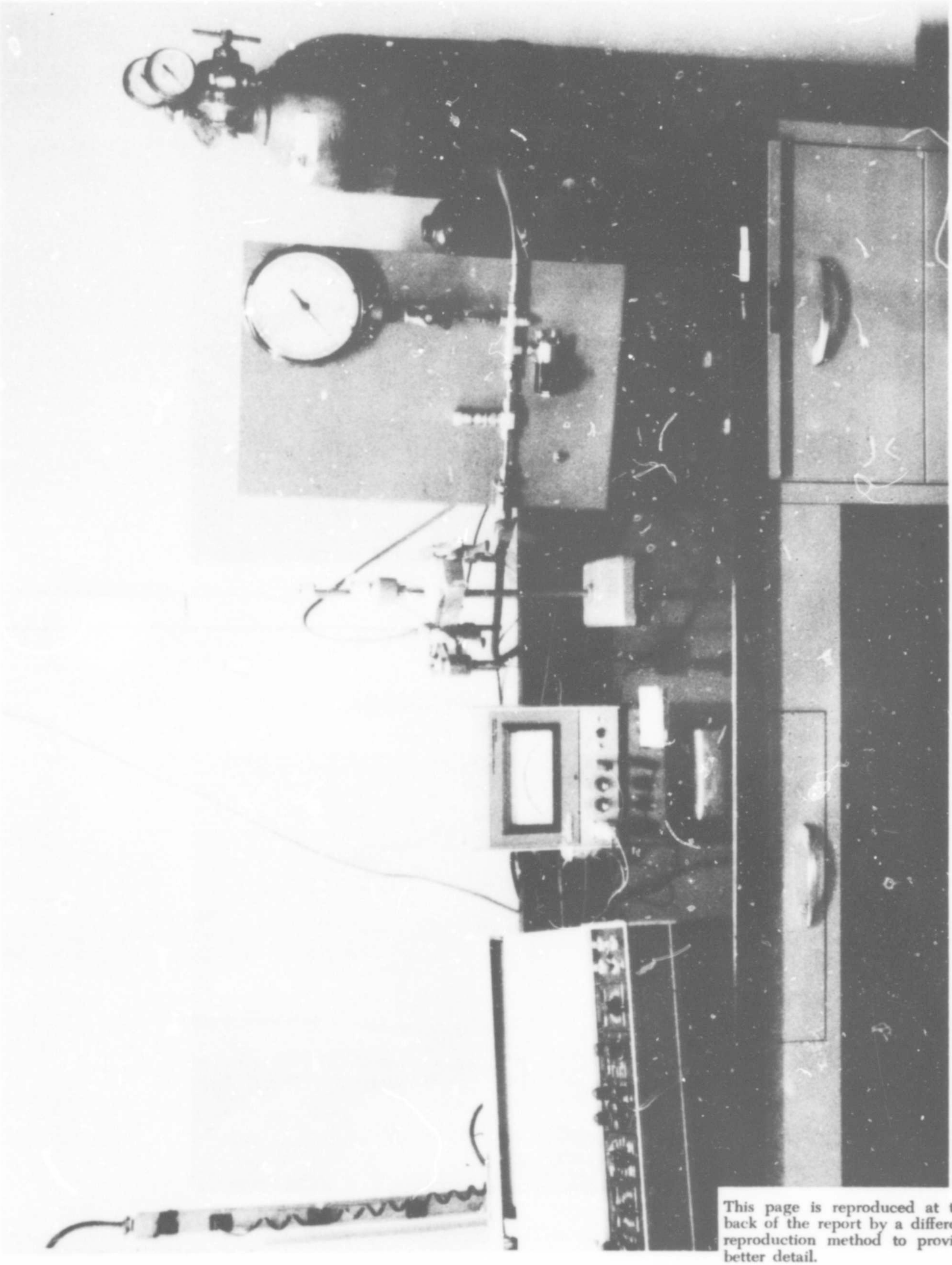

FIGURE 8 PHOTOGRAPH OF BLISTER TEST APPARATUS

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FIGURE 9

SPECIMEN ON SPECIMEN HOLDER

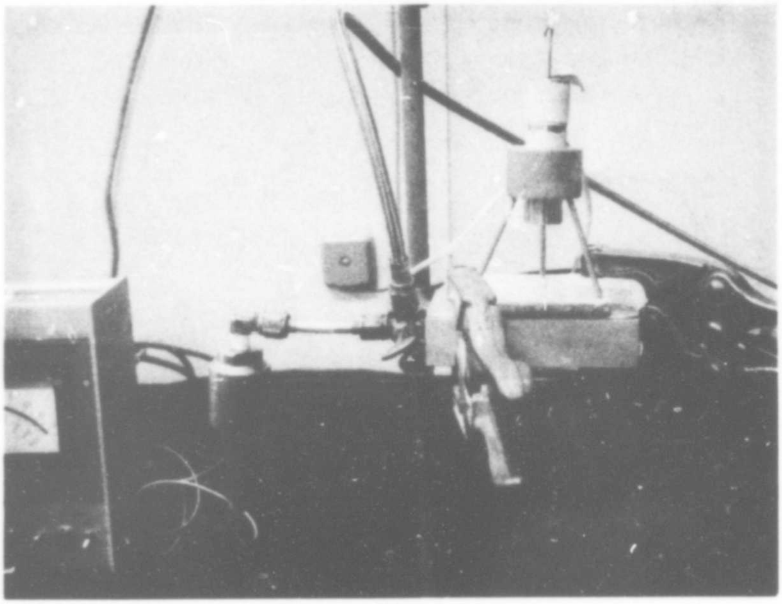
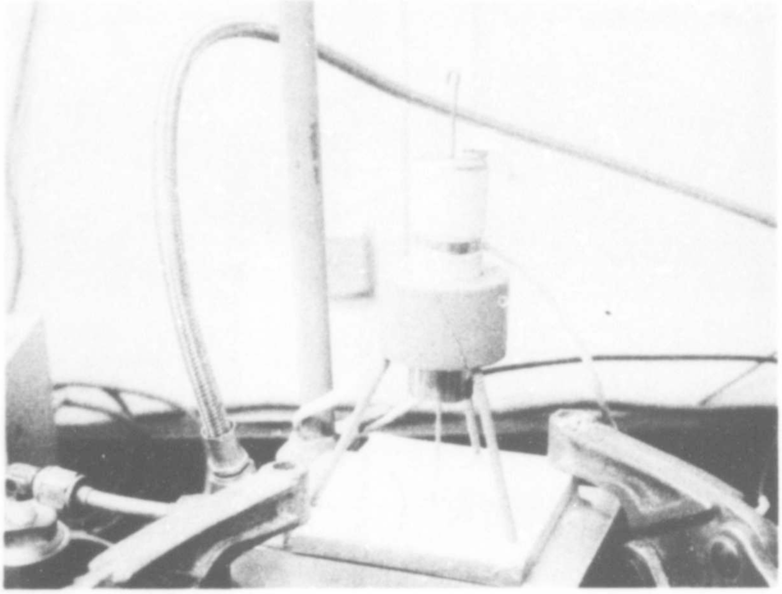


FIGURE 10

SCHAEVITZ LVDT ON SPECIMEN



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8. Release solenoid pressure release switch and allow pressure to increase in system until blister size starts to increase noted by change in slope of produced curve.

9. Depress solenoid pressure release switch to lower pressure below critical pressure.

10. Repeat steps 8 and 9 to obtain additional data until blister heights exceeds the scale span.

Results will then be calculated from the plot by determining the point where the slope change occurs indicating an increase in blister size.

Specimen evaluation on the "Blister Test" apparatus was done batch-wise as nearly as possible to minimize testing variables. When ever possible all processing variables with the same coating were tested the same day using the same diaphram (50 psig, 100 psig, or 200 psig) and the same instrument calibration.

2.2 PROCESSING STUDIES WITH TITANIUM ALLOY (Ti6Al4V)

2.2.1 Fluosilicic Acid/Aluminum Oxide Slurry Surface Treatment - A combination of Meseran Surface Analysis, electron scanning microscopic examination and lap shear strength values indicated that some form of mechanical action was necessary to remove the oxide layer in order to obtain good bonding. Discussions were held with Mr. G. W. Lively based on his previous experience with surface treatments of titanium alloys at VSD (contract no. F33615-71-C-1104) and reported in AFML-TR-72-70 (Reference 7). In this investigation it was found that for the titanium alloy (Ti6Al4V) the best results were obtained in bonding trials utilizing 2% fluosilicic acid ($H_2S_iF_6$) with 240 grit purified aluminum oxide (Al_2O_3). The ratio established for use in the Mechani-Chemical Surface Treatment Apparatus (Figure 11) was 500 ml of 240 grit Al_2O_3 to 2000 ml of 2% $H_2S_iF_6$.

Four variations of the surface treatment were selected based on grit blast time and chemical reaction time prior to being rinsed in distilled water and drying with filtered, dry compressed air.

Variation 1 - light grit blast - quick rinse
Grit blast the surface to be coated for 30 seconds using a cross coat pattern, rinse within one minute with distilled water and dry.

Variation 2 - light grit blast - delayed rinse to allow reaction time
Grit blast the surface to be coated for 30 seconds using a cross coat pattern, allow to stand wetted with the solution for 5 minutes, rinse with distilled water and dry.

Variation 3 - Light grit blast - reaction time - light grit blast
Grit blast the surface to be coated for 30 seconds using a cross coat pattern, allow to stand wetted with the solution for one minute, grit blast the surface to be coated for 30 seconds using a cross coat pattern and rinse within one minute with distilled water and dry.

Variation 4 - Heavy grit blast - extended reaction time.
Grit blast the surface to be coated until no original surfaces or other mechanical blemishes can be detected or a minimum of 2 minutes using a cross coat pattern. Allow to stand wetted with the solution for 15 minutes then rinse with distilled water and dry.

Twenty eight specimens were precleaned by vapor degreasing, hot alkaline cleaning and then processed by each of the 4 variations in preparation for coating application. After processing each specimen was marked for identification and wrapped in clean paper toweling to prevent contamination of the cleaned and processed surface.

2.2.2 Chromate Solution/Abrasive Nylon Pad - Chromate solution/abrasive nylon pad abrasion surface treatments have been evaluated in bonding applications to improve bond strengths. It had been found that 2 oz. of MIL-C-5541 chromate treatment compound (Material Code 8) per gallon of distilled water as the conversion coating and abrading the surface with an abrasive impregnated nylon pad improved adhesion. Four variations of the surface treatment were based on scrubbing time, chemical reaction and grade of the abrasive nylon pads, general purpose or ultrafine.

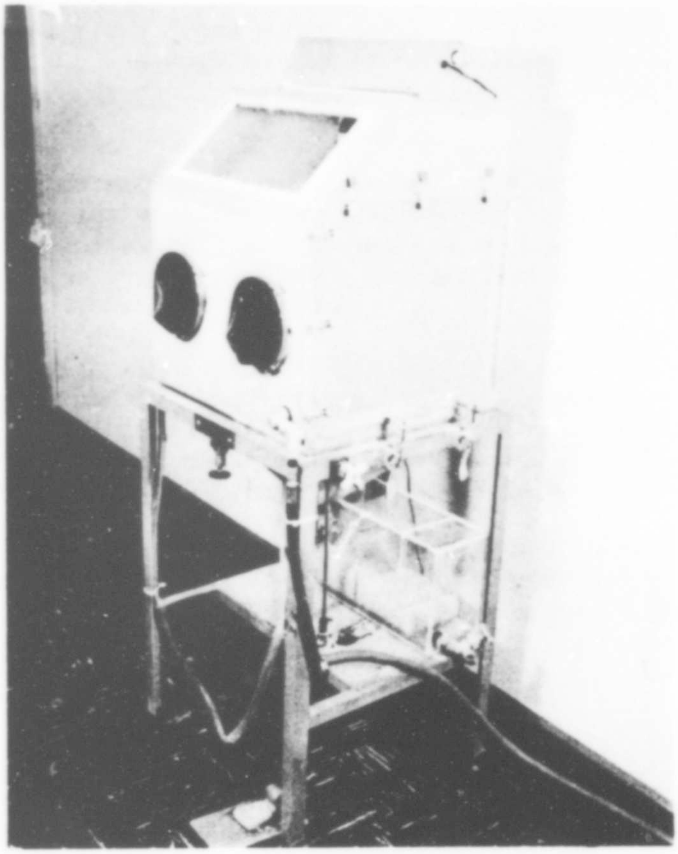

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FIGURE 11

MECHANI-CHEMICAL SURFACE TREATMENT APPARATUS

Variation 1 - Abrasion with fine pad - scrub the specimen thoroughly with an abrasive nylon pad (ultrafine) wetted with a 2 ounce per gallon solution of chromate compound for one minute. Rinse the specimen thoroughly with distilled water and air dry.

Variation 2 - Abrasion with coarse pad - scrub the specimen thoroughly with an abrasive nylon pad (general purpose) wetted with a 2 ounce per gallon solution of chromate compound for one minute. Rinse thoroughly with distilled water and air dry.

Variation 3 - Repeated abrasion with fine pad - scrub the specimen thoroughly with an abrasive nylon pad (ultrafine) wetted with a 2 ounce per gallon solution of chromate compound for one minute. Rinse the specimen thoroughly with distilled water and air dry. After one hour repeat the scrubbing, rinsing and drying so that each specimen receives two treatments.

Variation 4 - Repeated abrasion with coarse pad - scrub the specimen thoroughly with an abrasive nylon pad (general purpose) wetted with a 2 ounce per gallon solution of chromate compound for one minute. Rinse thoroughly with distilled water and air dry. After one hour repeat the scrubbing, rinsing and drying so that each specimen receives two treatments.

Four specimens were pre-cleaned by vapor degreasing followed by hot alkaline cleaning and then processed by each of the 4 variations in preparation for coating applications. Each specimen was marked for identification and wrapped in clean paper toweling to prevent contamination of the cleaned and processed surface.

2.2.3 Specimen Preparation - Fluosilicic Acid/Aluminum Oxide - four specimens were selected at random from each of the $H_2S_1F_6/Al_2O_3$, processed groups for coating with epoxy polyamide primer (MIL-P-23377). Each specimen was marked for identification, mounted on a board for spraying using double back masking tape. All 16 specimens were mounted on one board at random to be spray painted at the same time to minimize coating variables. A coating thickness of approximately .040 inch was obtained by repeated spray applications using a cross coat pattern.

Eight specimens were selected at random from each of the $H_2S_1F_6/Al_2O_3$ processed groups for coating with epoxy polyamide primer (MIL-P-23377) overcoated with polyurethane enamel (MIL-C-81773). Each specimen was marked for identification and mounted on boards with double back masking tape. The first four specimens of each variable were randomly placed on one board and the remainder were randomly placed on another board. Both boards were spray painted at the same time to minimize coating variables. A normal (0.5 to 0.7 mils) coating of epoxy polyamide primer was sprayed on each of the sets of 16 specimens using a cross coat pattern. After curing polyurethane enamel was applied. A coating thickness of approximately .040 inch was obtained by repeated spray applications using a cross coat pattern.

Eight specimens were selected at random from each of the $H_2S_1F_6/Al_2O_3$ processing groups for coating with Manganese cure Polysulfide Sealant (MIL-S-8802). Each specimen was marked for identification and a coating of approximately .050 inch was applied using the sealant coating fixture and allowed to cure.

The remaining eight specimens from each of the H_2SiF_6/Al_2O_3 processing groups were designated for coating with chromate cure polysulfide sealant (MIL-S-8802). Each specimen was marked for identification and a coating of approximately .050 inch was applied using the sealant coating fixture and allowed to cure.

A number of approaches were explored to select adhesion promoting additives or coupling agents for evaluation on the program. Vendors were contacted to obtain assistance in selection of coupling agents to be evaluated both as surface treatments and as additions to the epoxy polyamide primer. The following coupling agents were selected for evaluation.

1.	Trimethoxysilylpropethylenediamine	Material Code 11
2.	Trimethoxysilylpropethyleneepoxide	Material Code 12
3.	Vinyltriethoxysilane	Material Code 13
4.	Vinyltris (2-methoxy-ethoxy) silane	Material Code 14
5.	Hexamethyldisilane	Material Code 15

Preliminary review of blister test data on as prepared primer coated specimens indicated that all of the surface treatments yielded nearly equal results. Variations 2 and 4 were slightly superior, and variation 2 was selected as it required less time to process so would be more practical.

Twenty specimens were processed in accordance with variation 2 H_2SiF_6/Al_2O_3 prepared to evaluate the five coupling agents. Each of the five coupling agents was applied by brush to two specimens and then mixed with the epoxy polyamide primer and applied by brush to two additional specimens. The specimens were allowed to dry and then mounted on cardboard for spray coating with epoxy polyamide primer until a coating thickness of .040 inches minimum was obtained.

Chromate Solution/Abrasive Nylon Pad - Four specimens from each of the chromate solution/abrasive nylon pad processed groups for coating with epoxy polyamide primer (MIL-P-23377) were mounted on a board for spraying using double back masking tape. All specimens were mounted on one board at random to be spray painted at the same time to minimize variables. A coating thickness of approximately .040 inches was obtained by repeated spray applications using a cross coat pattern.

Preliminary review of blister test data on the primer coated specimens indicated that Variation 1 and Variation 4 yielded nearly equal results. Variation 1 was selected as it required less processing and 20 specimens were prepared for coupling agent evaluations. Each of the five coupling agents was applied by brush to two specimens and then mixed with the epoxy polyamide primer and applied by brush to two additional specimens. The specimens were allowed to dry and then mounted on cardboard for spray coating with epoxy polyamide primer until a coating thickness of .040 inches minimum was obtained.

2.2.4 Specimen Conditioning - Specimen conditioning of the coated titanium specimens consisted of the following treatments described in paragraph 2.1.5.

40 specimens subjected to 30 day intermittent salt water exposure
24 specimens subjected to 24 hour water soak
24 specimens subjected to 24 hours at 250°F

The conditioned specimens and the "as prepared" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to testing.

2.3 PROCESSING STUDIES WITH ALUMINUM ALLOYS (7075-T6)

2.3.1 Sulfuric Acid Anodize - Anodizing of aluminum greatly improves the corrosion resistance of the material and can be utilized as the interface surface treatment prior to coating. VSD and others have found great variability in adhesion resulting from this processing which has prevented taking full advantage of this corrosion protection.

The variation in processing shown in Table I were selected to determine the effect of cleaning prior to anodizing, concentration of electrolyte, current density and seal solution make up and temperature.

2.3.2 Specimen Preparation

Specimens were prepared in duplicate from each variation. Coating procedures reported in 2.2.3 were followed to prepare the following specimens for evaluation.

32 specimens -- coated with epoxy polyamide primer

80 specimens -- coated with coupling agent then epoxy polyamide primer

80 specimens -- coated with 1% coupling agent in epoxy polyamide primer then coated with epoxy polyamide primer

16 specimens -- coated with wash primer then coated with epoxy polyamide primer

32 specimens -- coated with epoxy polyamide primer then acrylic nitrocellulose lacquer

32 specimens -- coated with epoxy polyamide primer then polyurethane enamel

32 specimens -- coated with epoxy polyamide primer then a polyurethane elastomer

32 specimens -- coated with neoprene rain erosion coating

An additional 16 specimens were coated with the coupling agent found to be best based on a preliminary review of the blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning.

An additional 16 specimens were coated with coupling agent in epoxy polyamide primer found to be best, based on preliminary review of the blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning.

Polysulfide sealant, applied to 16 specimens cure was using the sealant coating fixture.

TABLE I

VARIATIONS IN SULFURIC ACID ANODIZING OF 7075 ALUMINUM *

VARIATION Specimen Marking	CLEANING	ANODIZING			SEALING		
		Amperage	Voltage	Time (Min)	Solution pH	Temperature °F	Time (Min)
1 7F1 - - -	Etch Clean then Specification	12	16	20	6.0	200	20
2 7F2 - - -	Specification	12	16	20	6.0	200	20
3 7F3 - - -	Specification	12	16	20	6.0	200	30
4 7F4 - - -	Specification	12	16	20	NO SEAL		
5 7F5 - - -	Specification	12	16	20	3.5 Chromic Acid	180	30
6 7F6 - - -	Specification	12	16	20	3.5 Chromic Acid	180	5
7 7F7 - - -	Specification	12	12	20	6.0	200	20
8 7F8 - - -	Specification	12	16	20 +	6.0	200	20

* CVA 9-14 Process Specification, Sulfuric Acid Anodic Treatment of Aluminum and Aluminum Alloys.
\+ Anodizing Solution Strength Lowered to 8.00% (by volume) from Specification Requirements.

2.3.3 Specimen Conditioning - Specimen conditioning of the coated aluminum consisted of 80 specimens be subjected to 30 day intermittent salt water exposure as described in paragraph 2.1.5.

The conditioned and "as prepared" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to testing.

2.4 PROCESSING STUDIES WITH ALUMINUM ALLOY (2024T3)

2.4.1 Chromic Acid Anodize - Anodizing of aluminum greatly improves the corrosion resistance of the material and can be utilized as the interface surface treatment prior to coating. VSD and other have found great variability in adhesion resulting from this processing which has prevented taking full advantage of this corrosion protection.

The variation in processing shown in Table II was selected to determine the effect of cleaning prior to anodizing, concentration of electrolyte, current density and seal solution makeup and temperature.

2.4.2 Specimen Preparation - Specimens were prepared in duplicate from each variation. Coating procedures reported in 2.2.3 were followed to prepare the following specimens for evaluation.

32 specimens -- coated with epoxy polyamide primer

80 specimens -- coated with coupling agent then epoxy polyamide primer.

80 specimens -- coated with 1% coupling agent in epoxy polyamide primer then coated with epoxy polyamide primer.

16 specimens -- coated with wash primer then epoxy polyamide primer

32 specimens -- coated with epoxy polyamide primer then acrylic nitrocellulose lacquer.

32 specimens -- coated with epoxy polyamide primer then polyurethane enamel

32 specimens -- coated with epoxy polyamide primer then a polyurethane elastomer

An additional 16 specimens were coated with the coupling agent found to be best based on a preliminary review of the blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning.

An additional 16 specimens were coated with the coupling agent in epoxy polyamide primer found to be best, based on preliminary review of the blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning.

Polysulfide sealant cure was applied to 16 specimens using the sealant cooling fixture.

2.4.3 Specimen Conditioning - Specimen conditioning of the coated aluminum consisted of 80 specimens being subjected to 30 day intermittent salt water exposure as described in paragraph 2.1.5.

The conditioned and "as prepared" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to testing.

TABLE II

VARIATIONS IN CHROMIC ACID ANODIZING OF 2024 ALUMINUM *

VARIATION Specimen Marking	CLEANING	ANODIZING			SEALING		
		Amperage per ft^2	Voltage	Time (Min)	Solution pH	Temperature (°F)	Time (Min)
2F1 1 ---	Etch Clean then Specification	1 Min.	22 ± 2	30	5.5	97	8
2F2 2 ---	Specification	1 Min	22 ± 2	30	5.5	97	8
2F3 3 ---	Specification	1 Min	22 ± 2	30	NO SEAL		
2F4 4 ---	Specification	1 Min	22 ± 2	30	3.5 Chromic Acid	180	5
2F5 5 ---	Specification	1 Min	22 ± 2	30	3.5 Chromic Acid	180	30
2F6 6 ---	Specification	1 Min	22 ± 2	30	3.5 Chromic Acid	150	5
2F7 7 ---	Specification	1 Min	22 ± 2	30	3.5 Chromic Acid	150	30
2F8 8 ---	Specification	1 Min	40 ± 2	40	5.4	97	8

* CVA 9-4 Process Specification, Chromic Acid Anodic Treatment of Aluminum and Aluminum Alloys.

2.5 PROCESSING STUDIES ON STEEL (4340)

2.5.1 Phosphate Chemical Conversion Treatment - Phosphate chemical conversion treatments have been found to improve the adhesion of coating to steel. Four variations of phosphate chemical conversion treatment were selected for evaluation including both dip and brush-on applications with two different property materials (Table III).

2.5.2 Specimen Preparation - Specimens were prepared in duplicate from each of the four variations of phosphate chemical conversion coatings. Coating procedures reported in 2.2.3 were followed to prepare the following specimens

16 specimens -- coated with epoxy polyamide primer

40 specimens -- coated with coupling agent then epoxy polyamide primer

16 specimens -- coated with epoxy polyamide primer then polyurethane enamel

An additional 8 specimens were coated with the coupling agent found to be best based on preliminary review of blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning.

Polysulfide sealant cure was applied to 8 specimens using the sealant coating fixture.

2.5.3 Specimen Conditioning - Specimen conditioning of the coated steel consisted of 24 specimens being subjected to 30 day intermittent salt water exposure as described in paragraph 2.1.5.

The conditioned and "as prepered" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to testing.

TABLE III

VARIATIONS IN PHOSPHATE CHEMICAL CONVERSION TREATMENTS OF STEEL

Variation Specimen Marking	Treating Solution	Application Method
4F1 - - -	Phosphate Treatment Concentrate * Mix 1 part with 2 parts refined water	Apply solution by brush coating and allow to stand in the wetted condition for 5 minutes. Flush surface with refined water and wipe off excess and allow to air dry.
4F2 - - -		Apply solution by brush coating and allow to stand in the wetted condition for 5 minutes DO NOT FLUSH Wipe off excess solution and allow to air dry.
4F3 - - -	Phosphate Treatment Concentrate. Mix 7 parts with 93 parts of refined water. Add 2 oz. by weight. Accelerator 130 per 100 gallons.	Immerse in treating solution at 185°F ± 10°F for 5 minutes. Rinse thoroughly in solution containing 4 oz. chromic acid and 4 oz phosphoric acid per 100 gallons. Wipe off excess rinse solution and allow to air dry.
4F4 - - -		Immerse in treating solution at 185°F ± 10°F for 10 minutes. Rinse thoroughly in solution containing 4 oz. chromic acid and 4 oz. phosphoric acid per 100 gallons. Wipe off excess rinse solution and allow to air dry.

* Material Code 16 per CVA 9-17
\+ Material Code 17 per CVA 9-84

2.6 PROCESSING STUDIES ON CADMIUM PLATED STEEL (4340)

2.6.1 Chromate and Phosphate Chemical Treatments - Adhesion to cadmium plated surface has always required a chemical conversion treatment to obtain good adhesion. Both chromate and phosphate chemical conversion are available, so four variations of each system were selected for evaluation (Table IV). Steel specimens were fabricated and then cadmium plated in accordance with Specification CVA 5-2, "Plating, Cadmium/Electro deposited."

2.6.2 Specimen Preparation - Specimens were prepared in duplicate from each of the eight processing variations. Coating procedures reported in 2.2.3 were followed to prepare the following specimens.

32 specimens -- coated with epoxy polyamide primer

80 specimens -- coated with coupling agent then epoxy polyamide primer

32 specimens -- coated with epoxy polyamide primer then polyurethane enamel

An additional 16 specimens were coated with the coupling agent found to be best based on preliminary review of blister test data and coated with epoxy polyamide primer for 30 day intermittent salt water conditioning. Polysulfide sealant cure was applied to 16 specimens using the sealant coating fixture.

2.6.3 Specimen Conditioning - Specimen conditioning of the coated cadmium plated steel consisted of 48 specimens being subjected to 30 day intermittent salt water exposure as described in paragraph 2.1.5.

The conditioned and "as prepared" specimens were placed in a 50% relative humidity cabinet for a minimum of 7 days prior to testing.

TABLE IV

VARIATIONS IN PHOSPHATE AND CHROMATE CHEMICAL CONVERSION TREATMENTS OF CADMIUM PLATED STEEL

Variation Specimen Marking	Treating Solution	Application Method
CF1 - - -	Chromate Treatment Concentrate* 1 oz per gallon in refined water	Apply solution by brush coating and allow to stand in the wetted condition for 30 seconds. Flush surfaces with refined water, wipe off excess and allow to air dry.
CF2 - - -	Chromate Treatment Concentrate* 1.5 oz per gallon in refined water	Apply solution by brush coating and allow to stand in the wetted condition for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.
CF3 - - -	Chromate Treatment Concentrate* 1 oz per gallon in refined water	Immerse in treating solution at 100°F ± 5° for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.
CF4 - - -	Chromate Treatment Concentrate* 1 oz per gallon in refined water	Immerse in treating solution at 150°F ± 5°F for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.
CF5 - - -	Phosphate Treatment Concen. + 1 part in 2 parts refined water	Apply solution by brush coating and allow to stand in the wetted condition for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.
CF6 - - -		Apply solution by brush coating and allow to stand in the wetted condition for 30 seconds. DO NOT RINSE Wipe off excess solution and allow to air dry.
CF7 - - -		Immerse in treating solution at 100°F ± 5°F for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.
CF8 - - -		Immerse in treating solution at 150°F ± 5°F for 30 seconds. Flush surface with refined water, wipe off excess and allow to air dry.

* Material Code 18 per CVA 9-17 (Chromate Chemical Conversion Treatment)
\+ Material Code 19 per CVA 9-17 (Phosphate Chemical Conversion Treatment)

3.0 RESULTS

3.1 RESULTS OF PROCESSING STUDIES WITH TITANIUM ALLOY (Ti6Al4V)

3.1.1 Evaluation of Epoxy Polyamide Primer Coated Specimens - Table V lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the variations of the Fluorosilicic Acid/Aluminum Oxide abrasion surface treatments. Two specimens of each variation were tested in the "as prepared" condition and two specimens of each variation were tested after being subjected to 30 days intermittent exposure to salt water.

Table V also lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the variations of the chromate solution/abrasive nylon pad abrasion treatment. Two specimens of each variation were tested in the as prepared condition and two specimens of each variation were tested after being subjected to 30 day intermittent salt water exposure. Visual examination of specimens during and after testing on the blister test apparatus indicated some of the data obtained were not valid. Some of the specimens cracked in the built-in flaw area and in other instances the blister would grow to one side and not produce a circular growth. Both of these problems ended in low results which should be eliminated from consideration.

3.1.2 Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer Specimens - Table VI lists the results obtained on specimens which were coated with epoxy polyamide primer then polyurethane enamel after receiving one of the variations of the Fluorosilicic/Aluminum Oxide abrasion surface treatments. Two specimens of each variation were tested in the "as prepared" condition, after 24 hour soak in distilled water, after being subjected to 30 day intermittent salt water exposure and after 24 hours exposure in air at 250°F.

Visual examination of these specimens found no cracks but some flaws extended in non circular configurations which yielded poor data.

3.1.3 Evaluation of Coupling Agents - The best variation of surface treatment of both the Fluosilicic Acid/Aluminum Oxide abrasion surface treatment and the chromate solution/abrasive nylon pad abrasion surface treatment was selected from a review of the data generated for the evaluation of the 5 coupling agents.

Table VII lists the results obtained when the coupling agents were applied to duplicate specimens of each of the surface treatment variations prior to coating with epoxy polyamide primer.

Table VIII lists the results of the same coupling agents mixed into epoxy polyamide primer and then overcoated with epoxy polyamide primer.

The coupling agents showed additional problems in obtaining meaningful data. Some of the material acted as parting agents and no bond was obtained between the treated surface and the epoxy polyamide primers. Very low results were obtained on other specimens due to the non-adherence of the primer to the treated surface.

When the coupling agent was mixed with the primer adhesion was somewhat better but when the flaw broke through the prime coat there was little adherence between the prime coat and the epoxy polyamide primer build up.

TABLE V

Evaluation of Epoxy Polyamide Primer on Titanium Alloy (Ti6Al4V)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ a*
TF1P-1 TF1P-2	Fluosilicic Acid/Aluminum Oxide Variation 1	Epoxy Polyamide Primer	As Prepared	1.39+ 2.40
TF1P-3 TF1P-4			30 Day Salt Water Exposure	1.79 1.96
TF2P-1 TF2P-2	Fluosilicic Acid/Aluminum Oxide Variation 2	Epoxy Polyamide Primer	As Prepared	1.88 2.48
TF2P-3 TF2P-4			30 Day Salt Water Exposure	1.55 1.75
TF3P-1 TF3P-2	Fluosilicic Acid/Aluminum Oxide Variation 3	Epoxy Polyamide Primer	As Prepared	1.83 1.94
TF3P-3 TF3P-4			30 Day Salt Water Exposure	1.83 1.62
TF4P-1 TF4P-2	Fluosilicic Acid/Aluminum Oxide Variation 4	Epoxy Polyamide Primer	As Prepared 30	1.19+ 1.90
TF4P-3 TF4P-4			30 Day Salt Water Exposure	1.58 2.04
TA1P-1 TA1P-2	Chromate Solution/Abrasive Nylon Pad Variation 1	Epoxy Polyamide Primer	As Prepared	1.59 1.09+
TA1P-3 TA1P-4			30 Day Salt Water Exposure	1.58 1.51
TA2P-1 TA2P-2	Chromate Solution/Abrasive Nylon Pad Variation 2	Epoxy Polyamide Primer	As Prepared	.93 .98
TA2P-3 TA2P-4			30 Day Salt Water Exposure	1.63 2.11

* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE V
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ a*
TA3P-1 TA3P-2	Chromate Solution/Abrasive Nylon Pad Variation 3	Epoxy Polyamide Primer	As Prepared	1.06 1.10
TA3P-3 TA3P-4	Chromate Solution/Abrasive Nylon Pad Variation 4	Epoxy Polyamide Primer	30 Day Salt Water Exposure	.98+ 1.59
TA4P-1 TA4P-2	Chromate Solution/Abrasive Nylon Pad Variation 4	Epoxy Polyamide Primer	As Prepared	1.56 1.44
TA4P-3 TA4P-4			30 Day Salt Water Exposure	1.50 1.58

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE VI

Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer
On Titanium Alloy (Ti6Al4V)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF1PU-1	Fluosilicic Acid/Aluminum Oxide Variation 1	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.28
TF1PU-2				2.14
TF1PU-3			24 Hours Distilled Water Soak	2.31
TF1PU-4				2.25
TF1PU-5			30 Day Salt Water Exposure	2.92
TF1PU-6				2.39
TF1PU-7			24 Hours at 250°F	2.86
TF1PU-8				2.24
TF2PU-1	Fluosilicic Acid/Aluminum Oxide Variation 2	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.06
TF2PU-2				2.13
TF2PU-3			24 Hour Distilled Water Soak	2.47
TF2PU-4				2.25
TF2PU-5			30 Day Salt Water Exposure	2.30
TF2PU-6				2.65
TF2PU-7			24 Hours at 250°F	3.30
TF2PU-8				1.29+
TF3PU-1	Fluosilicic Acid/Aluminum Oxide Variation 3	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	1.90
TF3PU-2				1.92
TF3PU-3			24 Hour Distilled Water Soak	2.68
TF3PU-4				2.72
TF3PU-5			30 Day Salt Water Exposure	1.92
TF3PU-6				2.02
TF3PU-7			24 Hours at 250°F	4.30
TF3PU-8				2.16+

* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE VI
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF4PU-1	Fluosilicic Acid/Aluminum Oxide	Epoxy Polyamide	As Prepared	1.92
TF4PU-2	Variation 4	Primer		2.58
		+		
TF4PU-3		Polyurethane	24 Hour Distilled	2.17
TF4PU-4		Enamel	Water Soak	2.35
TF4PU-5			30 Day Salt	2.03
TF4PU-6			Water Exposure	1.85
TF4PU-7			24 Hours at	2.92
TF4PU-8			250°F	2.68

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* Adhesion surface energy density (in-lbs/in^2)

\+ [illegible]ation during or after test indicated results not valid.

TABLE VII

Evaluation of Coupling Agents Applied to Surfaces of Titanium Alloy (Ti6Al4V)
Prior to Coating with Epoxy Polyamide Primer

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ a*
TFC+P-1 TFC+P-2	Fluosilicic Acid/Aluminum Oxide Variation 2	Coupling Agent 1 + Epoxy Polyamide Primer	As Prepared	.47 .66
TFC+P-3 TFC+P-4		Coupling Agent 2 + Epoxy Polyamide Primer	As Prepared	2.05 1.82
TFC+P-5 TFC+P-6		Coupling Agent 3 + Epoxy Polyamide Primer	As Prepared	1.72 1.41
TFC+P-7 TFC+P-8		Coupling Agent 4 + Epoxy Polyamide Primer	As Prepared	1.28 1.39
TFC+P-9 TFC+P-10		Coupling Agent 5 + Epoxy Polyamide Primer	As Prepared	No Adhesion+ No Adhesion+
TAC+P-1 TAC+P-2	Chromate Solution/Abrasive Nylon Pad Variation 1	Coupling Agent 1 + Epoxy Polyamide Primer	As Prepared	.57 .58
TAC+P-3 TAC+P-4		Coupling Agent 2 + Epoxy Polyamide Primer	As Prepared	.99 1.05
TAC+P-5 TAC+P-6		Coupling Agent 3 + Epoxy Polyamide Primer	As Prepared	1.27 1.39
TAC+P-7 TAC+P-8		Coupling Agent 4 + Epoxy Polyamide Primer	As Prepared	.65 .67
TAC+P-9 TAC+P-10		Coupling Agent 5 + Epoxy Polyamide Primer	As Prepared	No Test .86

* Adhesion surface energy density (in-lbs/in^2)
\+ Deformation during or after test indicated results not valid.

TABLE VIII

Evaluation of Coupling Agents Mixed in Epoxy Polyamide Primer Applied to Surface of Titanium Alloy (Ti6Al4V)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TFC/P-1 TFC/P-2	Fluosilicic Acid/Aluminum Oxide Variation 2	Coupling Agent 1 in Primer + Epoxy Polyamide Primer	As Prepared	2.34 2.46
TFC/P-3 TFC/P-4		Coupling Agent 2 in Primer + Epoxy Polyamide Primer	As Prepared	2.70 2.84
TFC/P-5 TFC/P-6		Coupling Agent 3 in Primer + Epoxy Polyamide Primer	As Prepared	2.50 2.16
TFC/P-7 TFC/P-8		Coupling Agent 4 in Primer + Epoxy Polyamide Primer	As Prepared	2.64 2.35
TFC/P-9 TFC/P-10		Coupling Agent 5 in Primer + Epoxy Polyamide Primer	As Prepared	2.53 2.55
TAC/P-1 TAC/P-2	Chromate Solution/Abrasive Nylon Pad Variation 1	Coupling Agent 1 in Primer + Epoxy Polyamide Primer	As Prepared	2.03 1.40+
TAC/P-3 TAC/P-4		Coupling Agent 2 in Primer + Epoxy Polyamide Primer	As Prepared	2.25 1.56+
TAC/P-5 TAC/P-6		Coupling Agent 3 in Primer + Epoxy Polyamide Primer	As Prepared	1.96 1.99
TAC/P-7 TAC/P-8		Coupling Agent 4 in Primer + Epoxy Polyamide Primer	As Prepared	2.07 1.72

* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE VIII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TFC/P-9 TFC/P-10		Coupling Agent 5 in Primer + Epoxy Polyamide Primer	As Prepared	2.58 2.15

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* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

3.1.4 **Evaluation of Polysulfide Sealants** - Table IX lists the results obtained on specimens which were coated with polysulfide sealant, manganese cure, after receiving one of the variations of the Fluosilicic Acid/Aluminum Oxide abrasion surface treatment. Two specimens of each were tested in the "as prepared" condition, after 24 hours soak in distilled water, after being subjected to 30 days intermittent salt water exposure and after 24 hours in air at 250°F.

Table X lists the results of the same evaluation using a chromate cure polysulfide sealant.

The sealant although adhering very well in most cases would fail prematurely in some instances. These were found to be mainly caused by flaws or air bubbles in the coating which would rupture, thus terminating the test.

TABLE IX

Evaluation of Manganese Cure Polysulfide Sealant on Titanium Alloy (Ti6Al4V)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF1SM-1	Fluosilicic Acid/Aluminum Oxide Variation 1	Sealant, Polysulfide Manganese Cure	As Prepared	3.05
TF1SM-2				2.96
TF1SM-3			24 Hour Distilled Water Soak	2.70
TF1SM-4				2.50
TF1SM-5			30 Day Salt Water Exposure	2.54
TF1SM-6				3.27
TF1SM-7			24 Hours at 250°F	1.54
TF1SM-8				.68+
TF2SM-1	Fluosilicic Acid/Aluminum Oxide Variation 2	Sealant, Polysulfide Manganese Cure	As Prepared	2.82
TF2SM-2				2.80
TF2SM-3			24 Hour Distilled Water Soak	2.82
TF2SM-4				3.28
TF2SM-5			30 Day Salt Water Exposure	3.40
TF2SM-6				2.62
TF2SM-7			24 Hours at 250°F	2.86
TF2SM-8				1.77
TF3SM-1	Fluosilicic Acid/Aluminum Oxide Variation 3	Sealant, Polysulfide Manganese Cure	As Prepared	2.77
TF3SM-2				----+
TF3SM-3			24 Hour Distilled Water Soak	2.70
TF3SM-4				3.02
TF3SM-5			30 Day Salt Water Exposure	3.05
TF3SM-6				3.06
TF3SM-7			24 Hours at 250°F	.626+
TF3SM-8				2.77

* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE IX
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF4SM-1	Fluosilicic Acid/Aluminum Oxide	Sealant, Polysulfide	As Prepared	3.19
TF4SM-2	Variation 4	Manganese Cure		3.21
TF4SM-3			24 Hours Distilled	3.52
TF4SM-4			Water Soak	3.02
TF4SM-5			30 Day Salt	3.28
TF4SM-6			Water Exposure	---- +
TF4SM-7			24 Hours at	1.467
TF4SM-8			250°F	-----+

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* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE X
Evaluation of Chromate Cure Polysulfide Sealant on Titanium Alloy (Ti6Al4V)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF1SC-1 TF1SC-2	Fluosilicic Acid/Aluminum Oxide Variation 1	Sealant, Polysulfide Chromate Cure	As Prepared	4.68 3.25
TF1SC-3 TF1SC-4			24 Hour Distilled Water Soak	4.76 1.21+
TF1SC-5 TF1SC-6			30 Day Salt Water Exposure	1.54+ 4.83
TF1SC-7 TF1SC-8			24 Hours at 250°F	1.68 1.68
TF2SC-1 TF2SC-2	Fluosilicic Acid/Aluminum Oxide Variation 2	Sealant, Polysulfide Chromate Cure	As Prepared	3.88 4.46
TF2SC-3 TF2SC-4			24 Hour Distilled Water Soak	2.83 4.38
TF2SC-5 TF2SC-6			30 Day Salt Water Exposure	4.11 3.21
TF2SC-7 TF2SC-8			24 Hours at 250°F	2.11 ----+
TF3SC-1 TF3SC-2	Fluosilicic Acid/Aluminum Oxide Variation 3	Sealant, Polysulfide Chromate Cure	As Prepared	4.65 4.43
TF3SC-3 TF3SC-4			24 Hour Distilled Water Soak	4.98 3.88
TF3SC-5 TF3SC-6			30 Days Salt Water Exposure	1.40+ 4.21

* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

TABLE X
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
TF3SC-7			24 Hours at	1.00
TF3SC-8			250°F	1.03
TF4SC-1	Fluosilicic Acid/Aluminum Oxide	Sealant, Polysulfide	As Prepared	4.47
TF4SC-2	Variation 4	Chromate Cure		5.07
TF4SC-3			24 Hour Distilled	3.25
TF4SC-4			Water Soak	4.36
TF4SC-5			30 Day Salt	4.44
TF4SC-6			Water Exposure	4.78
TF4SC-7			24 Hours at	3.91
TF4SC-8			250°F	2.92

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* Adhesion surface energy density (in-lbs/in^2)
\+ Lamination during or after test indicated results not valid.

3.2 RESULTS OF PROCESSING STUDIES WITH ALUMINUM/ALLOY (7075T6)

3.2.1 Evaluation of Epoxy Polyamide Primer Coated Specimens - Table XI lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the sulfuric acid anodize variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 days intermittent exposure to salt water.

3.2.2 Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer Specimens - Table XII lists the results obtained on specimens which were coated with epoxy polyamide primer then polyurethane enamel after receiving one of the sulfuric acid anodize variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent exposure to salt water.

The polyurethane enamel stretched more than was noted when testing the primer. Good agreement between four or five tests on the same specimen and agreement between duplicates indicate valid results.

3.2.3 Evaluation of Acrylic Nitrocellulose Lacquer Over Epoxy Polyamide Primer Specimens - Table XIII lists the results obtained on specimens which were coated with epoxy polyamide primer then acrylic nitrocellulose lacquer after receiving one of the sulfuric acid anodize variation processes. Two specimens of each variation in processing and lacquer application were tested in the "as prepared" condition.

The lacquer specimens were very brittle and did not lend themselves to this test method. Many specimens cracked when pressurization started thus no results were obtained.

3.2.4 Evaluation of Wash Primer with Epoxy Polyamide Primer - Table XIV lists the results obtained on specimens which were treated with wash primer prior to coating with epoxy polyamide primer after receiving one of the sulfuric acid anodize variation processes. Two specimens of each variation in processing were tested in the "as prepared" condition.

3.2.5 Evaluation of Coupling Agents - Table XV lists the results obtained on specimens which were coated with one of five coupling agents after receiving one of the sulfuric acid anodize variation processes. Two specimens of each processing variation were treated with one of the coupling agents prior to being coated with epoxy polyamide primer. All specimens were tested in "as prepared" condition.

The results obtained were reviewed and the coupling agent which yielded the best γ_a, adhesion surface energy density, was used to prepare duplicate specimens representing each anodizing processing variation. These were conditioned for 30 days in intermittent salt water exposure. The results of tests of these specimens are listed in Table XVI.

Table XVII lists the results obtained on specimens which were coated with one of five coupling agents mixed in epoxy polyamide primer after receiving one of the variations of sulfuric acid anodizing. Two specimens of each processing variation were treated with one of the coupling agents in epoxy polyamide primer mixture prior to being coated with epoxy polyamide primer. All specimens were tested in "as prepared" condition.

TABLE XI

Evaluation of Epoxy Polyamide Primer on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1P-1 7F1P-2	Sulfuric Acid Anodize Bariation 1	Epoxy Polyamide Primer	As Prepared	2.50 2.41
7F1P-3 7F1P-4			30 Day Salt Water Exposure	2.89 2.78
7F2P-1 7F2P-2	Sulfuric Acid Anodize Variation 2	Epoxy Polyamide Primer	As Prepared	3.03 2.84
7F2P-3 7F2P-4			30 Day Salt Water Exposure	2.86 2.65
7F3P-1 7F3P-2	Sulfuric Acid Anodize Variation 3	Epoxy Polyamide Primer	As Prepared	2.64 2.71
7F3P-3 7F3P-4			30 Day Salt Water Exposure	3.18 3.20
7F4P-1 7F4P-2	Sulfuric Acid Anodize Variation 4	Epoxy Polyamide Primer	As Prepared	2.33 2.42
7F4P-3 7F4P-4			30 Day Salt Water Exposure	2.18 2.62
7F5P-1 7F5P-2	Sulfuric Acid Anodize Variation 5	Epoxy Polyamide Primer	As Prepared	1.90 1.89
7F5P-3 7F5P-4			30 Day Salt Water Exposure	2.23 2.10
7F6P-1 7F6P-2	Sulfuric Acid Anodize Variation 6	Epoxy Polyamide Primer	As Prepared	2.45 2.08
7F6P-3 7F6P-4			30 Day Salt Water Exposure	2.53 2.18

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XI

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F7P-1	Sulfuric Acid Anodize Variation 7	Epoxy Polyamide Primer	As Prepared	2.43
7F7P-2				2.39
7F7P-3			30 Day Salt Water Exposure	2.72
7F7P-4				2.84
7F8P-1	Sulfuric Acid Anodize Variation 8	Epoxy Polyamide Primer	As Prepared	2.35
7F8P-2				2.48
7F6P-3			30 Day Salt Water Exposure	2.68
7F8P-4				2.82

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XII

Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	.98
7F1PU-2	Variation 1	Primer +		.82
		Polyurethane Enamel	3	
7F1PU-3			30 Day Salt	1.02
7F1PU-4			Water Exposure	1.04
7F2PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	.74
7F2PU-2	Variation 2	Primer +		.86
		Polyurethane Enamel		
7F2PU-3			30 Day Salt	.83
7F2PU-4			Water Exposure	.99
7F3PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	.74
7F3PU-2	Variation 3	Primer +		.88
		Polyurethane Enamel		
7F3PU-3			30 Day Salt	1.10
7F3PU-4			Water Exposure	1.07
7F4PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	1.10
7F4PU-2	Variation 4	Primer +		1.11
		Polyurethane Enamel		
7F4PU-3			30 Day Salt	.75
7F4PU-4			Water Exposure	1.21
7F5PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	.55
7F5PU-2	Variation 5	Primer +		.51
		Polyurethane Enamel		
7F5PU-3			30 Day Salt	.53
7F5PU-4			Water Exposure	.48
7F6PU-1	Sulfuric Acid Anodize	Epoxy Polyamide	As Prepared	.60
7F6PU-2	Variation 6	Primer +		.74
		Polyurethane Enamel		
7F6P-3			30 Day Salt	.72
7F6P-4			Water Exposure	.67

* Adhesion surface energy density (in-lbs/in^2)
\+ [illegible]nation during or after test indicated results not valid.

TABLE XII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F7PU-1	Sulfuric Acid Anodize Variation 7	Epoxy Polyamide Enamel + Polyurethane Enamel	As Prepared	.95
7F7PU-2				.90
7F7PU-3			30 Day Salt Water Exposure	.99
7F7Pu-4				1.03
7F8PU-1	Sulfuric Acid Anodize Variation 8	Epoxy Polyamide Enamel + Polyurethane Enamel	As Prepared	.88
7F8PU-2				.92
7F8PU-3			30 Day Salt Water Exposure	1.02
7F8PU-4				.98

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XIII

Evaluation of Acrylic Nitrocellulose Lacquer over Epoxy Polyamide Primer on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1PL-1 7F1PL-2	Sulfuric Acid Anodize Variation 1	Epoxy Polyamide Primer + Lacquer	As Prepared	.26 .33
7F1PL-3 7F1PL-4			Lacquer Coating after 24 Hours	.48 .40
7F2PL-1 7F2PL-2	Sulfuric Acid Anodize Variation 2	Epoxy Polyamide Primer + Lacquer	As Prepared	-----+ .46
7F2PL-3 7F2PL-4			Lacquer Coating After 24 Hours	.69 .44
7F3PL-1 7F3PL-2	Sulfuric Acid Anodize Variation 3	Epoxy Polyamide Primer + Lacquer	As Prepared	.30 .42
7F3PL-3 7F3PL-4			Lacquer Coating after 24 Hours	----+ Bad Spec+
7F4PL-1 7F4PL-2	Sulfuric Acid Anodize Variation 4	Epoxy Polyamide Primer + Lacquer	As Prepared	.23 .62
7F4PL-3 7F4PL-4			Lacquer Coating after 24 Hours	.25 -----+
7F5PL-1 7F5PL-2	Sulfuric Acid Anodize Variation 5	Epoxy Polyamide Primer + Lacquer	As Prepared	.24 .23
7F5PL-3 7F5PL-4			Lacquer Coating after 24 Hours	-----+ .45
7F6PL-1 7F6PL-2	Sulfuric Acid Anodize Variation 6	Epoxy Polymide Primer + Lacquer	As Prepared	-----+ .47
7F6PL-3 7F6PL-4			Lacquer Coating after 24 Hours	.19 .11

* Adhesion surface energy density (in-lbs/in^2)

\+ Deformation during or after test indicated results not valid.

TABLE XIII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F7PL-1 7F7PL-2	Sulfuric Acid Anodize Variation 7	Epoxy Polyamide Primer + Lacquer	As Prepared	.24 .22
7F7PL-3 7F7PL-4			Lacquer Coating after 24 Hours	.33 .25
7F8PL-1 7F8PL-2	Sulfuric Acid Anodize Variation 8	Epoxy Polyamide Primer + Lacquer	As Prepared	.37 .25
7F8PL-3 7F8PL-4			Lacquer Coating after 24 Hours	-----+ .48

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* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE XIV

Evaluation of Wash Primer Prior to Epoxy Polyamide Primer on Aluminum Alloy (7075T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1WP-1 7F1WP-2	Sulfuric Acid Anodize Variation 1	Wash Primer + Epoxy Polyamide	As Prepared	1.60 1.60
7F2WP-1 7F2WP-2	Sulfuric Acid Anodize Variation 2	Wash Primer + Epoxy Polyamide	As Prepared	1.71 1.48
7F3WP-1 7F3WP-2	Sulfuric Acid Anodize Variation 3	Wash Primer + Epoxy Polyamide	As Prepared	2.60 2.67
7F4WP-1 7F4WP-2	Sulfuric Acid Anodize Variation 4	Wash Primer + Epoxy Polyamide	As Prepared	2.13 1.87
7F5WP-1 7F5WP-2	Sulfuric Acid Anodize Variation 5	Wash Primer + Epoxy Polyamide	As Prepared	1.16 1.37
7F6WP-1 7F6WP-2	Sulfuric Acid Anodize Variation 6	Wash Primer + Epoxy Polyamide	As Prepared	1.58 1.88
7F7WP-1 7F7WP-2	Sulfuric Acid Anodize Variation 7	Wash Primer + Epoxy Polyamide	As Prepared	1.48 1.56
7F8WP-1 7F8WP-2	Sulfuric Acid Anodize Variation 8	Wash Primer + Epoxy Polyamide	As Prepared	1.61 1.45

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XV

Evaluation of Coupling Agents Applied to Surfaces of Aluminum Alloy (7075-T6) Prior To Coating with Epoxy Polyamide Primer

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.53
7F1C1-2	Variation 1	+ Epoxy Polyamide		.56
7F2C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.50
7F2C1-2	Variation 2	+ Epoxy Polyamide		.55
7F3C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.62
7F3C1-2	Variation 3	+ Epoxy Polyamide		.74
7F4C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.47
7F4C1-2	Variation 4	+ Epoxy Polyamide		.65
7F5C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.54
7F5C1-2	Variation 5	+ Epoxy Polyamide		.62
7F6C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.67
7F6C1-2	Variation 6	+ Epoxy Polyamide		.67
7F7C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.52
7F7C1-2	Variation 7	+ Epoxy Polyamide		.48
7F8C1-1	Sulfuric Acid Anodize	Coupling Agent 1	As Prepared	.53
7F8C1-2	Variation 8	+ Epoxy Polyamide]		.49
7F1C2-1	Sulfuric Acid Anodize	Coupling Agent 2	As Prepared	2.50
7F1C2-2	Variation 1	+ Epoxy Polyamide		2.67
7F2C2-1	Sulfuric Acid Anodize	Coupling Agent 2	As Prepared	2.94
7F2C2-2	Variation 2	+ Epoxy Polyamide		2.97
7F3C2-1	Sulfuric Acid Anodize	Coupling Agent 2	As Prepared	2.59
7F3C2-2	Variation 3	+ Epoxy Polyamide		2.64
7F4C2-1	Sulfuric Acid Anodize	Coupling Agent 2	As Prepared	2.10
7F4C2-2	Variation 4	+ Epoxy Polyamide		2.65

* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE XV

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F5C2-1 7F5C2-2	Sulfuric Acid Anodize Variation 5	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.92 2.76
7F6C2-1 7F6C2-2	Sulfuric Acid Anodize Variation 6	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.41 2.43
7F7C2-1 7F7C2-2	Sulfuric Acid Anodize Variation 7	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.45 2.51
7F8C2-1 7F8C2-2	Sulfuric Acid Anodize Variation 8	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.48 2.61
7F1C3-1 7F1C3-2	Sulfuric Acid Anodize Variation 1	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.30 1.01
7F2C3-1 7F2C3-2	Sulfuric Acid Anodize Variation 2	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.92+ 1.68
7F3C3-1 7F3C3-2	Sulfuric Acid Anodize Variation 3	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.73 1.03
7F4C3-1 7F4C3-2	Sulfuric Acid Anodize Variation 4	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.21 .63
7F5C3-1 7F5C3-2	Sulfuric Acid Anodize Variation 5	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.82 .56
7F6C3-1 7F6C3-2	Sulfuric Acid Anodize Variation 6	Coupling Agent 3 + Epoxy Polyamide	As Prepared	Blew + Blew +
7F7C3-1 7F7C3-2	Sulfuric Acid Anodize Variation 7	Coupling Agent 3 + Epoxy Polyamide	As Prepared	----+ 1.20
7F8C3-1 7F8C3-2	Sulfuric Acid Anodize Variation 8	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.18 1.22

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XV
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1C4-1 7F1C4-2	Sulfuric Acid Anodize Variation 1	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.39 .95+
7F2C4-1 7F2C4-2	Sulfuric Acid Anodize Variation 2	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.08 1.03
7F3C4-1 7F3C4-2	Sulfuric Acid Anodize Variation 3	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.35 1.26
7F4C4-1 7F4C4-2	Sulfuric Acid Anodize Variation 4	Coupling Agent 4 + Epoxy Polyamide	As Prepared A	1.55 1.38
7F5C4-1 7F5C4-2	Sulfuric Acid Anodize Variation 5	Coupling Agent 4 + Epoxy Polyamide	As Prepared	.54 .79
7F6C4-1 7F6C4-2	Sulfuric Acid Anodize Variation 6	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.29 .95
7F7C4-1 7F7C4-2	Sulfuric Acid Anodize Variation 7	Coupling Agent 4 + Epoxy Polyamide	As Prepared	.82 1.03
7F8C4-1 7F8C4-2	Sulfuric Acid Anodize Variation 8	Coupling Agent 4 + Epoxy Polyamide	As Prepared	.98 1.21
7F1C5-1 7F1C5-2	Sulfuric Acid Anodize Variation 1	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.14 .73
7F2C5-1 7F2C5-2	Sulfuric Acid Anodize Variation 2	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.93 .64
7F3C5-1 7F3C5-2	Sulfuric Acid Anodize Variation 3	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.14 1.03
7F4C5-1 7F4C5-2	Sulfuric Acid Anodize Variation 4	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.59 1.33

* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

TABLE XV

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F5C5-1 7F5C5-2	Sulfuric Acid Anodize Variation 5	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.45 .58
7F6C5-1 7F6C5-2	Sulfuric Acid Anodize Variation 6	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.93 .88
7F7C5-1 7F7C5-2	Sulfuric Acid Anodize Variation 7	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.81 .78
7F8C5-1 7F8C5-2	Sulfuric Acid Anodize Variation 8	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.93 1.01

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XVI

Evaluation of Coupling Agent Applied to Surface of Aluminum Alloy (7075-T6) Prior to Coating with Epoxy Polyamide Primer for Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.74
7F1CX-2	Variation 1	+ Epoxy Polyamide	Water Exposure	1.63
7F2CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.72
7F2CX-2	Variation 2	+ Epoxy Polyamide	Water Exposure	1.71
7F3CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.66
7F3CX-2	Variation 3	+ Epoxy Polyamide	Water Exposure	2.46
7F4CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.99
7F4CX-2	Variation 4	+ Epoxy Polyamide	Water Exposure	1.43+
7F5CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.84
7F5CX-2	Variation 5	+ Epoxy Polyamide	Water Exposure	1.86
7F6CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	Blew +
7F6CX-2	Variation 6	+ Epoxy Polyamide	Water Exposure	1.53
7F7CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.62
7F7CX-2	Variation 7	+ Epoxy Polyamide	Water Exposure	1.58
7F8CX-1	Sulfuric Acid Anodize	Coupling Agent 2	30 Day Salt	1.74
7F8CX-2	Variation 8	+ Epoxy Polyamide	Water Exposure	1.53

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XVII

Evaluation of Coupling Agents Mixed in Epoxy Polyamide Primer Applied to Surface of Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1C1-3 7F1C1-4	Sulfuric Acid Anodize Variation 1	Coupling Agent 1 in Primer + Epoxy Polyamide	As Prepared	2.93 2.76
7F2C1-3 7F2C1-4	Sulfuric Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.17 2.86
7F3C1-3 7F3C1-4	Sulfuric Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.18 1.39+
7F4C1-3 7F4C1-4	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.21 2.85
7F5C1-3 7F5C1-4	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	1.89+ 2.61
7F6C1-3 7F6C1-4	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.02 3.07
7F7C1-3 7F7C1-4	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.56 2.72
7F8C1-3 7F8C1-4	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.78 2.64
7F1C2-3 7F1C2-4	Sulfuric Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.44 2.68

* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE XVII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F2C2-3	Sulfuric Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.80
7F2C2-4				2.47
7F3C2-3	Sulfuric Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.80
7F3C2-4				2.63
7F4C2-3	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.05
7F4C2-4				3.10
7F5C2-3	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.87
7F5C2-4				2.55
7F6C2-3	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.91
7F6C2-4				2.55
7F7C2-3	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.42
7F7C2-4				2.58
7F8C2-3	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.61
7F8C2-4				2.42
7F1C3-3	Sulfuric Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.06
7F1C3-4				2.74
7F2C3-3	Sulfuric Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.62
7F2C3-4				2.61

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XVII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F3C3-3	Sulfuric Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.14
7F3C3-4				3.15
7F4C3-3	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.55
7F4C3-4				2.71
7F5C3-3	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	1.34
7F5C3-4				1.35
7F6C3-3	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.01
7F6C3-4				3.06
7F7C3-3	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.98
7F7C3-4				2.79
7F8C3-3	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.78
7F8C3-4				2.87
7F1C4-3	Sulfuric Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.81
7F1C4-4				2.73
7F2C4-3	Sulfuric Acid Anodize Variation 2	Coupling Agent Primer + Epoxy Polyamide	As Prepared	3.09
7F2C4-4				3.28
7F3C4-3	Sulfuric Acid Anodize Variation 3	Coupling Agent Primer + Epoxy Polyamide	As Prepared	2.99
7F3C4-4				3.15

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XVII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F4C4-3 7F4C4-4	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.04 2.75
7F5C4-3 7F5C4-4	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	1.38 1.36
7F6C4-3 7F6C4-4	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.93 2.18
7F7C4-3 7F7C4-4	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.72 2.78
7F8C4-3 7F8C4-4	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.64 2.71
7F1C5-3 7F1C5-4	Sulfuric Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.89 2.66
7F2C5-3 7F2C5-4	Sulfuric Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.00 3.27
7F3C5-3 7F3C5-4	Sulfuric Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.09 2.66
7F4C5-3 7F4C5-4	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.18 2.92

* Adhesion surface energy density (in-lbs/in^2)

\+ [illegible]nation during or after test indicated results not valid.

TABLE XVII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F5C5-3 7F5C5-4	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	1.66 1.57
7F6C5-3 7F6C5-4	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.70 3.11
7F7C5-3 7F7C5-4	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.64 2.82
7F8C5-3 7F8C5-4	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.62 2.88

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

The results obtained were reviewed and the coupling agent in epoxy polyamide mixture which yielded the best γa, adhesion surface energy density, was used to prepare duplicate specimens representing each anodizing processing variation for conditioning for 30 days in intermittent salt water exposure. The results of tests of these specimens are listed in Table XVIII. Visual examination of specimens and striping the coating from the specimen after testing showed very poor or no adhesion of the primer to the surface coated with the coupling agent in some instances. When the coupling agent was mixed in the primer, poor adhesion was obtained between the prime coat and the over coat of epoxy polyamide primer on some specimens.

The "built-in" flaw in the specimens did not always extend in a circular configuration on the specimens showing poor adhesion which could result in poor agreement between duplicates.

3.2.6 Evaluation of Neoprene Rain Erosion Coating - Table XIX lists the results obtained on specimens which were coated with neoprene rain erosion coating material after receiving one of the variations of sulfuric acid anodizing and a prime coat of epoxy polyamide primer. Two approaches were evaluated in applying the neoprene, 1 spray coat to build up the necessary thickness and, 2 brush coat one coat followed by spray coating to build up the necessary thickness. Two specimens of each processing variation were prepared and tested in the "as prepared" condition.

The coating was very elastic and stretched severely thus yielding low adhesion surface energy density measurements. The specimens which had the first coat brushed on the surface and followed by spray developed visual strains in the coating appearing to be brush strokes. Some rupturing of the blisters occurred when this was noted instead of an increase in blister diameter.

3.2.7 Evaluation of Polyurethane Elastomer - Table XX lists the results obtained on specimens which were coated with polyurethane elastomer after receiving one of the variations of sulfuric acid anodizing and a prime coat of epoxy polyamide primer. Two specimens of each processing variation were evaluated in the "as prepared" condition and two after being subjected to 30 days intermittent exposure to salt water.

The polyurethane elastomer stretched severely during testing which with other materials had caused low values. With this material it was necessary to use a 200 psi diaphram whereas a 50 psig or a 100 psig diaphram had been used on all other materials. Even with the high pressure the polyurethane failed by increases in blister diameter with good agreement on several failures on the same specimen and between duplicates.

3.2.8 Evaluation of Polysulfide Sealant - Table XXI lists the results obtained on specimens which were coated with polysulfide sealant, chromate cure, after receiving one of the variations of sulfuric acid anodizing. Two specimens of each variation were tested in the "as prepared" condition.

The polysulfide sealants gave some problems in testing due to entrapped bubbles or voids. Most specimens which yielded low values could be visually examined and the flaw located. The stretching of the material by this test method caused some of the blisters with flaws in them to rupture instead of extending in a circular mode.

TABLE XVIII

Evaluation of Coupling Agent Mixed in Epoxy Polyamide Primer Applied to Surface of Aluminum Alloy (7075-T6) for Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1CY-1 7F1CY-2	Sulfuric Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.15 1.24+
7F2CY-1 7F2CY-2	Sulfuric Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.29+ 2.23
7F3CY-1 7F3CY-2	Sulfuric Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.02 2.08
7F4CY-1 7F4CY-2	Sulfuric Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.29 2.41
7F5CY-1 7F5CY-2	Sulfuric Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.01 1.90
7F6CY-1 7F6CY-2	Sulfuric Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.26 1.86
7F7CY-1 7F7CY-2	Sulfuric Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.23+ 2.02
7F8CY-1 7F8CY-2	Sulfuric Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	2.14 1.88

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* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE XIX

Evaluation of Neoprene Rain Erosion Coating Over Epoxy Polyamide Primer on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1N-1 7F1N-2	Sulfuric Acid Anodize Variation 1	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	1.22 1.03
7F1N-3 7F1N-4			Brush As Prepared	.15 .15
7F2N-1 7F2N-2	Sulfuric Acid Anodize Variation 2	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	.63 .15
7F2N-3 7F2N-4			Brush As Prepared	.13 .79
7F3N-1 7F3N-2	Sulfuric Acid Anodize Variation 3	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	.32 .16
7F3N-3 7F3N-4			Brush As Prepared	.14 .61
7F4N-1 7F4N-2	Sulfuric Acid Anodize Variation 4	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	.15 .25
7F4N-3 7F4N-4			Brush As Prepared	.19 .60
7F5N-1 7F5N-2	Sulfuric Acid Anodize Variation 5	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	.12 .11
7F5N-3 7F5N-4			Brush As Prepared	.65 .10
7F6N-1 7F6N-2	Sulfuric Acid Anodize Variation 6	Epoxy Polyamide Primer + Neoprene	Spray As Prepared	.11 .12
7F6N-3 7F6N-4			Brush As Prepared	.57 .81

* Adhesion surface energy density (in-lbs/in^2)
\+ Delamination during or after test indicated results not valid.

TABLE XIX
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ a*
7F7N-1	Sulfuric Acid Anodize	Epoxy Polyamide	Spray	1.26
7F7N-2	Variation 7	Primer + Neoprene	As Prepared	.92
7F7N-3			Brush	.13
7F7N-4			As Prepared	.15
7F8N-1	Sulfuric Acid Anodize	Epoxy Polyamide	Spray	.88
7F8N-2	Variation 8	Primer + Neoprene	As Prepared	.97
7F8N-3			Brush	.16
7F8N-4			As Prepared	.12

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* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

TABLE XX

Evaluation of Polyurethane Elastomer Coating Over Epoxy Polyamide Primer on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
7F1PE-1 7F1PE-2	Sulfuric Acid Anodize Variation 1	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	13.5 14.5
7F1PE-3 7F1PE-4			30 Day Salt Water Exposure	15.6 15.0
7F2PE-1 7F2PE-2	Sulfuric Acid Anodize Variation 2	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	12.5 12.2
7F2PE-3 7F2PE-4			30 Day Salt Water Exposure	13.5 15.2
7F3PE-1 7F3PE-2	Sulfuric Acid Anodize Variation 3	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	13.8 13.1
7F3PE-3 7F3PE-4			30 Day Salt Water Exposure	11.5 10.9
7F4PE-1 7F4PE-2	Sulfuric Acid Anodize Variation 4	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	14.8 12.5
7F4PE-3 7F4PE-4			30 Day Salt Water Exposure	11.0 11.9
7F5PE-1 7F5PE-2	Sulfuric Acid Anodize Variation 5	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	13.6 14.0
7F5PE-3 7F5PE-4			30 Day Salt Water Exposure	13.6 13.0
7F6PE-1 7F6PE-2	Sulfuric Acid Anodize Variation 6	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	12.8 13.9
7F6PE-3 7F6PE-4			30 Day Salt Water Exposure	13.1 12.5

* Adhesion surface energy density (in-lbs/in^2)

\+ [illegible]ation during or after test indicated results not valid.

TABLE XX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	a*
7F7PE-1	Sulfuric Acid Anodize Variation 7	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	12.8
7F7PE-2				12.2
7F7PE-3			30 Day Salt Water Exposure	13.5
7F7PE-4				14.0
7F8PE-1	Sulfuric Acid Anodize Variation 8	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	13.2
7F8PE-2				13.5
7F8PE-3			30 Day Salt Water Exposure	12.5
7F8PE-4				13.8

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXI

Evaluation of Polysulfide Sealant on Aluminum Alloy (7075-T6)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	a*
7F1S-1 7F1S-2	Sulfuric Acid Anodize Variation 1	Sealant, Chromate Cure	As Prepared	----+ 3.22
7F2S-1 7F2S-2	Sulfuric Acid Anodize Variation 2	Sealant, Chromate Cure	As Prepared	3.47 ----+
7F3S-1 7F3S-2	Sulfuric Acid Anodize Variation 3	Sealant, Chromate Cure	As Prepared	2.07 1.90
7F4S-1 7F4S-2	Sulfuric Acid Anodize Variation 4	Sealant, Chromate Cure	As Prepared	3.81 3.82
7F5S-1 7F5S-2	Sulfuric Acid Anodize Variation 5	Sealant, Chromate Cure	As Prepared	.93+ 2.15
7F6S-1 7F6S-2	Sulfuric Acid Anodize Variation 6	Sealant, Chromate Cure	As Prepared	.88+ 3.97
7F7S-1 7F7S-2	Sulfuric Acid Anodize Variation 7	Sealant, Chromate Cure	As Prepared	----+ 3.12
7F8S-1 7F8S-2	Sulfuric Acid Anodize Variation 8	Sealant, Chromate Cure	As Prepared	2.29 2.84

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

3.3 RESULTS OF PROCESSING STUDIES WITH ALUMINUM ALLOY (2024-T3)

3.3.1 Evaluation of Epoxy Polyamide Primer Coated Specimens - Table XXII lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the chromic acid anodize variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 days intermittent exposure to salt water.

3.3.2 Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer Specimens - Table XXIII lists the results obtained on specimens which were coated with epoxy polyamide primer then polyurethane enamel after receiving one of the chromic acid anodize variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent exposure to salt water.

The polyurethane enamel stretched more than was noted when testing the primer. Good agreement between four or five tests on the same specimen and agreement between duplicates indicate valid results although some specimens did fail to yield valid results. The two specimens which gave invalid results failed due to flaws in the coating.

3.3.3 Evaluation of Acrylic Nitrocellulose Lacquer over Epoxy Polyamide Primer Specimens - Table XXIV lists the results obtained on specimens which were coated with epoxy polyamide primer then acrylic nitrocellulose lacquer after receiving one of the chromic acid anodize variation processes. Two specimens of each variation in processing and two lacquer application were tested in the "as prepared" condition.

The lacquer specimens were very brittle and did not lend themselves to this test method. Many specimens cracked when pressurization started thus no results or questionable results were obtained.

3.3.4 Evaluation of Wash Primer with Epoxy Polyamide Primer - Table XXV lists the results obtained on specimens which were treated with wash primer prior to coating with epoxy polyamide primer after receiving one of the chromic acid anodize variation processes. Two specimens of each variation in processing were tested in the "as prepared" condition.

3.3.5 Evaluation of Coupling Agents - Table XXVI lists the results obtained on specimens which were coated with one of five coupling agents after receiving one of the chromic acid anodize variations processes. Two specimens of each processing variation were treated with one of the coupling agents prior to being coated with epoxy polyamide primer. All specimens were tested in the "as prepared" condition.

The results obtained were reviewed and the coupling agent which yielded the best γ_a, adhesion surface energy density, was used to prepare duplicate speicmens representing each anodizing processing variation. These were conditioned for 30 days with intermittent exposure to salt water. The results are listed in Table XXVII.

TABLE XXII

Evaluation of Epoxy Polyamide Primer on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1P-1 2F1P-2	Chromic Acid Anodize Variation 1	Epoxy Polyamide Primer	As Prepared	3.39 3.32
2F1P-3 2F1P-4			30 Day Salt Water Exposure	3.09 2.81
2F2P-1 2F2P-2	Chromic Acid Anodize Variation 2	Epoxy Polyamide Primer	As Prepared	3.35 3.15
2F2P-3 2F2P-4			30 Day Salt Water Exposure	3.23 3.04
2F3P-1 2F3P-2	Chromic Acid Anodize Variation 3	Epoxy Polyamide Primer	As Prepared	3.45 3.20
2F3P-3 2F3P-4			30 Day Salt Water Exposure	3.05 2.81
2F4P-1 2F4P-2	Chromic Acid Anodize Variation 4	Epoxy Polyamide Primer	As Prepared	3.22 3.27
2F4P-3 2F4P-4			30 Day Sale Water Exposure	3.12 2.93
2F5P-1 2F5P-2	Chromic Acid Anodize Variation 5	Epoxy Polyamide Primer	As Prepared	3.39 3.29
2F5P-3 2F5P-4			30 Day Salt Water Exposure	3.37 3.12
2F6P-1 2F6P-2	Chromic Acid Anodize Variation 6	Epoxy Polyamide Primer	As Prepared	3.33 3.42
2F6P-3 2F6P-4			30 Day Salt Water Exposure	3.36 3.11

* Adhesion surface energy density (in-lbs/in^2)

\+ [illegible]mation during or after test indicated results not valid.

TABLE XXII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F7P-1 2F7P-2	Chromic Acid Anodize Variation A	Epoxy Polyamide Primer	As Prepared	3.21 3.33
2F7P-3 2F7P-4			30 Day Salt Water Exposure	3.47 3.35
2F8P-1 2F8P-2	Chromic Acid Anodize Variation B	Epoxy Polyamide Primer	As Prepared	3.28 3.36
2F8P-3 2F8P-4			30 Day Salt Water Exposure	3.12 2.96

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* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE XXIII

Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer on Aluminum Alloy (2024 - T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1PU-1 2F1PU-2	Chromic Acid Anodize Variation 1	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.90 3.06
2F1PU-3 2F1PU-4			30 Day Salt Water Exposure	1.11+ 1.60
2F2PU-1 2F2PU-2	Chromic Acid Anodize Variation 2	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.78 3.07
2F2PU-3 2F2PU-4			30 Day Salt Water Exposure	1.98 2.16
2F3PU-1 2F3PU-2	Chromic Acid Anodize Variation 3	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.68 2.45
2F3PU-3 2F3PU-4			30 Day Salt Water Exposure	2.42 2.66
2F4PU-1 2F4PU-2	Chromic Acid Anodize Variation 4	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.76 ----+
2F4PU-3 2F4PU-4			30 Day Salt Water Exposure	2.50 2.10
2F5PU-1 2F5PU-2	Chromic Acid Anodize Variation 5	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.51 3.05
2F5PU-3 2F5PU-4			30 Day Salt Water Exposure	2.63 2.35
2F6PU-1 2F6PU-2	Chromic Acid Anodize Variation 6	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	3.04 2.68
2F6PU-3 2F6PU-4			30 Day Salt Water Exposure	2.80 2.90

* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE XXIII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F7PU-1	Chromic Acid Anodize Variation 7	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	3.01
2F7PU-2				2.36
2F7PU-3			30 Day Salt Water Exposure	2.55
2F7PU-4				2.65
2F8PU-1	Chromic Acid Anodize Variation 8	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	2.32
2F8PU-2				2.48
2F8PU-3			30 Day Salt Water Exposure	2.64
2F8PU-4				2.54

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXIV

Evaluation of Acrylic Nitrocellulose Lacquer over Epoxy Polyamide Primer on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1PL-1	Chromic Acid Anodize Variation 1	**Epoxy Polyamide** Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	.50
2F1PL-2				.40
2F1PL-3				.54
2F1PL-4				.59
2F2PL-1	Chromic Acid Anodize Variation 2	Epoxy Polyamide Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	.62
2F2PL-2				.66
2F2PL-3				.67
2F2PL-4				.56
2F3PL-1	Chromic Acid Anodize Variation 3	Epoxy Polyamide Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	.66
2F3PL-2				.83
2F3PL-3				.72
2F3PL-4				.53
2F4PL-1	Chromic Acid Anodize Variation 4	Epoxy Polyamide Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	1.33
2F4PL-2				.97+
2F4PL-3				.79
2F4PL-4				1.19
2F5PL-1	Chromic Acid Anodize Variation 5	Epoxy Polyamide Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	.47
2F5PL-2				.90
2F5PL-3				1.30
2F5PL-4				1.33
2F6PL-1	Chromic Acid Anodize Variation 6	Epoxy Polyamide Primer + Lacquer	As Prepared Lacquer Coating after 24 Hours	.75
2F6PL-2				.91
2F6PL-3				----+
2F6PL-4				.73

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXIV

Evaluation of Acrylic Nitrocellulose Lacquer over Epoxy Polyamide Primer on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F7PL-1	Chromic Acid Anodize Variation 7	Epoxy Polyamide Primer + Lacquer	As Prepared	.98
2F7PL-2				.82
2F7PL-3			Lacquer Coating after 24 Hours	.68
2F7PL-4				.62
2F8PL-1	Chromic Acid Anodize Variation 8	Epoxy Polyamide Primer + Lacquer	As Prepared	.42
2F8PL-2				.48
2F8PL-3			Lacquer Coating after 24 Hours	.56
2F8PL-4				.59

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXV

Evaluation of Wash Primer Prior to Epoxy Polyamide Primer on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1WP-1	Chromic Acid Anodize Variation 1	Wash Primer + Epoxy Polyamide	As Prepared	.50
2F1WP-2				.49
2F2WP-1	Chromic Acid Anodize Variation 2	Wash Primer + Epoxy Polyamide	As Prepared	.49
2F2WP-2				.41
2F3WP-1	Chromic Acid Anodize Variation 3	Wash Primer + Epoxy Polyamide	As Prepared	.47
2F3WP-2				.66
2F4WP-1	Chromic Acid Anodize Variation 4	Wash Primer + Epoxy Polyamide	As Prepared	.48
2F4WP-2				.94
2F5WP-1	Chromic Acid Anodize Variation 5	Wash Primer + Epoxy Polyamide	As Prepared	.46
2F5WP-2				.49
2F6WP-1	Chromic Acid Anodize Variation 6	Wash Primer + Epoxy Polyamide	As Prepared	.72
2F6WP-2				.49
2F7WP-1	Chromic Acid Anodize Variation 7	Wash Primer + Epoxy Polyamide	As Prepared	.80
2F7WP-2				.59
2F8WP-1	Chromic Acid Anodize Variation 8	Wash Primer + Epoxy Polyamide	As Prepared	.46
2F8WP-2				.52

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVI

Evaluation of Coupling Agents Applied to Surfaces of Aluminum Alloy (2024-T3) Prior to Coating with Epoxy Polyamide Primer

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1C1-1 2F1C1-2	Chromic Acid Anodize Variation 1	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.61 .51
2F2C1-1 2F2C1-2	Chromic Acid Anodize Variation 2	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.66 .81
2F3C1-1 2F3C1-2	Chromic Acid Anodize Variation 3	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.78 .64
2F4C1-1 2F4C1-2	Chromic Acid Anodize Variation 4	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.62 .59
2F5C1-1 2F5C1-2	Chromic Acid Anodize Variation 5	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.71 .85
2F6C1-1 2F6C1-2	Chromic Acid Anodize Variation 6	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.84 .62
2F7C1-1 2F7C1-2	Chromic Acid Anodize Variation 7	Coupling Agent 1 + Epoxy Polyamide	As Prepared	Blew + .75
2F8C1-1 2F8C1-2	Chromic Acid Anodize Variation 8	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.40 Blew +
2F1C2-1 2F1C2-2	Chromic Acid Anodize Variation 1	Coupling Agent 1 + Epoxy Polyamide	As Prepared	2.81 2.65
2F2C2-1 2F2C2-2	Chromic Acid Anodize Variation 2	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.75 2.79
2F3C2-1 2F3C2-2	Chromic Acid Anodize Variation 3	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.85 3.03
2F4C2-1 2F4C2-2	Chromic Acid Anodize Variation 4	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.64 3.04

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVI

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F5C2-1 2F5C2-2	Chromic Acid Anodize Variation 5	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.39 2.61
2F6C2-1 2F6C2-2	Chromic Acid Anodize Variation 6	Coupling Agent 2 + Epoxy Polyamide	As Prepared	3.14 3.06
2F7C2-1 2F7C2-2	Chromic Acid Anodize Variation 7	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.78 2.14
2F8C2-1 2F8C2-2	Chromic Acid Anodize Variation 8	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.12 2.84
2F1C3-1 2F1C3-2	Chromic Acid Anodize Variation 1	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.20 1.40
2F2C3-1 2F2C3-2	Chromic Acid Anodize Variation 2	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.10 1.27
2F3C3-1 2F3C3-2	Chromic Acid Anodize Variation 3	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.20 .88
2F4C3-1 2F4C3-2	Chromic Acid Anodize Variation 4	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.97 .88
2F5C3-1 2F5C3-2	Chromic Acid Anodize Variation 5	Coupling Agent 3 + Epoxy Polyamide	As Prepared	Blew + 3.03
2F6C3-1 2F6C3-2	Chromic Acid Anodize Variation 6	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.32 .79
2F7C3-1 2F7C3-2	Chromic Acid Anodize Variation 7	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.15 1.70
2F8C3-1 2F8C3-2	Chromic Acid Anodize Variation 8	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.23 1.16

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVI

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1C4-1	Chromic Acid Anodize Variation 1	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.80
2F1C4-2				2.77
2F2C4-1	Chromic Acid Anodize Variation 2	Coupling Agent 4 + Epoxy Polyamide]	As Prepared	2.49
2F2C4-2				2.92
2F3C4-1	Chromic Acid Anodize Variation 3	Coupling Agent 4 + Epoxy Polyamide]	As Prepared	3.11
2F3C4-2				3.02
2F4C4-1	Chromic Acid Anodize Variation 4	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.77
2F4C4-2				2.89
2F5C4-1	Crhomic Acid Anodize Variation 5	Coupling Agent 4 + Epoxy Polyamide	As Prepared	3.11
2F5C4-2				2.69
2F6C4-1	Chromic Acid Anodize Variation 6	Coupling Agent 4 + Epoxy Polyamide	As Prepared	3.00
2F6C4-2				2.81
2F7C4-1	Chromic Acid Anodize Variation 7	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.64+
2F7C4-2				3.01
2F8C4-1	Chromic Acid Anodize Variation 8	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.74
2F8C4-2				2.91
2F1C5-1	Chromic Acid Anodize Variation 1	Coupling Agent 5 + Epoxy Polyamide	As Prepared	3.01
2F1C5-2				2.99
2F2C5-1	Chromic Acid Anodize Variation 2	Coupling Agent 5 + Epoxy Polyamide	As Prepared	3.06
2F2C5-2				2.97
2F3C5-1	Chromic Acid Anodize Variation 3	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.99
2F3C5-2				3.08
2F4C5-1	Chromic Acid Anodize Variation 4	Coupling Agent 5 + Epoxy Polyamide	As Prepared	3.14
2F4C5-2				3.36

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVI

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F5C5-1 2F5C5-2	Chromic Acid Anodize Variation 5	Coupling Agent 5 + Epoxy Polyamide	As Prepared	3.32 4.08
2F6C5-1 2F6C5-2	Chromic Acid Anodize Variation 6	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.84 2.70
2F7C5-1 2F7C5-2	Chromic Acid Anodize Variation 7	Coupling Agent 5 + Epoxy Polyamide	As Prepared	3.33 3.21
2F8C5-1 2F8C5-2	Chromic Acid Anodize Variation 8	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.82 2.96

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVII

Evaluation of Coupling Agent Applied to Surface of Aluminum Alloy (2024-T3)
Prior to Coating with Epoxy Polyamide Primer for Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.81
2F1CX-2	Variation 1	Epoxy Polyamide	Water Exposure	2.10
2F2CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	----+
2F2CX-2	Variation 2	Epoxy Polyamide	Water Exposure	1.01
2F3CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt 1.	1.95
2F3CX-2	Variation 3	Epoxy Polyamide	Water Exposure	1.88
2F4CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.80
2F4CX-2	Variation 4	Epoxy Polyamide	Water Exposure	1.96
2F5CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.45
2F5CX-2	Variation 5	Epoxy Polyamide	Water Exposure	1.39
2F6CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.70
2F6CX-2	Variation 6	Epoxy Polyamide	Water Exposure	1.68
2F7CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.96
2F7CX-2	Variation 7	Epoxy Polyamide	Water Exposure	1.48
2F8CX-1	Chromic Acid Anodize	Coupling Agent 2 +	30 Day Salt	1.51
2F8CX-2	Variation 8	Epoxy Polyamide	Water Exposure	1.62

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* Adhesion surface energy density (in-lbs/in^2)
+ Examination during or after test indicated results not valid.

Table XXVIII lists the results obtained on specimens which were coated with one of five coupling agents mixed in epoxy polyamide primer after receiving one of the variations of acid anodizing. Two specimens of each processing variation were treated with one of the coupling agent in epoxy polyamide primer mixture prior to being coated with epoxy polyamide primer. All specimens were tested in "as prepared" condition.

The results obtained were reviewed and the coupling agent in epoxy polyamide mixture which yielded the best γ_a, adhesion surface energy density, was used to prepare duplicate specimens representing each anodizing processing variation for conditioning for 30 days in intermittent salt water exposure. The results of tests of these specimens are listed in Table XXIX.

The "built-flaw in the specimens did not always extend in a circular configuation on the specimens showing poor adhesion which could result in poor agreement between duplicates. Some specimens adhesion was so poor that the flaw or blister would extend to the side as soon as sufficient pressure was applied to release the adhesive backing of the built in flaw. These in effect "blew" to side of the specimen eliminating obtaining any data.

3.3.6 Evaluation of Polyurethane Elastomer - Table XXX lists the results obtained on specimens which were coated with polyurethane elastomer after receiving one of the variations of chromic acid anodizing and a prime coat of epoxy polyamide primer. Two specimens of each processing variation were evaluated in the "as prepared" condition and two after being subjected to 30 days intermittent exposure to salt water.

The polyurethane elastomer stretched severely during testing which with other materials had caused low values. With this material it was necessary to use a 200 psi diaphram whereas a 50 psig or a 100 psig diaphram had been used on all other materials. Even with the high pressure the polyurethaen failed by increases in blister diameter with good agreement on several failures on the same specimen and between duplicates.

3.3.7 Evaluation of Polysulfide Sealant - Table XXXI lists the results obtained on specimens which were coated with polysulfide sealant, chromate cure, after receiving one of the variations of chromic acid anodizing. Two specimens of each variation were tested in the "as prepared" condition.

The polysulfide sealants gave some problems in testing due to entrapped bubbles or voids. Most specimens which yielded low values could be visually examined and the flaw located. The stretching of the material by this test method caused some of the blisters with flaws in them to rupture instead of extending in a circular mode.

TABLE XXVIII

Evaluation of Coupling Agents Mixed in Epoxy Polyamide Primer Applied to Surface of Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1C1-3 2F1C1-4	Chromic Acid Anodize Variation 1	Coupling Agent 1 in Primer + Epoxy Polyamide	As Prepared	2.95 2.90
2F2C1-3 2F2C1-4	Chomic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.03 2.97
2F3C1-3 2F3C1-4	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.08 2.98
2F4C1-3 2F4C1-4	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.33 3.27
2F5C1-3 2F5C1-4	Chromic Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.34 3.31
2F6C1-3 2F6C1-4	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.46 2.97
2F7C1-3 2F7C1-4	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.97 3.21
2F8C1-3 2F8C1-4	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.83 3.10
2F1C2-3 2F1C2-4	Chromic Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.51 2.70

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVIII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_2*
2F2C2-3 2F2C2-4	Chromic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.87 2.93
2F3C2-3 2F3C2-4	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.80 2.85
2F4C2-3 2F4C2-4	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.27 3.31
2F5C2-3 2F5C2-4	Chromic Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.11 3.14
2F6C2-3 2F6C2-4	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.11 3.08
2F7C2-3 2F7C2-4	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.91 3.28
2F8C2-3 2F8C2-4	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.08 2.90
2F1C3-3 2F1C3-4	Chromic Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.96 3.00
2F2C3-3 2F2C3-4	Chromic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.05 2.50

* Adhesion surface energy density (in-lbs/in^2)

\+ Delamination during or after test indicated results not valid.

TABLE XXVIII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ a*
2F3C3-3 2F3C3-4	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.74 3.28
2F4C3-3 2F4C3-4	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.65 3.03
2F5C3-3 2F5C3-4	Chromic Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.32 2.96
2F6C3-3 2F6C3-4	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.46 3.37
2F7C3-3 2F7C3-4	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.41 3.49
2F8C3-3 2F8C3-4	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.86 3.24
2F1C4-3 2F1C4-4	Chromic Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.19 3.23
2F2C4-3 2F2C4-4	Chromic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.62 3.06
2F3C4-3 2F3C4-4	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.24 3.03

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVIII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F4C4-3 2F4C4-4	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.28 3.49
2F5C4-3 2F5C4-4	Chromic AcidAnodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.10 3.06
2F6C4-3 2F6C4-4	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.34 3.42
2F7C4-3 2F7C4-4	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.06 3.38
2F8C4-3 2F8C4-4	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.17 3.10
2F1C5-3 2F1C5-4	Chromic Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.17 2.19
2F2C5-3 2F2C5-4	Chromic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.23 2.19
2F3C5-3 2F3C5-4	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.30 2.31
2F4C5-3 2F4C5-4	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.69 2.30

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXVIII

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F5C5-3	Chromic Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.79
2F5C5-4				2.39
2F6C5-3	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	2.91
2F6C5-4				2.54
2F7C5-3	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	As Prepared	3.32
2F7C5-4				3.07
2F8C5-3	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy	As Prepared	2.14
2F8C5-4				2.18

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXIX

Evaluation of Coupling Agent Mixed in Epoxy Polyamide Primer Applied to Surface of Aluminum Alloy (2024-T3) for Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1CY-1 2F1CY-2	Chromic Acid Anodize Variation 1	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.97 1.86
2F2CY-1 2F2CY-2	Chromic Acid Anodize Variation 2	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.72 1.20+
2F3CY-1 2F3CY-2	Chromic Acid Anodize Variation 3	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.98 1.93
2F4CY-1 2F4CY-2	Chromic Acid Anodize Variation 4	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.58 1.24+
2F5CY-1 2F5CY-2	Chromic Acid Anodize Variation 5	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.52 1.88
2F6CY-1 2F6CY-2	Chromic Acid Anodize Variation 6	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.42 1.83
2F7CY-1 2F7CY-2	Chromic Acid Anodize Variation 7	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.35 1.41
2F8CY-1 2F8CY-2	Chromic Acid Anodize Variation 8	Coupling Agent in Primer + Epoxy Polyamide	30 Day Salt Water Exposure	1.26+ 2.11

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXX

Evaluation of Polyurethane Elastomer Coating over Epoxy Polyamide Primer on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1PE-1 2F1PE-2	Chromic Acid Anodize Variation 1	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	14.0 13.6
2F1PE-3 2F1PE-4			30 Day Salt Water Exposure	10.3 8.0
2F2PE-1 2F2PE-2	Chromic Acid Anodize Variation 2	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	12.3 11.5
2F2PE-3 2F2PE-4			30 Day Salt Water Exposure	13.3 13.2
2F3PE-1 2F3PE-2	Chromic Acid Anodize Variation 3	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	13.5 14.3
2F3PE-3 2F3PE-4			30 Day Salt Water Exposure	10.8 11.0
2F4PE-1 2F4PE-2	Chromic Acid Anodize Variation 4	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	12.6 14.0
2F4PE-3 2F4PE-4			30 Day Salt Water Exposure	9.6 13.0
2F5PE-1 2F5PE-2	Chromic AcidAnodize Variation 5	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	11.7 11.7
2F5PE-3 2F5PE-4			30 Day Salt Water Exposure	10.8 9.3
2F6PE-1 2F6PE-2	Chromic Acid Anodize Variation 6]	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	11.4 12.8
2F6PE-3 2F6PE-4			30 Day Salt Water Exposure	10.8 12.4

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F7PE-1 2F7PE-2	Chromic Acid Anodize Variation 7	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	----+ 8.0
2F7PE-3 2F7PE-4			30 Day Salt Water Exposure	11.8 11.0
2F8PE-1 2F8PE-2	Chromic Acid Anodize Variation 8	Epoxy Polyamide Primer + Polyurethane Elastomer	As Prepared	10.8 11.6
2F8PE-3 2F8PE-4			30 Day Salt Water Exposure	12.4 10.8

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXI

Evaluation of Polysulfide Sealant on Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
2F1S-1	Chromic Acid Anodize Variation 1	Sealant, Chromate Cure	As Prepared	1.58
2F1S-2				3.60
2F2S-1	Chromic Acid Anodize Variation 2	Sealant, Chromate Cure	As Prepared	1.62
2F2S-2				1.76
2F3S-1	Chromic Acid Anodize Variation 3	Sealant, Chromate Cure	As Prepared	4.01
2F3S-2				----+
2F4S-1	Chromic Acid Anodize Variation 4	Sealant, Chromate Cure	As Prepared	3.24
2F4S-2				4.13
2F5S-1	Chromic Acid Anodize Variation 5	Sealant, Chromate Cure	As Prepared	2.92+
2F5S-2				3.81
2F6S-1	Chromic Acid Anodize Variation 6	Sealant, Chromate Cure	As Prepared	3.56
2F6S-2				4.02
2F7S-1	Chromic Acid Anodize Variation 7	Sealant, Chromate Cure	As Prepared	3.81
2F7S-2				3.73
2F8S-1	Chromic Acid Anodize Variation 8	Sealant, Chromate Cure	As Prepared	1.64
2F8S-2				----+

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

3.4 RESULTS OF PROCESSING STUDIES WITH STEEL 4340

3.4.1 Evaluation of Epoxy Polyamide Primer Coated Specimens - Table XXXII lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the phosphate chemical conversion treatment variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent salt water exposure. Some of the specimens cracked in the built-in flaw area and in other instances the blister would grow to one side and not produce a circular growth. Both of these problems ended in low results which should be eliminated from consideration.

3.4.2 Evaluation of Polyurethane Enamel Over Epoxy Polyamide Primer Specimens - Table XXXIII lists the results obtained on specimens which were coated with epoxy polyamide primer then polyurethane enamel after receiving one of the phosphate chemical conversion treatment variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent salt water exposure.

Visual examination of these specimens found no cracks but some flaws extended in non-circular configurations which yielded poor data.

3.4.3 Evaluation of Coupling Agents - Table XXXIV lists the results obtained on specimens which were coated with one of 5 coupling agents after receiving one of the phosphate chemical conversion treatment variation processes. Two specimens of each processing variation were treated with one of the coupling agents prior to being coated with epoxy polyamide primer. All specimens were tested in the "as prepared" condition.

The results obtained were reviewed and the coupling agent which yielded the best Ya, adhesion surface energy density, was used to prepare duplicate specimens representing each phosphate chemical conversion treatment variation. These were conditioned for 30 days with intermittent exposure to salt water. The results are listed in Table XXXV.

The coupling agents showed additional problems in obtaining meaningful data. Some of the material acted as parting agents and no bond was obtained between the treated surface and the epoxy polyamide primers. Very low results were obtained on other specimens due to the non-adherence of the primer to the treated surface.

3.4.4 Evaluation of Polysulfide Sealant - Table XXXVI lists the results obtained on specimens which were coated with polysulfide sealant, chromate cure, after receiving one of the variations of phosphate chemical conversion treatment. Two specimens of each variation were tested in the "as prepared" condition.

TABLE XXXII

Evaluation of Epoxy Polyamide Primer on Steel (4340)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
4F1P-1 4F1P-2	Phosphate Conversion Coating Variation #1	Epoxy Polyamide Primer	As Prepared	1.99 1.69
4F1P-3 4F1P-4	Phosphate Conversion Coating Variation #1	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.33 1.35
4F2P-1 4F2P-2	Phosphate Conversion Coating Variation #2	Epoxy Polyamide Primer	As Prepared	1.96 1.85
4F2P-3 4F2P-4	Phosphate Conversion Coating Variation #2	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.49 1.48
4F3P-1 4F3P-2	Phosphate Conversion Coating Variation #3	Epoxy Polyamide Primer	As Prepared	1.17 + 1.88
4F3P-3 4F3P-4	Phosphate Conversion Coating Variation #3	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.77 .92+
4F4P-1 4F4P-2	Phosphate Conversion Coating Variation #4	Epoxy Polyamide Primer	As Prepared	1.49 1.48
4F4P-3 4F4P-4	Phosphate Conversion Coating Variation #4	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.45 1.23

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIII

Evaluation of Polyurethane Enamel over Epoxy Polyamide Primer on Steel (4340)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
4F1PU-1 4F1PU-2	Phosphate Conversion Coating Variation #1	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	1.71 1.53
4F1PU-3 4F1PU-4	Phosphate Conversion Coating Variation #1		30 Day Salt Water Exposure	2.18 2.24
4F2PU-1 4F2PU-2	Phosphate Conversion Coating Variation #2	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	.71+ .53+
4F2PU-3 4F2PU-4	Phosphate Conversion Coating Variation #2		30 Day Salt Water Exposure	1.26 1.32
4F3PU-1 4F3PU-2	Phosphate Conversion Coating Variation # 3	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	1.33 1.35
4F3PU-3 4F3PU-4	Phosphate Conversion Coating Variation #3		30 Day Salt Water Exposure	2.29 1.95
4F4PU-1 4F4PU-2	Phosphate Conversion Coating Variation #4	Epoxy Polyamide Primer + Polyurethane Enamel	As Prepared	1.37 1.47
4F4PU-3 4F4PU-4	Phosphate Conversion Coating Variation #4		30 Day Salt Water Exposure	1.74 1.93

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIV

Evaluation of Coupling Agents Applied to Surfaces of Steel (4340) Prior to Coating with Epoxy Polyamide Primer

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	Υ_a*
4F1PC1-1 4F1PC1-2	Phosphate Conversion Coating Variation #1	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.62 1.64
4F2PC1-1 4F2PC1-2	Phosphate Conversion Coating Variation #2	Coupling Agent 1 +EEpoxy Polyamide	As Prepared	.55 .31
4F3PC1-1 4F3PC1-2	Phosphate Conversion Coating Variation #3	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.59 .35
4F4PC1-1 4F4PC1-2	Phosphate Conversion Coating Variation #4	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.18 .37
4F1PC2-1 4F1PC2-2	Phosphate Conversion Coating Variation #1	Coupling Agent 2 + Epoxy Polyamide	As Prepared	3.04 2.64
4F2PC2-1 4F2PC2-2	Phosphate Conversion Coating Variation #2	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.13 1.53
4F3PC2-1 4F3PC2-2	Phosphate Conversion Coating Variation #3	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.20 1.39
4F4PC2-1 4F4PC2-2	Phosphate Conversion Coating Variation #4	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.90 2.20
4F1PC3-1 4F1PC3-2	Phosphate Conversion Coating Variation #1	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.41 ----+
4F2PC3-1 4F2PC3-2	Phosphate Conversion Coating Variation #2	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.78 .78+
4F3PC3-1 4F3PC3-2	Phosphate Conversion Coating Variation #3	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.24 .41+
4F4PC3-1 4F4PC3-2	Phosphate Conversion Coating Variation #4	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.94 2.03

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIV

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
4F1PC4-1 4F1PC4-2	Phosphate Conversion Coating Variation #1	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.25 2.85
4F2PC4-1 4F2PC4-2	Phosphate Conversion Coating Variation #2	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.65 1.57
4F3PC4-1 4F3PC4-2	Phosphate Conversion Coating Variation #3	Coupling Agent 4 + Epoxy Polyamide	As Prepared	.78+ 1.87
4F4PC4-1 4F4PC4-2	Phosphate Conversion Coating Variation #4	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.54 1.38
4F1PC5-1 4F1PC5-2	Phosphate Conversion Coating Variation #1	Coupling Agent 5 + Epoxy Polyamide	As Prepared	.92 1.52
4F2PC5-1 4F2PC5-2	Phosphate Conversion Coating Variation #2	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.33 1.53
4F3PC5-1 4F3PC5-2	Phosphate Conversion Coating Variation #3	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.27 1.61
4F4PC5-1 4F4PC5-2	Phosphate Conversion Coating Variation #4	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.68 1.61

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXV

Evaluation of Coupling Agent Applied to Surface of Steel (4340) Prior to Coating with Epoxy Polyamide Primer for Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
4F1PCX-1	Phosphate Conversion Coating Variation #1	Coupling Agent 5 + Epoxy Polyamide	30 Day Salt Water Exposure	1.28
4F2PCX-1	Phosphate Conversion Coating Variation #2	Coupling Agent 5 + Epoxy Polyamide	30 Day Salt Water Exposure	1.25
4F3PCX-1	Phosphate Conversion Coating Variation #3	Coupling Agent 5 + Epoxy Polyamide	30 Day Salt Water Exposure	1.40
4F4PCX-1	Phosphate Conversion Coating Variation #4	Coupling Agent 5 + Epoxy Polyamide	30 Day Salt Water Exposure	1.42

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXVI

Evaluation of Polysulfide Sealant on Steel (4340)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
4F1S-1	Phosphate Conversion Coating Variation #1	Polysulfide Sealant Chromate Cure	As Prepared	1.07
4F1S-2	Phosphate Conversion Coating Variation #1	Polysulfide Sealant Chromate Cure	As Prepared	1.01
4F2S-1	Phosphate Conversion Coating Variation #2	Polysulfide Sealant Chromate Cure	As Prepared	1.41
4F2S-2	Phosphate Conversion Coating Variation #2	Polysulfide Sealant Chromate Cure	As Prepared	1.41
4F3S-1	Phosphate Conversion Coating Variation #3	Polysulfide Sealant Chromate Cure	As Prepared	1.49
4F3S-2	Phosphate Conversion Coating Variation #3	Polysulfide Sealant Chromate Cure	As Prepared	1.50
4F4S-1	Phosphate Conversion Coating Variation #4	Polysulfide Sealant Chromate Cure	As Prepared	1.30
4F4S-2	Phosphate Conversion Coating Variation #4	Polysulfide Sealant Chromate Cure	As Prepared	1.50

* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

3.5 RESULTS OF PROCESSING STUDIES WITH CADMIUM PLATED STEEL (4340)

3.5.1 Evaluation of Epoxy Polyamide Primer Coated Specimens - Table XXXVII lists the results obtained on specimens which were coated with epoxy polyamide primer after receiving one of the chromate or phosphate chemical conversion treatment variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent salt water exposure. Some of the specimens cracked in the built-in flaw area and in other instances the blister would grow to one side and not produce a circular growth. Both of these problems ended in low results which should be eliminated from consideration.

3.5.2 Evaluation of Polyurethane Enamel Over Epoxy Polyamide Primer Specimens - Table XXXVIII lists the results obtained on specimens which were coated with epoxy polyamide primer then polyurethane enamel after receiving one of the chromate or phosphate chemical conversion treatment variation processes. Two specimens of each variation were tested in the "as prepared" condition and two after being subjected to 30 day intermittent salt water exposure. Visual examination during blister test evaluation noted that some of the "built-in" flaws would extend to the side instead of in a circular mode. Other specimens would stretch and rupture through the coating so that meaningful data could not be obtained.

3.5.3 Evaluation of Coupling Agents - Table XXXIX lists the results obtained on specimens which were coated with one of 5 coupling agents after receiving one of the chromate or phosphate chemical conversion treatment variation processes. Two specimens of each processing variation were treated with one of the coupling agents prior to being coated with epoxy polyamide primer. All specimens were tested in the "as prepared" condition.

The results obtained were reviewed and the coupling agent which yielded the best Ya, adhesion surface energy density, was used to prepare duplicate specimens representing each of the chromate or phosphate chemical conversion treatment variation. These were conditioned for 30 days with intermittent exposure to salt water. The results are listed in Table XXXX.

The coupling agents showed additional problems in obtaining meaningful data. Some of the material acted as parting agents and no bond was obtained between the treated surface and the epoxy polyamide primers. Very low results were obtained on other specimens due to the non-adherence of the primer to the treated surface.

3.5.4 Evaluation of Polysulfide Sealant - Table XXXXI lists the results obtained on specimens which were coated with polysulfide sealant, chromate cure, after receiving one of the variations of the chromate or phosphate chemical conversion treatment. Two specimens of each variation were tested in the "as prepared" condition.

View Graph
#12

TABLE XXXVII (TYPICAL)
~~TABLE XXXVII~~

Evaluation of Epoxy Polyamide Primer on Cadmium Plated Steel (4340)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF1P-1 CF1P-2	Chromate Conversion Coating Variation #1	Epoxy Polyamide Primer	As Prepared	2.07 1.86
CF1P-3 CF1P-4	Chromate Conversion Coating Variation #1	Epoxy Polyamide Primer	30 Day Salt Water Exposure	.83+ 1.34
CF2P-1 CF2P-2	Chromate Conversion Coating Variation #2	Epoxy Polyamide Primer	As Prepared	1.81 1.75
CF2P-3 CF2P-4	Chromate Conversion Coating Variation #2	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.30 ----+
CF3P-1 CF3P-2	Chromate Conversion Coating Variation #3	Epoxy Polyamide Primer	As Prepared	1.24 1.67
CF3P-3 CF3P-4	Chromate Conversion Coating Variation #3	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.15+ 1.01+
CF4P-1 CF4P-2	Chromate Conversion Coating Variation #4	Epoxy Polyamide Primer	As Prepared	1.61 2.07
CF4P-3 CF4P-4	Chromate Conversion Coating Variation #4	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.80 1.19+
CF5P-1 CF5P-2	Phosphate Conversion Coating Variation #5	Epoxy Polyamide Primer	As Prepared	1.59 1.80
CF5P-3 CF5P-4	Phosphate Conversion Coating Variation #5	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.15 1.67
CF6P-1 CF6P-2	Phosphate Conversion Coating Variation #6	Epoxy Polyamide Primer	As Prepared	1.80 1.53
CF6P-3 CF6P-4	Phosphate Conversion Coating Variation #6	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.65 1.46

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* Adhesion surface energy density (in-lbs/in^2)

TABLE XXXVII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF7P-1	Phosphate Conversion Coating Variation #7	Epoxy Polyamide Primer	As Prepared	.88
CF7P-2				1.37
CF7P-3	Phosphate Conversion Coating Variation #7	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.54
CF7P-4				1.37
CF8P-1	Phosphate Conversion Coating Variation #8	Epoxy Polyamide Primer	As Prepared	1.57
CF8P-2				1.65
CF8P-3	Phosphate Conversion Coating Variation #8	Epoxy Polyamide Primer	30 Day Salt Water Exposure	1.35
CF8P-4				.98

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* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

TABLE XXXVIII

Evaluation of Polyurethane Enamel Over Epoxy Polyamide Primer On Aluminum Alloy (2024-T3)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF1PU-1 CF1PU-2	Chromate Conversion Coating Variation #1	Epoxy Polyamide & Polyurethane Enamel	As Prepared	2.33 1.68
CF1PU-3 CF1PU-4	Chromate Conversion Coating Variation #1	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	2.36 1.26+
CF2PU-1 CF2PU-2	Chromate Conversion Coating Variation #2	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.79 1.59
CF2PU-3 CF2PU-4	Chromate Conversion Coating Variation #2	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	.94 .76
CF3PU-1 CF3PU-2	Chromate Conversion Coating Variation #3	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.95 1.94
CF3PU-3 CF3PU-4	Chromate Conversion Coating Variation #3	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	2.09 1.69
CF4PU-1 CF4PU-2	Chromate Conversion Coating Variation #4	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.59 1.69
CF4PU-3 CF4PU-4	Chromate Conversion Coating Variation #4	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	2.36 2.03
CF5PU-1 CF5PU-2	Phosphate Conversion Coating Variation #5	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.89 1.83

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid

TABLE XXXVIII
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF5PU-3 CF5PU-4	Phosphate Conversion Coating Variation #5	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	2.34 2.12
CF6PU-1 CF6PU-2	Phosphate Conversion Coating Variation #6	Epoxy Polyamide & Polyurethane Enamel	As Prepared	2.09 2.13
CF6PU-3 CF6PU-4	Phosphate Conversion Coating Variation #6	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	2.39 1.04+
CF7PU-1 CF7PU-2	Phosphate Conversion Coating Variation #7	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.74 1.84
CF7PU-3 CF7PU-4	Phosphate Conversion Coating Variation #7	Epoxy Polyamide & Polyurethane Enamel	30 Day Salt Water Exposure	----+ 1.87
CF8PU-1 CF8PU-2	Phosphate Conversion Coating Variation #8	Epoxy Polyamide & Polyurethane Enamel	As Prepared	1.57 1.64
CF8PU-3 CF8PU-4	Phosphate Conversion Coating Variation #8	Epoxy Polyamide & Polyurethane	30 Day Salt Water Exposure	1.68 1.89

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* Adhesion surface energy density (in-lbs/in^2)
\+ Examination during or after test indicated results not valid.

TABLE XXXIX

Evaluation of Coupling Agents Applied to Surfaces of Cadmium Plated Steel (4340) Prior to Coating with Epoxy Polyamide Primer

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF1PC1-1	Chromate Conversion Coating Variation #1	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.05
CF1PC1-2	Chromate Conversion Coating Variation #1	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.08
CF2PC1-1	Chromate Conversion Coating Variation #2	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.72
CF2PC1-2	Chromate Conversion Coating Variation #2	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.80
CF3PC1-1	Chromate Conversion Coating Variation #3	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.24
CF3PC1-2	Chromate Conversion Coating Variation #3	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.64
CF4PC1-1	Chromate Conversion Coating Variation #4	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.56
CF4PC1-2	Chromate Conversion Coating Variation #4	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.93
CF5PC1-1	Phosphate Conversion Coating Variation #5	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.40
CF5PC1-2	Phosphate Conversion Coating Variation #5	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.68
CF6PC1-1	Phosphate Conversion Coating Variation #6	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.82
CF6PC1-2	Phosphate Conversion Coating Variation #6	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.36

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF7PC1-1	Phosphate Conversion Coating Variation #7	Coupling Agent 1 + Epoxy Polyamide	As Prepared	3.17
CF7PC1-2	Phosphate Conversion Coating Variation #7	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.31
CF8PC1-1	Phosphate Conversion Coating Variation #8	Coupling Agent 1 + Epoxy Polyamide	As Prepared	1.01
CF8PC1-2	Phosphate Conversion Coating Variation #8	Coupling Agent 1 + Epoxy Polyamide	As Prepared	.92
CF1PC2-1	Chromate Conversion Coating Variation #1	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.99
CF1PC2-2	Chromate Conversion Coating Variation #1	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.94
CF2PC2-1	Chromate Conversion Coating Variation #2	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.75
CF2PC2-2	Chromate Conversion Coating Variation #2	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.78
CF3PC2-1	Chromate Conversion Coating Variation #3	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.81
CF3PC2-2	Chromate Conversion Coating Variation #3	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.34
CF4PC2-1	Chromate Conversion Coating Variation #4	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.03
CF4PC2-2	Chromate Conversion Coating Variation #4	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.98

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF5PC2-1	Phosphate Conversion Coating Variation #5	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.39
CF5PC2-2	Phosphate Conversion Coating Variation #5	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.59
CF6PC2-1	Phosphate Conversion Coating Variation #6	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.22
CF6PC2-2	Phosphate Conversion Coating Variation #6	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.72
CF7PC2-1	Phosphate Conversion Coating Variation #7	Coupling Agent 2 + Epoxy Polyamide	As Prepared	2.28
CF7PC2-2	Phosphate Conversion Coating Variation #7	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.67
CF8PC2-1	Phosphate Conversion Coating Variation #8	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.80
CF8PC2-2	Phosphate Conversion Coating Variation #8	Coupling Agent 2 + Epoxy Polyamide	As Prepared	1.66
CF1PC3-1	Chromate Conversion Coating Variation #1	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.51+
CF1PC3-2	Chromate Conversion Coating Variation #1	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.80
CF2PC3-1	Chromate Conversion Coating Variation #2	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.92+
CF2PC3-2	Chromate Conversion Coating Variation #2	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.88+

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX
(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF3PC3-1	Chromate Conversion Coating Variation #3	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.86
CF3PC3-2	Chromate Conversion Coating Variation #3	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.05
CF4PC3-1	Chromate Conversion Coating Variation #4	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.83
CF4PC3-2	Chromate Conversion Coating Variation #4	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.44
CF5PC3-1	Phosphate Conversion Coating Variation #5	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.59
CF5PC3-2	Phosphate Conversion Coating Variation #5	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.20
CF6PC3-1	Phosphate Conversion Coating Variation #6	Coupling Agent 3 + Epoxy Polyamide	As Prepared	2.45
CF6PC3-2	Phosphate Conversion Coating Variation #6	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.78
CF7PC3-1	Phosphate Conversion Coating Variation #7	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.53
CF7PC3-2	Phosphate Conversion Coating Variation #7	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.90
CF8PC3-1	Phosphate Conversion Coating Variation #8	Coupling Agent 3 + Epoxy Polyamide	As Prepared	.89
CF8PC3-2	Phosphate Conversion Coating Variation #8	Coupling Agent 3 + Epoxy Polyamide	As Prepared	1.61

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_2*
CF1PC4-1	Chromate Conversion Coating Variation #1	Coupling Agent 4 + Epoxy Polyamide	As Prepared	.99+
CF1PC4-2	Chromate Conversion Coating Variation #1	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.94
CF2PC4-1	Chromate Conversion Coating Variation #2	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.25
CF2PC4-2	Chromate Conversion Coating Variation #2	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.16
CF3PC4-1	Chromate Conversion Coating Variation #3	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.90
CF3PC4-2	Chromate Conversion Coating Variation #3	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.14
CF4PC4-1	Chromate Conversion Coating Variation #4	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.58
CF4PC4-2	Chromate Conversion Coating Variation #4	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.94
CF5PC4-1	Phosphate Conversion Coating Variation #5	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.80
CF5PC4-2	Phosphate Conversion Coating Variation #5	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.95
CF6PC4-1	Phosphate Conversion Coating Variation #6	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.48
CF6PC4-2	Phosphate Conversion Coating Variation #6	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.45

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF7PC4-1	Phosphate Conversion Coating Variation #7	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.94
CF7PC4-2	Phosphate Conversion Coating Variation #7	Coupling Agent 4 + Epoxy Polyamide	As Prepared	1.67
CF8PC4-1	Phosphate Conversion Coating Variation #8	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.02
CF8PC4-2	Phosphate Conversion Coating Variation #8	Coupling Agent 4 + Epoxy Polyamide	As Prepared	2.00
CF1PC5-1	Chromate Conversion Coating Variation #1	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.59
CF1PC5-2	Chromate Conversion Coating Variation #1	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.55
CF2PC5-1	Chromate Conversion Coating Variation #2	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.77
CF2PC5-2	Chromate Conversion Coating Variation #2	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.24
CF3PC5-1	Chromate Conversion Coating Variation #3	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.44
CF3PC5-2	Chromate Conversion Coating Variation #3	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.90
CF4PC5-1	Chromate Conversion Coating Variation #4	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.92
CF4PC5-2	Chromate Conversion Coating Variation #4	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.29

* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXIX

(Continued)

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF5PC5-1	Phosphate Conversion Coating Variation #5	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.78
CF5PC5-2	Phosphate Conversion Coating Variation #5	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.44
CF6PC5-1	Phosphate Conversion Coating Variation #6	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.48
CF6PC5-2	Phosphate Conversion Coating Variation #6	Coupling Agent 5 + Epoxy Polyamide	As Prepared	2.84
CF7PC5-1	Phosphate Conversion Coating Variation #7	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.77
CF7PC5-2	Phosphate Conversion Coating Variation #7	Coupling Agent 5 + Epoxy Polyamide	As Prepared	----+
CF8PC5-1	Phosphate Conversion Coating Variation #8	Coupling Agent 5 + Epoxy Polyamide	As Prepared	1.86
CF8PC5-2	Phosphate Conversion Coating Variation #8	Coupling Agent 5 +	As Prepared	3.14

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* Adhesion surface energy density (in-lbs/in^2)

+ Examination during or after test indicated results not valid.

TABLE XXXX

Evaluation of Coupling Agents Applied To Surfaces of Cadmium Plated Steel (4340) after Salt Water Exposure

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF1PCX-1	Chromate Conversion Coating Variation #1	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.53
CF2PCX-1	Chromate Conversion Coating Variation #2	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.32
CF3PCX-1	Chromate Conversion Coating Variation #3	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.35
CF4PCX-1	Chromate Conversion Coating Variation #4	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	.83
CF5PCX-1	Phosphate Conversion Coating Variation #5	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.47
CF6PCX-1	Phosphate Conversion Coating Variation #6	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	.96
CF7PCX-1	Phosphate Conversion Coating Variation #7	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.35
CF8PCX-1	Phosphate Conversion Coating Variation #8	Coupling Agent 4 + Epoxy Polyamide	30 Day Salt Water Exposure	1.13

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

TABLE XXXXI

Evaluation of Chromate Cure Polysulfide Sealant on Cadmium Plated Steel 4340

SPECIMEN NUMBER	SURFACE PREPARATION	COATING	CONDITION	γ_a*
CF1S-1	Chromate Conversion Coating Variation #1	Polysulfide Sealant Chromate Cure	As Prepared	1.08
CF2S-1	Chromate Conversion Coating Variation #2	Polysulfide Sealant Chromate Cure	As Prepared	1.05
CF3S-1	Chromate Conversion Coating Variation #3	Polysulfide Sealant Chromate Cure	As Prepared	1.17
CF4S-1	Chromate Conversion Coating Variation #4	Polysulfide Sealant Chromate Cure	As Prepared	1.07
CF5S-1	Phosphate Conversion Coating Variation #5	Polysulfide Sealant Chromate Cure	As Prepared	1.28
CF6S-1	Phosphate Conversion Coating Variation #6	Polysulfide Sealant Chromate Cure	As Prepared	1.27
CF7S-1	Phosphate Conversion Coating Variation #7	Polysulfide Sealant Chromate Cure	As Prepared	1.22
CF8S-1	Phosphate Conversion Coating Variation #8	Polysulfide Sealant Chromate Cure	As Prepared	1.14

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* Adhesion surface energy density (in-lbs/in^2)

\+ Examination during or after test indicated results not valid.

4.0 DISCUSSION OF RESULTS

4.1 BLISTER TEST METHOD

The blister test method as utilized on this program is a good method to obtain meaningful data. Good agreement was obtained between individual tests on the same specimens as long as the built-in-flaw increased in a circular mode and the membrane under stress did not rupture, crack or stretch excessively. When the membrane or blister would crack low results would be obtained. Excessive stretching also resulted in low results when the membrane stretched beyond the elastic limit of the material under test.

Duplicate specimens may not be sufficient on some types of materials as many specimens were invalid due to the failure modes discussed. Materials which are very brittle, such as the acrylic nitrocellulose lacquer, which are very brittle do not lend themselves to this type of evaluation as any flexing, such as the blister, cracks the surface.

The fluid flow rate is critical when repeated tests are to be made on the same specimen. Too high a flow rate will cause the blister to expand so rapidly so that the pressure can not be released fast enough to prevent a rupture. However, too low a flow rate yeilds curves difficult to analyze as the change in slope is very gradual. The diaphram used should be selected to be the lowest psig rating which will allow the test to be accomplished so that the best curve resolution will occur. In most cases on this program the 100 psig diaphram was utilized but when possible the 50 psig diaphram was used. Only in one instance was it necessary to use the 200 psig diaphram and that was with the VSD polyurethane elastomer.

Sample preparation causes some problems as it is difficult to build up sufficient coating thickness to prevent co-hesive failure of the coating material. A heavier build up coating to apply over the coating to be tested would be desirable provided it did not affect the overall results by either increasing or decreasing the measured adhesion surface energy density.

The preparation of the sealant specimens using the sealant coating fixture at first appeared to produce good specimens but during testing many were found to contain small air bubbles or voids. Many of the results obtained with sealant specimens are invalid due to rupture or excessive stretching in these areas. The air bubbles serve as additional flaws which lower the apparent adhesion much as notching does metal specimens. This fracture mechanics principal is the basis for the blister test and the adhesion strength will be detrimentally affected by the bubbles. Much of the data scatter has been reported to be caused by the presence of bubbles as a function of size and quantity in the fracture plane.

The cure of the coatings should be as near alike as possible when the surface preparation is being evaluated. For this reason specimens should be coated at the same time, conditioned together and tested the same day or as nearly so as possible. Humidity can affect some coating systems so specimens should be brought to some standard humidity prior to testing. The 50% relative humidity was chosen for this program as a base which can easily be obtained.

The X-Y recorder used on the program was very satisfactory as the ranges were easily adjusted and calibrated to allow maximum resolution both for pressure and blister deflection. Calibrations were found to hold from one day operation to the next with very little drift once a voltage regulator was installed in the system to prevent line voltage fluctuations.

4.2 TITANIUM ALLOY (Ti6Al4V)

The titanium alloy specimen testing proceeded well with no real problems until the evaluation of the coupling agents was begun. The coupling agents applied directly to the surface of some specimens seemed to act almost like parting agents. When the pressure was applied to lift the built-in flaw it exceeded the pressure required to fail the specimen. Great care had to be taken to prevent the blister extending to the edge of the specimen once started. Examination of the specimen found very little or no adhesion between the surface of the substrate and the coating membrane. A slight pull could remove the entire coating membrane from the specimen. This problem resulted in data scatter and invalid results.

When the coupling agent was mixed with the epoxy polyamide primer and applied to the surface higher adhesion surface energy densities were obtained. Some data scatter was noted and upon examination it was found that the epoxy polyamide primer used to build up to necessary coating thickness was not adhering well to the epoxy polyamide primer/coupling agent coating and could be peeled off. It appeared, when the low values were obtained, the blister had cohesively failed through the primer/coupling agent coating and the values obtained were the adhesion between the two systems.

4.3 ALUMINUM ALLOY (7075-T6)

All variations of the sulfuric acid anodize process resulted in acceptable adhesion values. The variations were selected to vary the process from the specification norm to slightly outside the limits in an attempt to find the causes that sometime affect the adhesion of coating to sulfuric acid anodized surfaces. With the epoxy polyamide primer the results would not really eliminate any of the variations tested as most fell within the data scatter and were above 2 in-lbs/in^2.

The lacquer specimens produced very scattered data and low results. Visual observations during testing and examination of the specimens after testing found the main problem was caused by the brittle nature of the coating material. Cracks would develop in the surface of the blister or deflected area sometimes causing low results and sometimes rupturing the coating. No interruption of the best application method or surface preparation method could be made due to the scattering of the data.

The results obtained on the polyurethane enamel over epoxy polyamide primer were much lower than anticipated. The data and specimens were reviewed but no explanation was reached which would explain the results. It was noted, however, that the results were not due to improper cure as over two months elapsed between coating and evaluation of some of the specimens. A comparison review of the 2024 data did not reveal the same problem although both alloys were coated at the same time with the same materials. The problem seems to be a compatibility problem of the polyurethane with the primer coated sulfuric acid anodized surface. This could either be a chemical reactive problem or solvency problem causing the epoxy polyamide primer to lift or release from the surface.

The use of wash primer did not appear to be effective in increasing the adhesion surface energy density of the epoxy polyamide primer.

The results obtained with the LTV-VSD formulated polyurethane elastomer were much higher than had been expected. Although the adhesion surface energy density value obtained may be high due to the elasticity of the material causing increased blister height the pressures required show the adhesion values should be high. Testing with the 100 psig diaphram in the system failed to cause failure in adhesion or cohesive failure of the material. A 200 psig diaphram was installed and pressures were increased to the 170 psig to 190 psig range before the specimen would fail in adhesion. It is thought the data scatter is due to the elasticity of the material but the values obtained are reasonable correct. The specimens were examined and the adhesion parting was between the metal surface and the epoxy polyamide primer as the primer remained on the polyurethane elastomer. This would indicate that the addition of the polyurethane elastomer increased the adhesion of the primer to the metal surfaces.

4.4 ALUMINUM ALLOY (2024-T3)

The variations in the chromic acid anodize process were selected to establish which parameters from the normal specification affected the adhesion of coatings. All the variations tested with epoxy polyamide primer were found to be satisfactory and showed good adhesion. The other coating did not show a significant variance between the eight variations to select any variation which would improve the adhesion characteristics of the presently used specification process.

The testing with the coupling agent mixed in the primer yielded less scatter although the adhesion surface energy density was not improved. Examination of the specimens still showed poor adhesion between the epoxy polyamide primer/coupling agent and the epoxy polyamide primer overcoat. The lack of as much scatter may have been the result in operator improved skill in testing of the specimens.

The remainder of the testing of specimens was normal with no additional problem encountered and except for the results obtained on the polyurethane elastomer over epoxy polyamide primer followed the same trends discussed under 4.2. The polyurethane enamel results although lower than obtained with epoxy polyamide primer appeared reasonable.

4.5 STEEL (4340)

The coated steel specimens yielded lower adhesion surface energy densities with epoxy polyamide primer than obtained on the aluminum alloys. No real differences were noted in the four variations evaluated. The variations were selected to determine the effects of brush coat versus immersion processing. The results would indicate that both are equally satisfactory when properly applied.

It was also noted that the adhesion surface energy density for polysulfide sealant and polyurethane enamel over epoxy polyamide primer were of the same magnitude as with only the primer.

4.6 CADMIUM PLATED STEEL (4340)

No significant differences were found between the chromate and phosphate chemical conversion treatments except that variation 4 (hotter of the chromate chemical conversion immersion treatments) yielded slightly improved adhesion. This was true both with the epoxy polyamide primer coated specimens and the polyurethane enamel specimens over epoxy polyamide primer.

5.0 CONCLUSIONS

GENERAL

- The "Blister Test Method" was shown to be another valuable method to evaluate coating materials adhesion to substrates. The duplication of results indicate the method is valid for selection of surface treatments for coating systems.
- Some coating systems are much more "forgiving" than others as to surface treatments so that minor variances in processing do not seriously affect the adhesion characteristics.
- The coupling agents selected for evaluation were not not effective when applied directly to the surface of the substrate. When the coupling agent was mixed with the epoxy polyamide primer prior to application some improvement in adhesion was noted; however, primer applied over the coupling agent in primer treated surface showed very poor adhesion.
- Adhesion values of epoxy polyamide primer were altered, both higher and lower when coatings were applied to epoxy polyamide primed surfaces. This would indicate that the coating material affects the primer adhesion.

TITANIUM ALLOY (Ti6Al4V)

- The fluosilicic acid/aluminum oxide abrasion surface treatment was found to be superior to the chromate solution/abrasive nylon pad abrasion treatment.
- Variation 2 of the fluosilicic acid/aluminum oxide process was selected as the best based on equivalent results and economics of processing.
- Variation 1 of the chromate solution/abrasive nylon pad processes was selected as the best based on equivalent results and economics of processing.
- The chromate cure polysulfide sealant yielded overall higher adhesion than the manganese cure sealant.

ALUMINUM ALLOY (7075-T6)

- Sulfuric acid anodize variations selected for evaluation did not indicate any significant differences with epoxy polyamide primer prepared surfaces. The VSD Specification treatment (variation 2) yielded as high an adhesion as was obtained.

- The polyurethane elastomer coating over epoxy polyamide primer yielded much higher adhesion than the primer alone.

- The polyurethane enamel coating over epoxy polyamide primer on a sulfuric acid anodized surface significantly lowered the adhesion.

ALUMINUM ALLOY (2024-T3)

- No significant difference was found on the variation in chromic acid anodizing processing variation selected when epoxy polyamide primer was used.
- Wash primer on the aluminum alloy significantly lowered the adhesion with epoxy polyamide primer.
- The polyurethane applied over the epoxy polyamide primer showed significant increase in adhesion.

STEEL (4340)

- No significant difference was found in the variations selected for the phosphate chemical conversion treatment. Brush treatment was just as satisfactory as immersion treatment when properly applied.

CADMIUM PLATED STEEL (4340)

- Brush treatment of the phosphate or chromate chemical conversion coating treatment appeared to yield equally good results when properly applied. The hotter chromate chemical conversion immersion treatment (variation 4) yielded slightly higher results.

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MATERIAL CODE DESCRIPTION

Code No.	
1.	Epoxy Polyamide Primer (MIL-P-23377)
2.	Polyurethane Enamel (MIL-P-081773)
3.	Acrylic Nitrocellulose Lacquer (MIL-L-081773)
4.	Sealant Polysulfide (Chromate Cure) (MIL-S-8802)
5.	Sealant Polysulfide (Manganese Cure) (MIL-S-8802)
6.	Neoprene Rain Erosion Coating (MIL-C-27315)
7.	Polyurethane Elastomer (LTV-VSD-207-9-427)
8.	Chromate Solution, (MIL-C-5541) Alodine 1200S, Amchem Products Inc.
9.	Abrasive Nylon Pads, General Purpose, part # 7447, and Ultrafine, part # 4448, Scotch-Brite, 3M Company
10.	Pressure sensitive paint masking disks, Goodman Paper Products.
11.	Trimethoxysilylpropethylenediamine, Z6020 Silane Dow Corning Corporation, Midland, Michigan
12.	Trimethoxysilylpropethyleneepoxide, Z6040 Silane Dow Corning Corporation, Midland Michigan
13.	Vinyltriethoxysilane, SC3933, General Electric Silicone Products Department, Waterford, New York
14.	Vinyltris (2-methoxy-ethoxy) silane, SC3735, General Electric, Silicone Products Department, Waterford, New York
15.	Hexamethyldisilane, Z6079 Silane, Dow Corning Corporation, Midland, Michigan
16.	Phosphate Treatment, Concentrate, Granodine #50 Amchem Products, Inc. Amper Penn.
17.	Phosphate Treatment, Concentrate, Bonderite 32 Parker Rust Proof Company
18.	Chromate Treatment Concentrate, Macrobronze #4 Mac Dermid, Inc., Waterbury, Conn.
19.	Phosphate Treatment Concentrate, Lithoform #2, Amchem Products, Inc.

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