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NON-CHROMATED SURFACE PRETREATMENTS FOR ALUMINUM

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Chromates, particularly Chromium VI, have been widely used in aerospace processes and materials due to their excellent performance as corrosion inhibitors. These processes range from inorganic pretreatment processes (alkaline cleaners, deoxidizers, conversion coatings, etc.) to organic protective coatings (primers, sealants, fuel tank coatings, etc.). Recently, regulatory agencies have enacted legislation to limit or prohibit the use and disposal of chromium containing materials because Chromium VI is now a known carcinogen. These rules affect the majority of the pretreatment processes used on naval aircraft, necessitating the development of non-chromated replacements for the current materials. After an extensive research and development effort, several promising alternatives to chromated alkaline cleaners, deoxidizers and conversion coatings have been identified. As a result of this work, implementation of non-chromate cleaning and deoxidizing materials at Naval Aviation Depots have already resulted in significant cost savings in both processing and waste disposal. Full implementation of these alternatives at all naval levels of maintenance (organizational, intermediate and depot facilities) will result in a major reduction of hazardous waste generated by the Navy. This report describes the RDT&E effort to develop these non-chromated alternatives.

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INTRODUCTION

Aluminum is the most commonly used material in military airframe and aerospace structures due to its high specific strength compared to other structural alloys. During design, component structural and operational requirements are the primary concerns; however, reliability and maintainability are also key requirements. If left unprotected, these systems would rapidly corrode, resulting in unacceptable aircraft and equipment downtime. Therefore, inorganic surface pretreatments and organic coatings are specified for virtually all military equipment and aerospace systems. MIL-S-5002C, "Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems" describes cleaning requirements and surface treatments for aluminum alloys. MIL-F-7179, "Finish, Coatings and Sealants for the Protection of Aerospace Weapons Systems" provides the requirements for organic coatings used on military aircraft. References 1 and 2 provide detailed descriptions of corrosion control documents and finishing systems for military aerospace equipment.

Proper surface preparation and pretreatment are important factors in the overall effectiveness of these finishing systems. Chromates, particularly Chromium VI, are primary ingredients in aerospace pretreatment processes and materials such as alkaline cleaners, deoxidizers, conversion coatings and anodize films. They have been used because of their outstanding performance properties as corrosion inhibitors for aluminum. This property is particularly important to the Navy due to the extensive use of aluminum in naval aircraft and aerospace systems, and the severe corrosive environment in which these systems operate. For example, chromate conversion coatings such as those produced in accordance with MIL-C-5541 using materials conforming to MIL-C-81706 are excellent surface pretreatments for aluminum alloys (3). These materials form a surface oxide film which enhances the overall adhesion and corrosion prevention properties of subsequent protective finishes applied over this oxide film. These conversion coatings have been an essential part of the standard Navy finishing system for aircraft for several decades. Unfortunately, they have been found to be carcinogenic and their use in these processes causes health and safety problems as well as the generation of hazardous waste.

In recent years, Federal, state and local Environmental Agencies have issued legislation that governs the handling, use and disposal of chromate containing materials. The Clean Air Act, Clean Water Act, Resource Conservation and Recovery Act and Air Quality Management Districts (AQMD) rules are all regulations that limit or prohibit the use and/or disposal of chromates and more stringent regulations currently are being initiated. The Department of Defense has determined that the majority of hazardous materials and hazardous waste generated by the DOD comes from its maintenance depots and operations (4). The bulk of these hazardous compounds are associated with cleaning, pretreating, plating, painting and paint removal processes. Chromium is one of the major components in the hazardous waste which is generated. Therefore, while current chromated materials used in these operations perform satisfactorily, non-chromated alternative materials, which exhibit the same outstanding properties, need to be developed. The following is a description of an on-going research and development effort aimed at the elimination or reduction of chromate containing materials used in aerospace processes. This effort addresses environmental problems at all levels of fleet operation: depot, intermediate and organizational. This technology also will have application to other industries.

DESCRIPTION OF SURFACE PREPARATION AND PRETREATMENT PROCESSES

Surface preparation is an essential step in the effectiveness of protective pretreatments for aluminum. Surface preparation consists of several steps: cleaning, deoxidizing and pretreatment. Many non-chromated materials for these steps were identified for this effort. However, most of these candidates were eliminated because of poor performance in the initial screening tests and will not be described in this report. Table 1 lists the promising materials from this evaluation which will be discussed.

ALKALINE CLEANERS

Cleaning is the first step in the preparation of the aluminum surface for pretreating and painting. During this process, organic contaminates on the surface are removed using materials based on high pH soluble salts. Unfortunately, aluminum (Al) is easily corroded by alkaline solutions. Therefore inhibitors, normally chromates, are incorporated to protect the Al against degradation. Furthermore, these cleaners can be used to etch the aluminum surface to reduce surface in-homogeneities. Etchant cleaners are formulated by the addition of sodium hydroxide and sodium salts (i.e. carbonates, phosphates, silicates). Silicate inhibitors are used to regulate the etching rate of these cleaners. These silicates, however, leave a residue on the surface which can result in problems in the subsequent pretreating steps. Lack of oxide film formation, contamination of pretreatment solutions, and surface defects are among some of the problems caused by these residues. Two non-silicated, non-chromated alkaline cleaners were identified for full evaluation in this effort.

DEOXIDIZERS

Deoxidizers are used to remove any remaining surface oxides on the aluminum. These solutions contain chromic acid and some other acid (usually phosphoric or sulfuric). They provide a relatively uniform (chemical and physical) surface for the subsequent chemical treating. Two non-chromated deoxidizers were investigated during this effort. These materials were based on acid solutions (often Nitric) with other additives (Hydrofluoric acid, etc.).

TABLE 1. CLEANERS, DEOXIDIZERS, AND PRETREATMENTS

ALKALINE CLEANERS

Standard Chromated, Silicated Cleaner (MIL-S-5002)

Allied-Kelite's Chemidize 740 (non-silicated & non-chromated)

Turco's 4215 NC-LT (non-silicated & non-chromated)

DEOXIDIZERS

Standard Chromated Deoxidizer (MIL-S-5002)

Sanchem Inc.'s Product #1000 (non-chromated)

Turco's Smut-Go-NCB (non-chromated)

PRETREATMENTS

Alodine 1200S (MIL-C-81706/MIL-C-5541 Chromate Conversion Coating)

Sanchem Safegard CC (Non-Chromate Conversion Coating)

Turcoat #6787 (Non-Chromate Surface Pretreatment)

Alumicoat 6788 (Non-Chromate Surface Pretreatment)

Lockheed's Non-Chromate Surface Pretreatment

PRETREATMENTS

Three methods to obtain a non-chromated alternative to the current chromate conversion coating (CCC) were identified for the pretreatment replacement effort. The first approach was a direct replacement of the current conversion coating with a non-chromated version. This is the simplest solution, allowing for a one for one replacement of the performance properties without any changes to the current requirements.

Only one viable direct replacement material was identified from industry survey. This material was an immersion process from Sanchem Inc. called Safegard CC. This non-chromate conversion coating process attains the final surface film from an alkaline pathway using a multi-stage process. This process uses cleaning and deoxidizing steps similar to the current conversion coating, except that all materials are non-chromated and the alkaline cleaner is also non-silicated. Furthermore, the Sanchem process itself is a multi-stage tank process operated at elevated temperatures, therefore requiring several heated tanks for production of the protective film. This differs from the traditional chromic acid based conversion coatings which can be applied by either a single stage immersion tank process or by spray application. Finally, the waste streams from these two processes are different. The Sanchem process effluent is void of any chromium and does not have to be treated as hazardous, whereas, the standard CCC waste stream contains chromium and must be disposed as a hazardous waste.

An alternative or secondary pathway was made possible by the improved high performance qualities of current primers. This "total system" approach consists of an adhesion promotion pretreatment used in conjunction with a subsequent primer. The performance of the adhesion promoter/primer system was evaluated against the combined performance of the CCC and the standard primer. This total system approach was derived from a previous 6.1 research effort at NAVAIR-WARCENACDIVWAR which investigated the water disbondment characteristics and interfacial bonding of coating/substrate systems (5). This total system concept would require a re-definition of finishing processes and specifications. Three adhesion promotion candidates (Table 1) were identified for the total system concept. These adhesion promoters were evaluated with emphasis on their performance with several standard primer systems.

Thirdly, a possible short term alternative is the use of non-rinse chromate conversion coatings. These materials are applied and then dried in place, thereby eliminating the water rinse used in the present CCC process. Although these treatments do not eliminate chromates, they do significantly limit the amount of chromates in the effluent stream which has to be sent for waste disposal. Since waste disposal is a current issue, this approach could be a viable temporary solution. None of the non-rinse chromated pretreatments evaluated to date passed the initial screening tests with coating adhesion being the primary deficiency. Therefore, they will not be discussed in this report.

Finally, numerous other pretreatment candidates have been identified for evaluation. Testing of these materials is currently in progress and results on these materials will be discussed in a future report.

EXPERIMENTAL

The objective of this effort was to develop non-chromated pretreatments for Navy aircraft aluminum components. In order to accomplish this task, an industry wide survey was performed to identify existing technologies. A number of candidate raw materials and pretreatments were uncovered from this survey. The performance of these experimental materials was evaluated on common aluminum alloys and with standard Navy coating systems. Physical performance tests (i.e. corrosion resistance, adhesion, etc.) were used to screen these pretreatments. Most of the non-chromate conversion coating candidates resulted in poor adhesion and/or poor corrosion resistance. These materials were eliminated from further study. In addition, most of the adhesion promotion pretreatments initially performed well; however, their performance in a marine environment significantly deteriorated to an unacceptable level and they also were eliminated from further evaluation. The promising pretreatments or processes from the initial study were used to develop the optimum replacement systems. The effectiveness of these pretreatment systems were evaluated both alone and in conjunction with standard Navy aircraft coating systems. Corrosion resistance tests, adhesion tests, and electrochemical impedance spectroscopy (EIS) were used to analyze the physical and electrochemical performance properties of these pretreatment/coating "total" systems. Specimens with the standard conversion coating as well as no pretreatment were used as controls. The following is a description of the substrates, coatings, and experimental procedures used in this investigation.

MATERIALS

The substrates for this study were bare 2024 T-3, 7075 T-6 and 6061 T-3 aluminum alloys. Tables 1 and 2 list the pretreatments and coatings used on these substrates in this investigation. All test specimens were prepared at our laboratories following the manufacturers' recommended procedures, except for the Lockheed test specimens and the initial tests on the Turcoat product which were prepared by Lockheed and Turco, respectively. Non-chromated cleaning and deoxidizing procedures were used in the preparation of all candidate pretreatments. The chromate conversion coating control specimens represent the common pretreatment found on military aircraft prior to painting.

TABLE 2. SPECIFICATIONS FOR ORGANIC COATING SYSTEMS

1. MIL-P-23377D, Type 1 "Primer Coatings, Epoxy Polyamide, Chemical and Solvent Resistant." Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

2. MIL-P-85582A, Type 1 "Primer Coatings: Epoxy, Waterborne." Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

3. TT-P-2760, Type 1 "Primer Coating: Polyurethane, Elastomeric." Film thickness: 20.3 to 30.5 microns (0.0008 to 0.0012 inches).

4. MIL-P-23377D, Type 1, Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

   MIL-C-83286B, Type 1, "Coating Urethane, Aliphatic Isocyanate, for Aerospace Application." Film thickness: 45.7 to 55.9 microns (0.0018 to 0.0022 inches).

5. TT-P-2756 "Polyurethane Coating: Self-Priming Topcoat, Low Volatile Organic Compounds (VOC)." Film thickness: 50.8 to 55.9 microns (0.0020 to 0.0022 inches).

6. MIL-P-23377D, Type 1, Film thickness: 15.2 to 22.9 microns (0.0006 to 0.0009 inches).

   MIL-C-85285B, Type 1, "Coating: Polyurethane, High Solids." Film thickness: 45.7 to 55.9 microns (0.0018 to 0.0022 inches).

The above coatings were applied by conventional air spray and were allowed to cure for seven days prior to testing.

EXPERIMENTAL PROCEDURES

Adhesion and Water Resistance

Adhesion of organic coating systems to the pretreated specimens was evaluated using two methods: wet tape adhesion and scrape adhesion. The wet tape test is a modified version of the American Society for Testing and Materials ASTM D 3359, method A. This test was performed by immersing a specimen in distilled water for a period of time at a specific temperature. Three immersion conditions were used for this test: 24 hours at 23°C, 96 hours at 49°C, and 168 hours at 65°C. Upon removal, two parallel scribes, 3/4 inch apart, were cut through the coating and into the substrate. An "X" was subsequently scribed through the coating between the two initial scribes. A strip of 3M 250 masking tape was applied firmly to the coating surface perpendicular to the scribe lines and immediately removed with one quick motion. The specimens were examined for removal and uplifting of the coating from the substrate and the adhesion rating based on the percentage of coating remaining on the surface was recorded. Table 3 gives the performance description for these adhesion ratings. In addition, the water resistance of the pretreatment/coating systems was characterized by examining the test panels for softening, uplifting, blistering, and other coating defects and substrate corrosion which may have resulted from the exposure.

The scrape test was performed in accordance with ASTM D 2197, method A on specimens with a section of the substrate surface exposed. The instrument used to perform this test was a SG-1605 Scrape Adhesion Test Apparatus manufactured by Gardner Laboratory. The test was performed by guiding a weighted stylus at a 45° angle to the specimen along the exposed substrate into the coating system. The scrape adhesion was recorded as the heaviest weight used without shearing the coating from the substrate.

Corrosion Resistance

Five aluminum specimens of each pretreatment finishing system were exposed in 5% salt spray (ASTM B 117) for 336 hours. Upon removal, the panels were inspected for evidence of corrosion. Another set of panels were exposed to SO_2/salt spray (ASTM G 85) for 48 hours and then examined for signs of corrosion.

Four aluminum specimens for each pretreatment/coating system were scribed with a figure "X" through the coating into the substrate. Two specimens each were exposed in 5% salt spray (ASTM B 117) for 2000 hours and two were exposed to SO_2/salt spray (ASTM G 85) for 500 hours. The panels were then inspected for corrosion in the scribe area and blistering of the coating. Subsequently, one panel from each exposure was chemically treated to remove the organic coating without disturbing the substrate and the specimen was examined for corrosion.

Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were made using an EG&G Princeton Applied Research Corp. (PARC) Model 388 AC Impedance System. The test cell used for this investigation consisted of a glass O-ring joint clamped onto a coated metal specimen as described in Reference 6. The electrolyte used for specimen exposure was a 3.5% NaCl solution with a pH of 6. EIS tests were performed on systems based on combinations of aluminum alloys (i.e. 2024-T3, 7075-T6, etc.), surface pretreatments (i.e. chromate conversion coatings and Sanchem non-chromate treatment), and coating systems (i.e. epoxy primer and epoxy primer/urethane topcoat). The specimens were exposed to the electrolyte solution at room temperature and periodic impedance measurements were made over the test exposure time. The first series of tests was performed after 24 hours of exposure in order to allow the electrochemical system to reach equilibrium.

RESULTS AND DISCUSSION

The primary goal of this effort was the elimination of chromates from aerospace surface preparation and pretreatment processes for aluminum. These toxic materials have been used because of their outstanding performance properties, especially as corrosion inhibitors for aluminum. Therefore, in order to develop effective replacements for Chromium VI, particular attention was placed on corrosion resistance and adhesion tests. Non-chromated alternatives for alkaline cleaners, deoxidizers, and conversion coating processes were investigated for this effort.

ALKALINE CLEANERS AND DEOXIDIZERS

The best way to demonstrate the effectiveness of alkaline cleaners and deoxidizers is to use them in the chromate conversion coating process. Of all the pretreating processes, the requirements of this process are the most difficult to pass. Residues as well as oxides remaining on the surface after cleaning and deoxidizing will interfere with the quality of the chemical film produced. Two commercial non-silicated, non-chromated alkaline cleaners (Allied-Kelite's Chemidize 740 and Turco's 4215 NC-LT) and two non-chromated deoxidizers (Sanchem Inc.'s Product #1000 and Turco's Smut-Go-NCB) were evaluated in this effort. These materials were selected based on initial screening tests along with information from the Aerospace Chromium VI Elimination (ACE) Team, which is a group of aerospace manufacturers who have banned together to solve a common problem.

Test panels were processed with the non-chromate cleaners and deoxidizers then treated with Alodine 1200S (chromate conversion coating). In addition, control panels were processed with the standard chromated cleaners and deoxidizers. Test specimens for each candidate and the controls were exposed in salt spray (ASTM B-117) for 336 hours on 6o racks as per the MIL-C-81706 specification requirement. Also, coating adhesion tests were performed using MIL-P-23377 epoxy primer and the adhesion tests described earlier. All materials met the 336 hour corrosion resistance and the 24 hour wet tape coating adhesion requirements of the MIL-C-81706 conversion coating specification.

Information on the etch rates and intergranular attack (IGA) of these materials was obtained through investigations conducted by other members of the ACE Team (Rockwell, Grumman, etc.). Most of the non-chromated deoxidizer alternatives were less active (lower etch rates) than the chromated materials. To compensate for this, the operating temperature was increased, thereby shortening the processing time while remaining within safe limits for IGA. Results on the non-chromated alternatives evaluated in this program were coordinated with the Naval Air Systems Command and some of the recommended alternatives have already been implemented at several Navy facilities. One added benefit derived from these substitutes is that most of them are suitable for direct substitution into existing procedures.

The deoxidizers and alkaline cleaners from this study have been successfully demonstrated at two Naval Aviation Depots (Jacksonville and North Island). A cost benefit analysis was performed at NADEP Jacksonville for the implementation of the non-chromate deoxidizer. The results of this analysis showed a cost savings of $23K per year for this process. In addition, there was a reduction of over 3 tons in the amount of chromium waste generated from this process.

PRETREATMENTS

The most promising non-chromated pretreatments and adhesion promotion materials were investigated using laboratory physical tests as well as electrochemical impedance spectroscopy. The following is a summary and discussion of the results for these materials as well as the control specimens.

Adhesion And Water Resistance

The results of the adhesion/water resistance tests are provided in Table 4. Expanded adhesion information on the Sanchem material is presented in Table 5. A standard aerospace requirement for scrape adhesion is 3 kg. The scrape adhesion results for the various pretreatment systems ranged from 1.0 Kg to >10.5 Kg. The results for Alodine ranged from 1.0 to 5.0 Kg with an average of 3.0 Kg for the different replicates performed. This indicated that other factors (such as the coating edge effects, pretreatment thickness, pre-paint surface cleanliness, etc.) affected the outcome of the tests. Another example is the Sanchem unsealed process which averaged around 2.0 Kg but some results were as high as 10.5 Kg.

TABLE 3. ASTM D3359 ADHESION RATINGS

Rating	Description
5A	No peeling or removal
4A	Trace peeling or removal along incisions
3A	Jagged removal along incisions up to 1/16 in. (1.6 mm) on either side
2A	Jagged removal along most of incisions up to 1/8 in. (3.2 mm) on either side
1A	Removal from most of the area of the X under the tape
0A	Removal beyond the area of the X

TABLE 4. ADHESION/WATER RESISTANCE TEST RESULTS

ALLOY/ PRETREATMENT	MIL-P-23377 (Epoxy Primer) SCRAPE (Kg)	DRY TAPE	WET TAPE (24*)	WET TAPE (96)	WET TAPE (168)	TT-P-2756 (Unicoat) SCRAPE (Kg)	DRY TAPE	WET TAPE (24)	WET TAPE (96)	WET TAPE (168)
2024-T3 Al Alloy										
Alodine 1200S	3.0	5A	5A	5A	5A	5.0	5A	5A	5A	5A
Sanchem Safegard-CC	1.5	5A	5A	5A	5A	3.5	5A	4A	4A	4A
Sanchem (Unsealed)	2.0	5A	5A	5A	5A	3.5	5A	4A	4A	4A
Turcoat 6787 (1)	2.0	4A	0A	0A	0A	4.5	1A	0A	0A	0A
Turcoat 6787 (2)	3.0	5A	3A/5A#	5A	—	—	5A	5A	5A	—
Alumicoat 6788	1.0	5A	4A	4A	5A	10+	5A	5A	5A	5A
Lockheed Process	6.0	5A	5A	5A	5A	—	—	—	—	—
7075-T6 Al Alloy										
Alodine 1200S	3.0	5A	5A	5A	5A	5.0	5A	5A	5A	5A
Sanchem Safegard-CC	1.5	5A	5A	5A	5A	3.0	5A	5A	4A	5A
Sanchem (Unsealed)	1.5	5A	5A	5A	5A	3.0	5A	5A	4A	4A
Turcoat 6787	2.0	4A	0A	0A	0A	4.0	2A	0A	0A	0A
Alumicoat 6788	1.0	5A	4A	3A	5A	7.0	5A	5A	4A	4A
Lockheed Process	2.5	5A	5A	5A	5A	—	—	—	—	—

— Test not performed

* Hours immersion in deionized distilled water

Results were 5As except for one 3A

(1) Results for panels processed at NAWCADWAR.

(2) Results for initial panels submitted by Turco.

TABLE 5. EXPANDED ADHESION TEST RESULTS FOR SANCHEM AND CONTROL SYSTEMS

ALLOY PRETREATMENT	MIL-P-23377 DRY	MIL-P-23377 WET+	MIL-P-85582 DRY	MIL-P-85582 WET	TT-P-2760 DRY	TT-P-2760 WET	UNICOAT DRY	UNICOAT WET
2024-T3 Al Alloy								
Chromated Deoxidize	5A	5A	5A	5A	5A	0A	5A	0A
Non-Chromated Deox	5A	5A	5A	5A	5A	5A	5A	5A
Alodine 1200S	5A	5A	5A	5A	5A	5A	5A	5A
Sanchem Safegard-CC	5A	5A	5A	5A	5A	5A	5A*	5A
Aged Sanchem (#)	5A	5A	5A	5A	5A*	5A	5A*	5A
7075-T6 Al Alloy								
Chromated Deoxidize	5A	0A	5A	5A	5A	0A	5A	5A
Non-Chromated Deox	5A	3A	5A	5A	5A	5A	4A	0A/5A^
Alodine 1200S	5A	5A	5A	5A	5A	5A	5A	5A
Sanchem Safegard-CC	5A	5A	5A	5A	5A	5A	5A	5A
Aged Sanchem (#)	5A	5A	5A	5A	5A	5A	5A	5A
6061-T3 Al Alloy								
Alodine (Class3)	5A	5A	5A	5A	5A	5A/0A	5A	5A/2A
Sanchem Safegard-CC	5A	5A	5A	5A	5A	5A	5A	5A
Aged Sanchem (#)	5A	5A	5A	5A	5A	5A	5A	5A

* 1 Small spot of removal

\# Painted and tested 3 months after pretreatment was applied

^ Most of panel rated 0A with parts rated at 5A

\+ 24 hour immersion in deionized distilled water at 23°F

The tape tests showed that all systems with both the MIL-P-23377 primer and the self-priming topcoat had adhesion values of 5A or 4A, except the Turco and one Alumicoat 7075. This indicates good adhesion under the test conditions. Furthermore, these tests were performed immediately after the 7 day cure time for the coatings. With aging of the finishing system, adhesion normally improves. The Alumicoat 6788 on 7075 after 4 days decreased from a 5A to a borderline 3A rating, however, the 7 day at 150 specimen were 5A. The only drastic failure came from the Turco 6787 pretreatment panels processes at NAWCADW where the coatings peeled completely from the substrates after water immersion. This indicated a severe attack of the pretreatment/coating interface by water. This phenomenon is in contrast with the results obtained for the panels prepared by Turco which had ratings of 5A indicating virtually no susceptibility to coating-substrate disbondment upon exposure to water. After consulting with the manufacturer, the differences were attributed to variances in the processing of the panels (drying, pretreatment thickness, etc.). Further tests are being performed to rectify this dilemma. In the expanded adhesion tests (Table 5), excellent adhesion results were obtained for the Alodine and Sanchem treatments with various coating systems. The specimens that were only deoxidized, however, showed a susceptibility to water disbondment (5). Most of the pretreatment systems exhibited excellent water resistance which is evidenced by the tape test results after extended immersion in water. These results are not unexpected, since pretreatments are applied to enhance adhesion of subsequent organic coatings.

Bare Corrosion Resistance

Unpainted specimens for the Sanchem, Lockheed and standard conversion coating processes were exposed to 5% salt spray and examined for evidence of corrosion at 168 hours and 336 hours. A summary of the evaluation is provided in Table 6. The Alodine pretreatment on all alloys and the Sanchem 7075 specimens passed 336 hours of exposure without any evidence of surface corrosion indicating excellent system performance. The Sanchem 2024 specimens, however, began to show signs of corrosion after 200 hours of exposure and at 336 hours they had about 8-10 spots around 1/64 inch in diameter per panel. The Lockheed process had moderate corrosion over most of the panels by 168 hours and heavy corrosion over the entire panels by 336 hours. This poor unprimed performance is unacceptable. However, since the Lockheed process could still be a candidate for the total system approach, it was evaluated further. Since the Turcoat, Alumicoat and unsealed Sanchem processes were submitted for the total system approach, they were not tested for bare corrosion resistance. Their performance in this test, however, was suspected to be poor based on the manufacturers information.

Alodine and Sanchem specimens were exposed to SO_2/salt spray for 48 hours and then examined for corrosion on the surface. These results are summarized in Table 7. The SO_2/salt spray environment simulates industrial stack gases such as

TABLE 6. 5% NaCl Salt Spray Results for Unpainted Panels

ALLOY	- PRETREATMENT	168 HOUR TEST RESULTS
2024-T3	- Alodine	No surface corrosion
2024-T3	- Sanchem	No surface corrosion
2024-T3	- Lockheed	Moderate surface corrosion (90%)#
7075-T6	- Alodine	No surface corrosion
7075-T6	- Sanchem	No surface corrosion
7075-T6	- Lockheed	Moderate surface corrosion (60%)
6061-T6	- Alodine 3^	No surface corrosion
6061-T6	- Sanchem	8-10 small surface spots (1/64")+

ALLOY	- PRETREATMENT	336 HOUR TEST RESULTS
2024-T3	- Alodine	No surface corrosion
2024-T3	- Sanchem*	8-10 small surface spots (1/64")+
2024-T3	- Lockheed	Very heavy corrosion (90%)
7075-T6	- Alodine	No surface corrosion
7075-T6	- Sanchem	No surface corrosion
7075-T6	- Lockheed	Very heavy corrosion (100%)

* First signs of corrosion occurred at 200 hours.
\# Percentage of surface corroded.
^ Class 3 conversion coating
\+ Maximum spot diameter

TABLE 7. SO_2 Salt Spray Results for Unpainted Panels

PANEL	-	PRETREATMENT	48 HOUR TEST RESULTS
6061-T6	-	Sanchem	Small pits over entire surface (1)
6061-T6	-	Alodine 3^	Small pits over entire surface (2)
2024-T3	-	Sanchem	Small pits over entire surface (1)
2024-T3	-	Alodine	Small pits over entire surface (2)
7075-T6	-	Sanchem	Small pits over entire surface (1)
7075-T6	-	Alodine	Small pits over entire surface (2)

^ Class 3 Conversion Coating

* Ranking of pretreatments on the particular alloy where (1) had a lesser density of smaller pits vs (2).

those found on diesel powered carriers, and it is an extremely aggressive environment. The exposure period was selected based on the performance of the specimen. Both sets of test panels had spotted corrosion on the entire surface within 48 hours, although, the Sanchem panels appeared to have less overall corrosion than the Alodine. This better SO_2 resistance may be due to the fact that the film from the Sanchem process is formed by an alkaline process, whereas the Alodine conversion coating is formed from an acidic one. However, since both systems failed in a short test duration the bare SO_2 testing was discontinued for all future material testing.

Painted Corrosion Resistance

Corrosion resistance is an important property for Navy aircraft coatings due to the severe operational environment in which the aircraft are deployed. Therefore, most aircraft primer specifications have a minimum of 1000 hours exposure to salt spray as the corrosion resistance requirement. The pretreatment plays an integral role in meeting this requirement by maintaining the integrity of the coating/substrate interface. To evaluate this property, painted specimens for each pretreatment were exposed to 5% salt spray and examined for corrosion in the scribe area and blistering of the coating. A summary of the evaluation is provided in Table 8. Most of the pretreatment systems, with both the standard epoxy primer and the epoxy primer-polyurethane topcoat coating systems, passed 1000 hours of exposure. There were no corrosion products in the scribe or any blistering of the coating. Only the Turcoat and Alumicoat pretreatments on 2024-T3 Al with the standard primer-topcoat system exhibited some evidence of corrosion along the scribe between 500 and 1000 hours of exposure. This is unexpected, since the un-topcoated specimen passed this exposure time. Permeability, adhesion, or intercoat stresses could have been the cause of this discrepancy. Also, between 500 and 1000 hours, the untreated deoxidized panels showed signs of corrosion and blistering with both the standard epoxy primer and the epoxy primer-polyurethane topcoat coating systems. This degradation continued with these systems until they were a borderline failure at 1500 hours and a complete failure at 2000 hours.

Since most systems performed well for over 1000 hours on both substrates, the test was continued for another 1000 hours. At both 1500 hours and 2000 hours, there were no corrosion products in the scribe or blistering of the coating for any of the Alodine or Sanchem (both sealed and unsealed) specimens. Subsequently, the coatings were carefully removed from the surface with a chemical stripper, without disturbing the underlying substrate. Upon further examination, there was no evidence of underlying corrosion on these panels. At 1500 hours, all of the primed pretreatments still passed. However, at 2000 hours, the Turcoat primed specimens had borderline results on both alloys. There were some slight spots of scribe corrosion and small coating blisters were noticeable. At 1500 hours, the primed and topcoated

TABLE 8. CORROSION RESISTANCE TEST RESULTS (5% SALT SPRAY)

ALLOY/ PRETREATMENT	MIL-P-23377 HOURS OF EXPOSURE				MIL-P-23377/MIL-C-85285 HOURS OF EXPOSURE			
	500	1000	1500	2000	500	1000	1500	2000
2024-T3 Al Alloy								
Chromated Deox only	P	+	—	F	P	+	—	F
Non-Chromated Deox	P	+	—	F	P	+	—	F
Alodine 1200S	P	P	P	P	P	P	P	P
Sanchem Safegard-CC	P	P	P	P	P	P	P	P
Sanchem (Unsealed)	P	P	P	P	P	P	P	P
Turcoat 6787 (1)	P	P	P	—	P	+	—	F
Turcoat 6787 (2)	P	P	P	P	P	P	P	P
Alumicoat 6788	P	P	P	P	P	+	+	—
Lockheed Process	P	P	P	P	P	P	+	+
7075-T6 Al Alloy								
Chromated Deox only	P	+	—	F	P	+	—	F
Non-Chromated Deox	P	+	—	F	P	+	—	F
Alodine 1200S	P	P	P	P	P	P	P	P
Sanchem Safegard-CC	P	P	P	P	P	P	P	P
Sanchem (Unsealed)	P	P	P	P	P	P	P	P
Turcoat 6787 (1)	P	P	P	+	P	P	P	P
Alumicoat 6788	P	P	P	P	P	P	P	P
Lockheed Process	P	P	P	P	P	P	—	—

P = Pass (no evidence of corrosion on panel)

\+ = Borderline Pass (trace of corrosion in scribe, no other corrosion)

— = Borderline Failure (traces of scribe corrosion and other corrosion)

F = Failure (corrosion in scribe and on panel surface)

(1) Results for panels processed at NAWCADW.

(2) Results for initial panels submitted by Turco.

Turcoat and Alumicoat 2024-T3 specimens and the Lockheed specimens (both alloys) had borderline results. This trend continued at 2000 hours except that the 2024-T3 Turcoat specimens were complete failures showing corrosion and pitting in the scribe along with many blisters on the panel surface. In general, the only systems to show no evidence of corrosion under any of the conditions were the Alodine and Sanchem specimens.

Painted specimens exposed to SO_2/salt spray also were examined for damage to the coating and corrosion in and away from the scribe and these results are summarized in Table 9. The SO_2/salt spray environment simulates industrial stack gases such as those found on aircraft carriers from engine exhausts, and it is an extremely aggressive environment. Most aircraft coating specifications do not have exposure to SO_2/salt spray as a corrosion resistance requirement, therefore, the exposure periods selected were based on differences in finishing system performance.

Primed panels after being exposed for 168 hours were examined for signs of corrosion. On both alloys, the Alodine panels had some scribe corrosion and slight blistering of the coating, but they were considered borderline failures relative to the extensive corrosion observed on the deoxidized specimens. The Lockheed pretreated specimens were also failures after this duration. The rest of the primed pretreatment specimens passed at this point. At 336 hours, however, all of the primer specimens failed except for the unsealed Sanchem on 2024. This result was unexpected since the 7075 specimen failed at this point and in the unprimed corrosion resistance performance tests, 7075 was better than 2024. Even at 500 hours the Sanchem 2024 specimen was a borderline pass. Again, the Sanchem panels performed better than the Alodine panels in the SO_2/salt spray environment.

The primed and topcoated specimen results were similar to those obtained for the primer only. The deoxidized only specimens failed completely at 168 hours. This poorer performance in all of the corrosion tests and the adhesion tests reaffirm the necessity for the use of some type of pretreatment in the protective finishing system. The Sanchem specimens passed 168 hours on both alloys, out-performing the Alodine which again were borderline failures. The Lockheed panels as well as the Turcoat 7075 panels were also borderline failures for this duration. At 336 hours, the Alumicoat on both alloys passed and continued to show only slight signs of failure at 500 hours. The Turcoat specimens remained borderline failures for these additional exposures while the Lockheed panels failed by 336 hours. The Sanchem panels were borderline failures at 336 hours on 7075 Aluminum, while the Alodine and Sanchem 2024 specimens failed. Finally, at 500 hours the 7075 Sanchem specimen had failed. Once again, the better performance of the Sanchem material, relative to Alodine, in this test may be due to the pH of the processes used for film formation.

TABLE 9. CORROSION RESISTANCE TEST RESULTS (SO_2/SALT SPRAY)

ALLOY PRETREATMENT	MIL-P-23377 HOURS OF EXPOSURE 168	336	500	MIL-P-23377MIL-C-85285 HOURS OF EXPOSURE 168	336	500
2024-T3 Al Alloy						
Chromated Deox only	F			F		
Non-Chromated Deox	F			F		
Alodine 1200S	—	F		—	F	
Sanchem Safegard-CC	P	F		P	F	
Sanchem (Unsealed)	P	P	+	P	F	
Turcoat 6787	P	F		P	—	—
Alumicoat 6788	P	F		P	P	+
Lockheed Process	F			—	F	
7075-T6 Al Alloy						
Chromated Deox only	F			F		
Non-Chromated Deox	F			F		
Alodine 1200S	—	F		—	F	
Sanchem Safegard-CC	P	F		P	—	F
Sanchem (Unsealed)	P	F		P	—	F
Turcoat 6787	P	F		—	—	—
Alumicoat 6788	P	F		P	P	+
Lockheed Process	F			—	F	

(P = Pass, + = Borderline Pass, - = Borderline Failure, F = Failure)

In general, the corrosion resistance of the pretreatment systems in combination with the standard epoxy primer or the epoxy primer-polyurethane topcoat coating systems was as good as the performance of the Alodine controls. In some cases such as the Sanchem panels in the SO_2 environment, the performance was slightly better than that of the Alodine. This equivalent performance for these treatments as compared to the chromate conversion coating, is due to a high degree of interfacial integrity between the pretreatments and the coatings.

Electrochemical Impedance Spectroscopy

Electrochemical impedance spectroscopy (EIS) provides qualitative and quantitative information about the corrosion resistance properties of both the coating and the substrate in addition to providing insight on the nature of their interfacial adhesion. Reference (7) provides a detailed description of EIS and its application for analyzing organic coating/metal substrate systems. Figures 1-3 contain Bode magnitude and phase diagrams of the EIS test results obtained at various exposure intervals for several of the coating/pretreatment systems. These specific spectra represent the significant EIS trends that were identified during this investigation.

After 24 hours immersion (in 3.5% NaCl), the Sanchem pretreatment on 2024 aluminum had impedance values around 10^9 ohms in the low frequency range (10^{-2} Hz). The same results were observed for this system throughout the 4000 hour test duration as illustrated in Figure 1. This high impedance value correlates to a coating system with low conductivity that provides good barrier protection to the substrate to which it is applied. This impedance value is far above 10^7 ohms which is widely accepted as the lower limit below which the barrier protection provided by the coating is no longer a significant factor (7). In the high frequency range, the Sanchem specimens had phase angles around -80°. These phase angles also indicate a good barrier system, where -90° would be a perfect capacitor/barrier. In addition, the shape of the curve for the impedance magnitude is virtually straight over most of the frequency range with a negative slope, again indicating capacitance behavior (i.e. good barrier properties). For both alloys, the low frequency impedance remained around 10^9 ohms, while the high frequency phase angles continued to exhibit capacitive behavior, with phase angles remaining between -70° and -90°. There was a small shift in the low frequency phase angle curve for the Sanchem/2024/primer system over the test duration. The change from capacitive to resistive behavior had shifted slightly to the left (lower frequency) indicating better barrier properties. This change could have resulted from several sources. Possible explanations of this phenomenon include decreased micropore size within the coating due to swelling of the polymer or increased coating adhesion with time as noted in Reference (8).

Figure 2 shows the EIS results for the Sanchem and Alodine specimens with MIL-P-23377 primer on 2024-T3 aluminum alloy substrates. The Alodine system had an

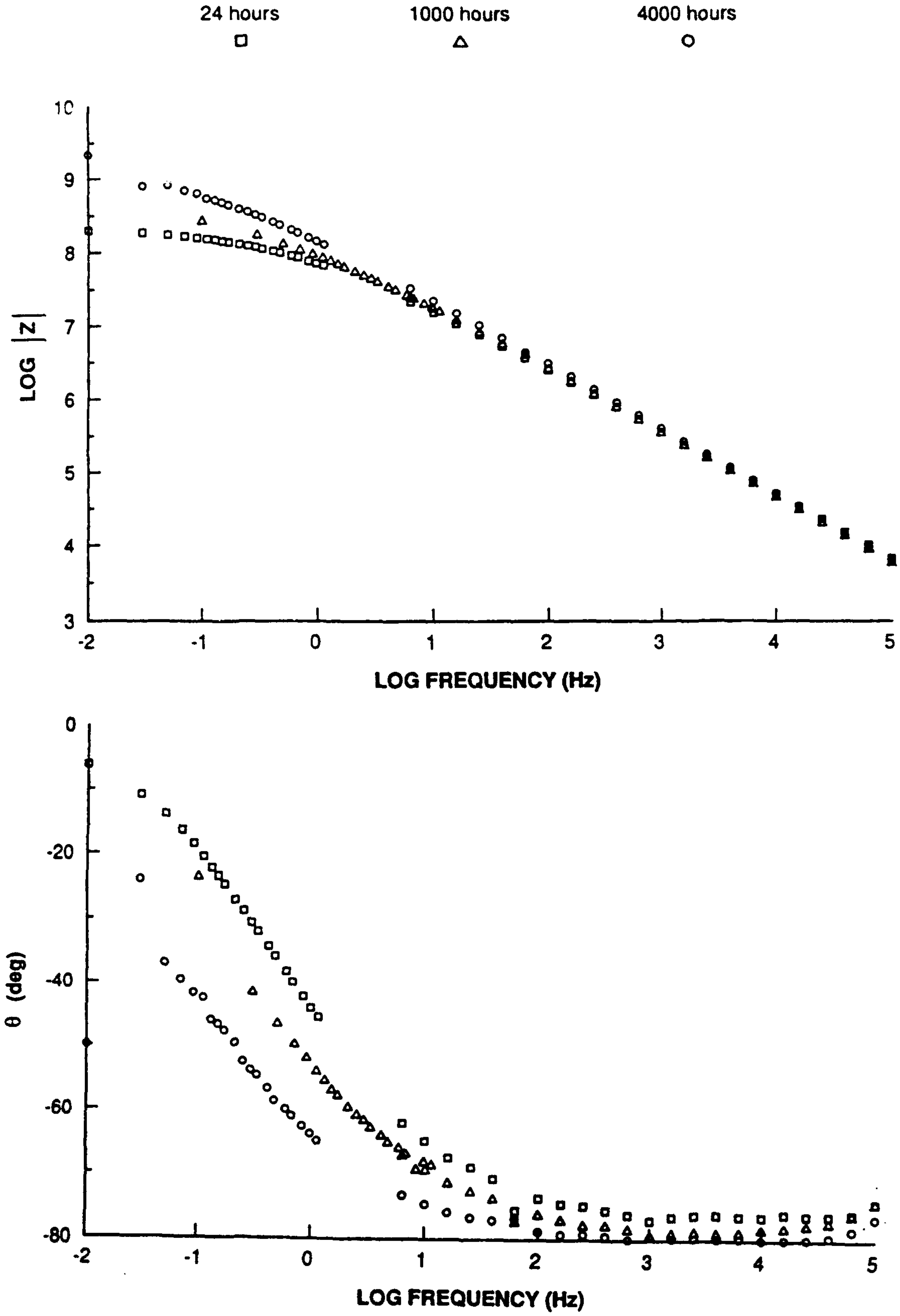

Figure 1. Bode Plot Of Sanchem Pretreatment On 2024-T3 Aluminum With MIL-P-23377 Epoxy Primer.

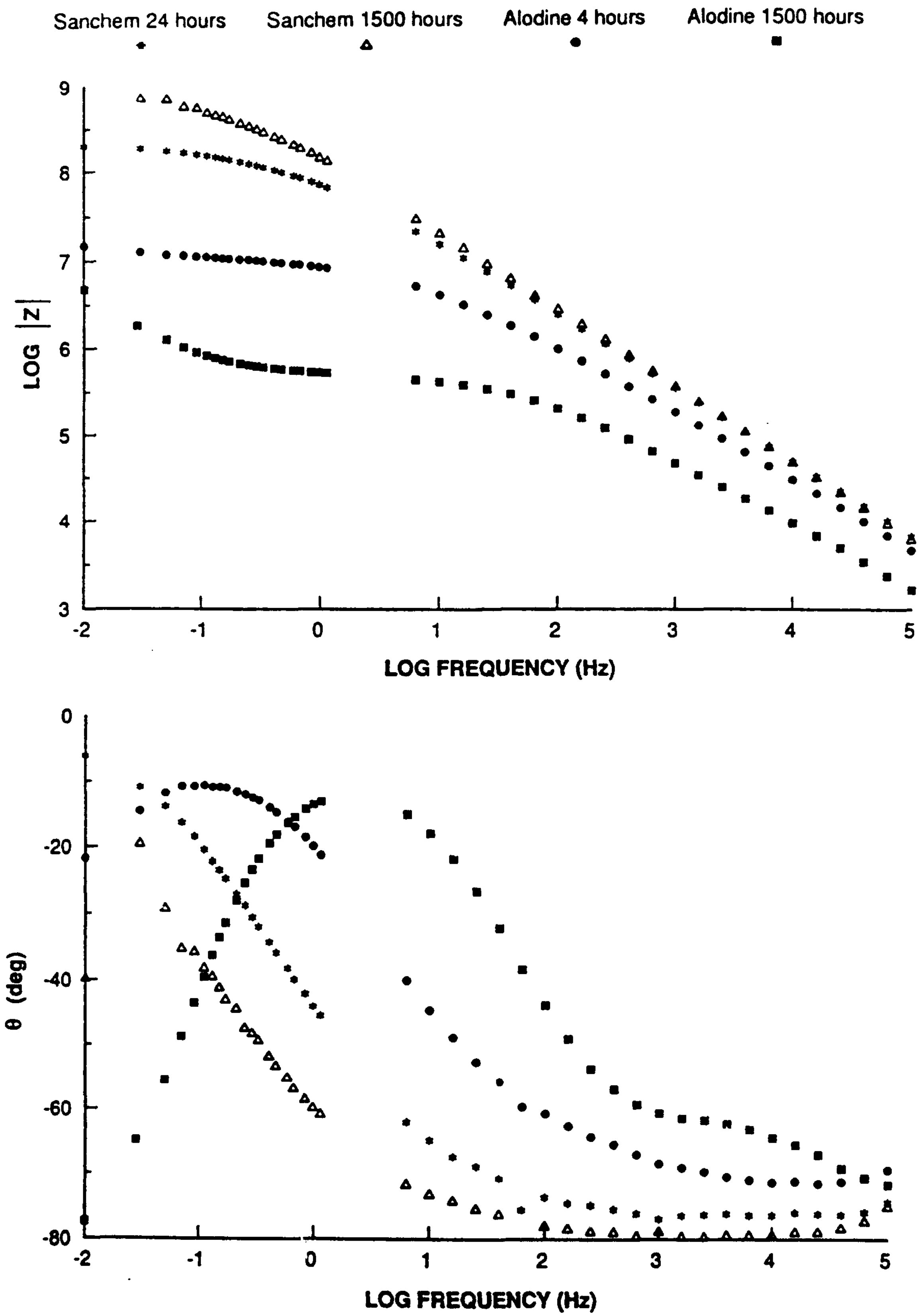

Figure 2. Bode Plot Of Sanchem And Alodine Pretreatment On 2024-T3 Aluminum With MIL-P-23377 Epoxy Primer.

impedance of around 10^7 ohms after 4 hours immersion. However, unlike the Sanchem results which improved with time, this value dropped to 10^6 ohms after 1500 hours immersion. This indicates that the barrier protection provided by the Alodine pretreatment is not as good as the Sanchem material. This lower barrier protection is also apparent in the phase diagram where the phase angles for the Alodine system were between -50° and -70°, showing less capacitive behavior. Although the Alodine-primer system was not as good a barrier as the Sanchem-primer system, it does provide excellent chemical corrosion protection as indicated in the salt spray results and References (9 & 10).

The results for primed and topcoated pretreatment specimens showed similar impedance magnitudes and phase angles for both pretreatments over the entire exposure time. Figure 3 contains the results for 2000 hours which were representative of the systems performance throughout the EIS analysis. The low frequency impedance magnitude of these systems remained around 10^9 ohms and the shape of the curves were similar over 4000 hours of exposure. The phase angle curves for both of these systems were beginning to develop a peak in the high frequency range. This trend indicates the presence of electrolyte at the interface resulting from some adhesion loss. Also, some type of electrochemical reactions were occurring at the interface, probably corresponding to chemical inhibition of the corrosion process by the inhibitors within the primer.

Similar results showing the same kind of equivalent performance between the Sanchem pretreatment and the standard chromate conversion coating were obtained by the Army Materials Technology Laboratory in Watertown, MA through a joint investigation. Representative EIS Bode plots for their results are shown in Appendix A. All of these EIS results indicate that the Sanchem pretreatment is equivalent to the standard chromate conversion coating. Further EIS testing of these pretreatments and the others described in this report will be included in the next report.

Of all the pretreatments evaluated the Sanchem's Safegard CC non-chromate conversion coating process was the best alternative to the current conversion coating based on performance alone. Full scale laboratory testing has been completed on this process and service tests on access panels for the F-18 aircraft are being initiated. Based on the laboratory program, a pilot scale Sanchem process line is scheduled to be set up at the National Defense Center of Environmental Excellence in Johnstown, PA in order to demonstrate the capability to produce a non-chromate surface pretreatment for aluminum. In addition, the waste stream from this process would be void of any chromium and would not have to be treated as hazardous.

However, since the Sanchem process is a multi-tank process operated at elevated temperatures, additional heated tanks would be required for production line

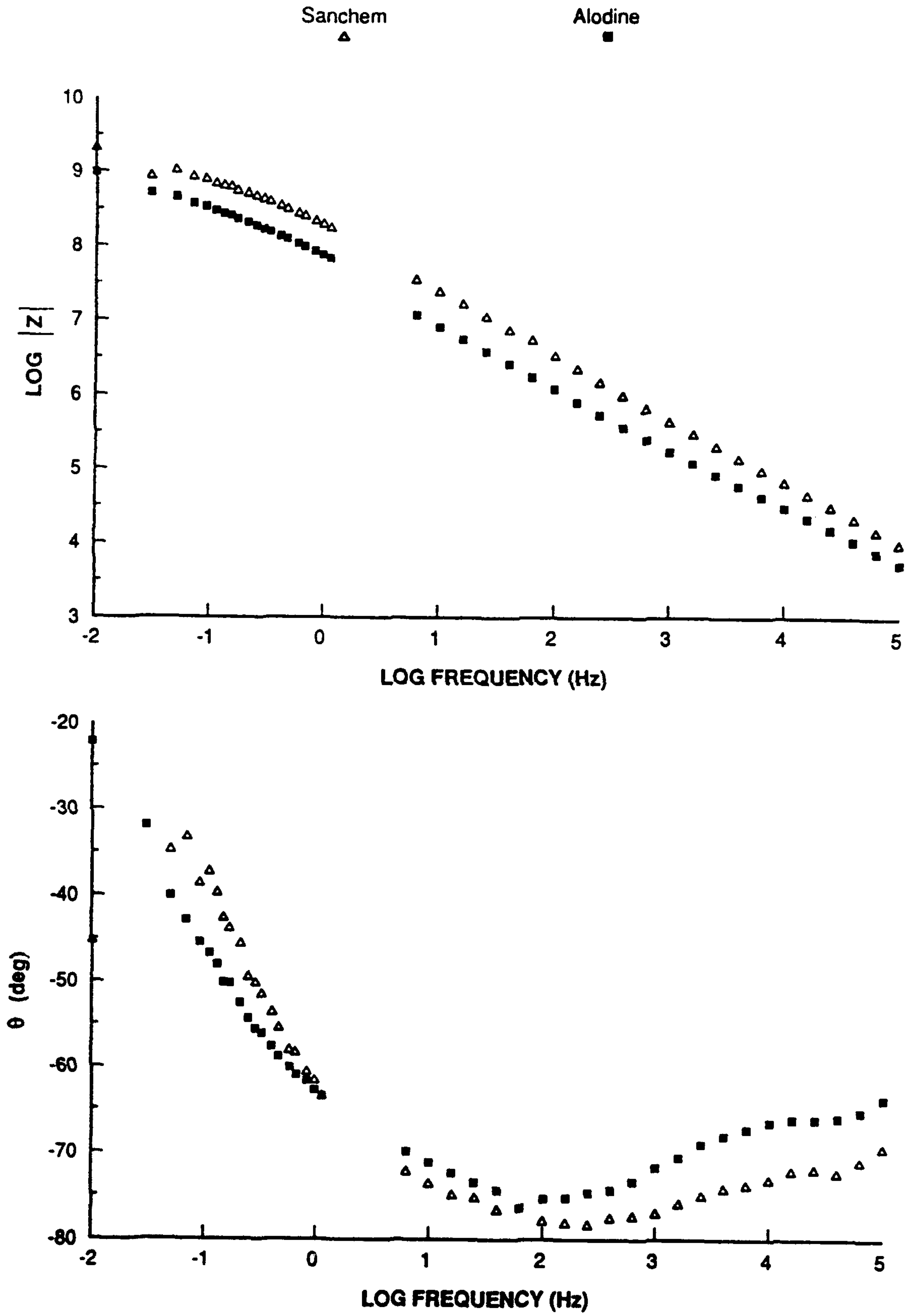

Figure 3. Bode Plot Of Primed And Topcoated 2024-T3 Specimens With Sanchem And Alodine Pretreatments After 2000 Hours Of Exposure.

implementation. Furthermore, unlike the chromate conversion coating process which can be applied by either immersion or spray application, this new pretreatment is a multi-staged, elevated temperature immersion process and is not directly applicable for large components or aircraft skins. To address this issue, efforts to modify the process for spray application are in progress. Incorporating steam cleaning technology to provide the necessary process parameters has shown some preliminary success and is being pursued further. Finally, joint test programs with the Army Materials Technology Lab, Watertown MA and many industry personnel (Aerospace Chromium Elimination Team members) were initiated from this effort and these process evaluations also show promising results.

SUMMARY

This program was aimed at the development of non-chromated alternatives for current aerospace materials and processes. The deoxidizers and alkaline cleaners from this study have been successfully demonstrated at Naval Aviation Depots. The specimens that were only deoxidized showed reduced performance in all of the corrosion tests and the adhesion tests. This information reaffirms the necessity for the use of a pretreatment in the protective finishing system.

Based on the laboratory test results, the Sanchem's Safegard CC non-chromate conversion coating process was the best overall alternative to the current conversion coating. However, this multi-stage process would require additional heated tanks for production line implementation. Joint test programs with the Army Materials Technology Lab, Watertown MA and many industry personnel (Aerospace Chromium Elimination Team members) were initiated from this effort and these process evaluations also show promising results. Production line scale-up is scheduled to be set up at the National Defense Center for Environmental Excellence in Johnstown, PA to demonstrate this process. Also, steam cleaning technology is being investigated to modify the process for spray application. Finally, additional testing is being performed on the other processes to optimize their performance.

Further investigation into these materials and other new materials and processes is continuing. Successful candidates will be demonstrated at Navy field activities and fleet level operations. Transitioning and full implementation of these materials and processes for fleet maintenance operations use is being accomplished through the development or modification of military specifications, revision of maintenance manuals and by changing aircraft and system design plans. The use of these new maintenance materials and processes allow the Navy to meet stringent environmental standards while maintaining operational readiness and efficiency of system performance. In addition, significant cost savings will be recognized by the implementation of the environmentally compliant materials.

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APPENDIX A

ARMY MATERIALS TECHNOLOGY LABORATORY'S

EIS RESULTS

MATERIALS TECHNOLOGY LABORATORY

US ARMY
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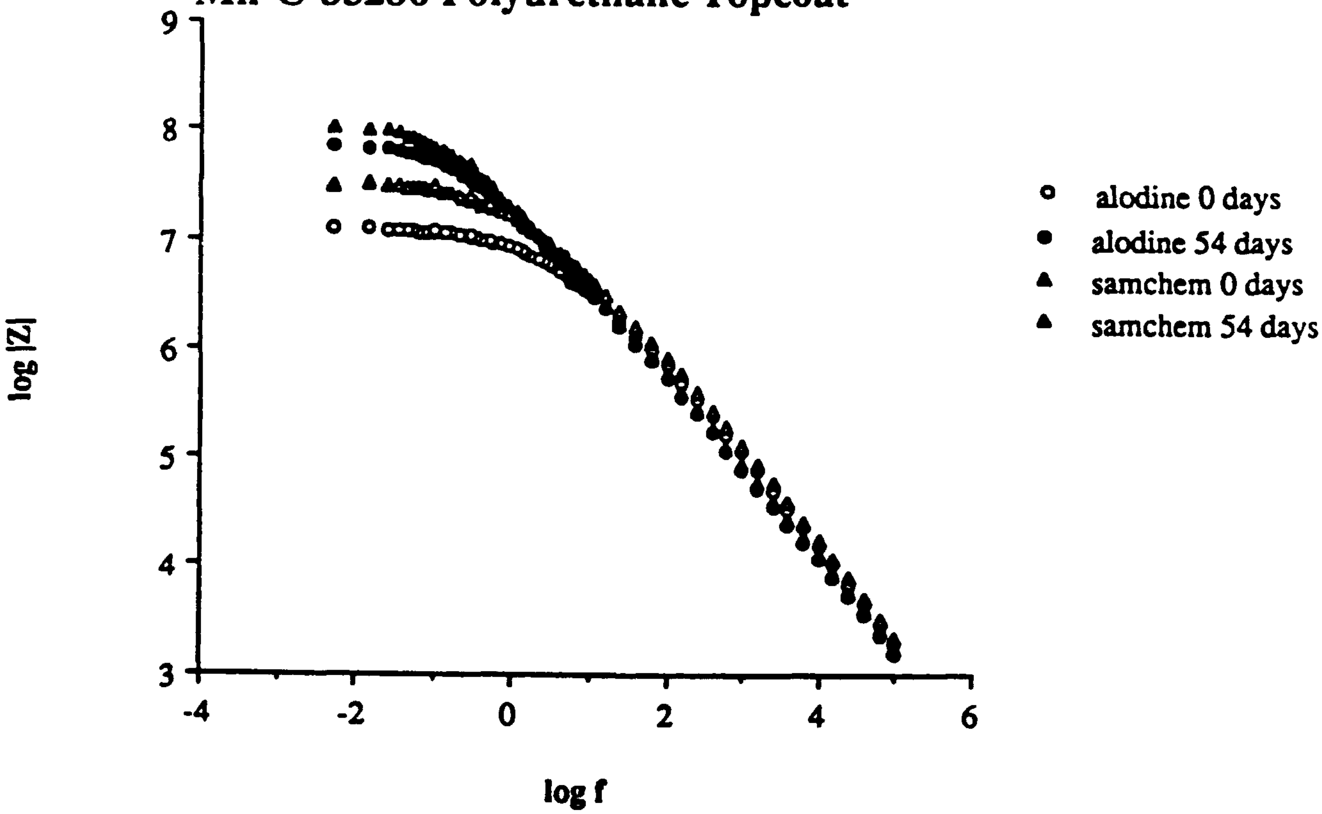
Alodine Vs. Samchem on 7075-T6 with Mil-P-23377 Epoxy Polyamide Primer and Mil-C-83286 Polyurethane Topcoat



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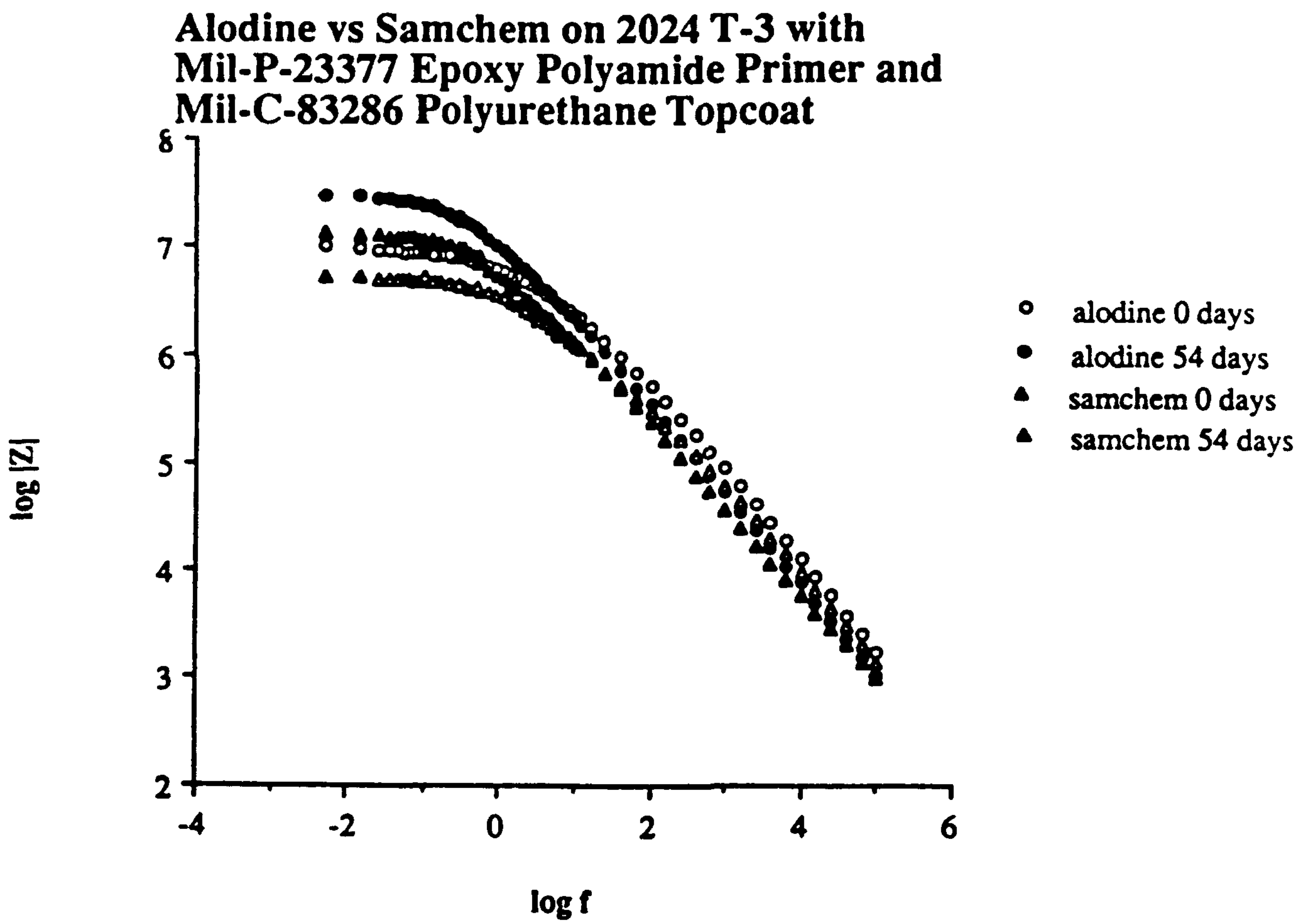

TECHNOLOGY TO WIN

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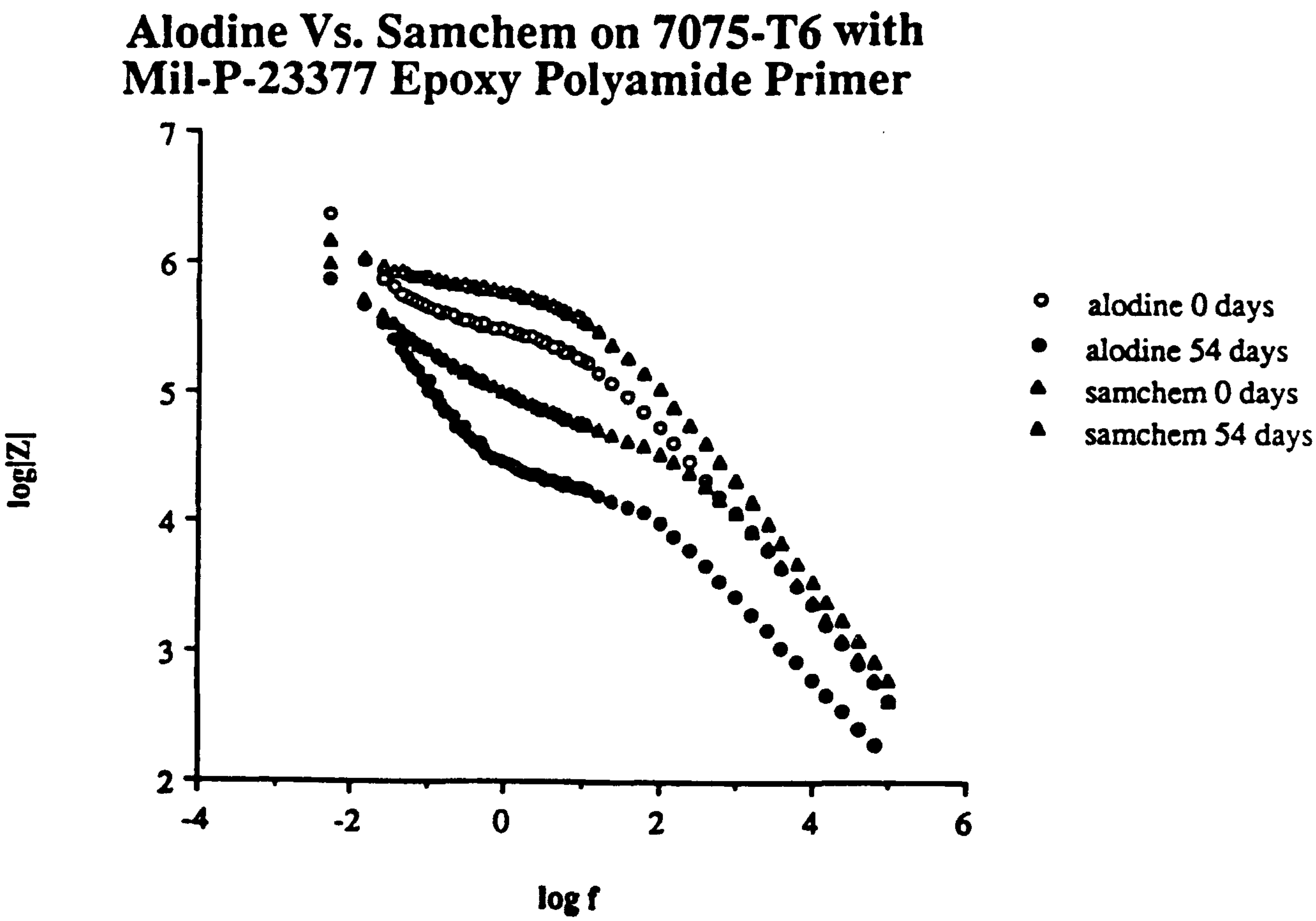

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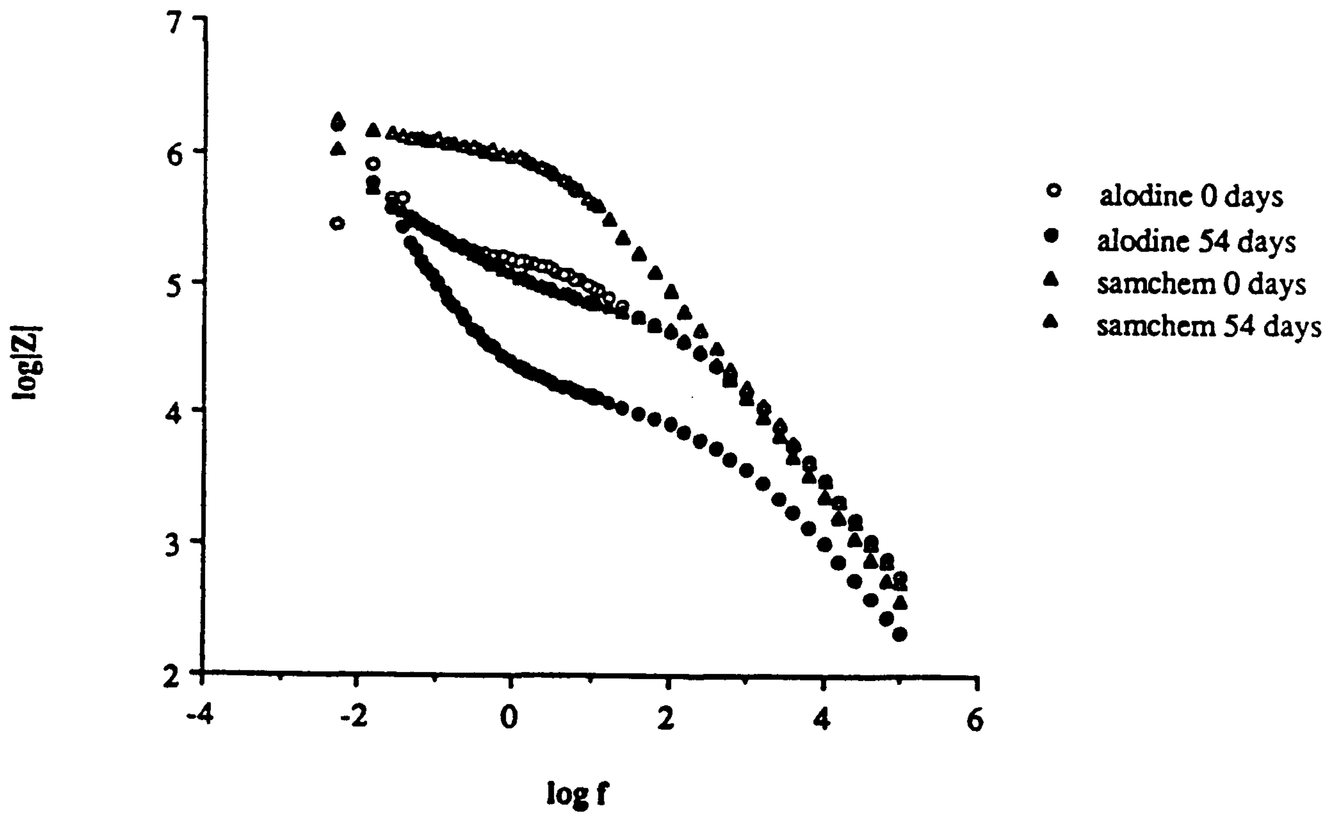
Alodine vs. Samchem on 2024 T-3 with Mil-P-23377 Epoxy Polyamide Primer



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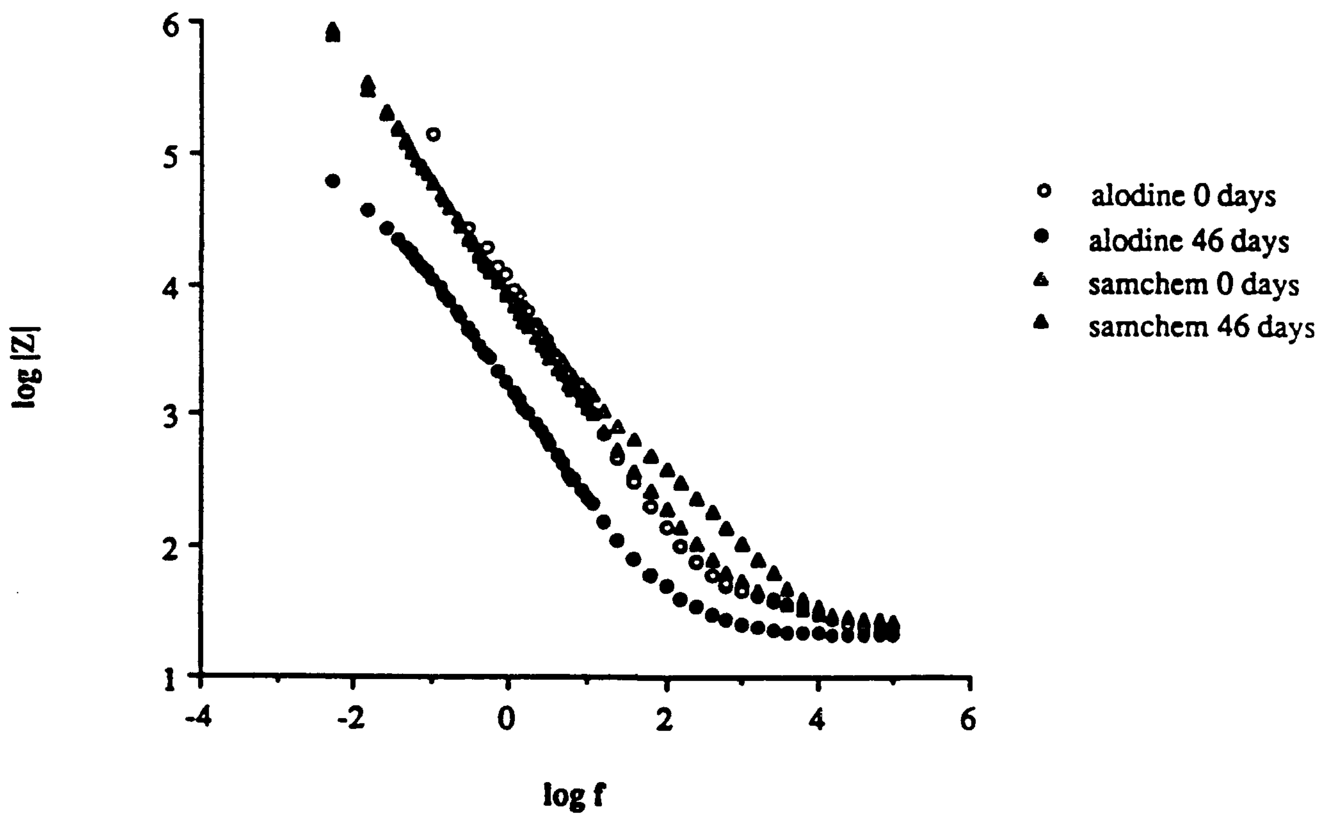
Alodine Vs Samchem on 2024-T3



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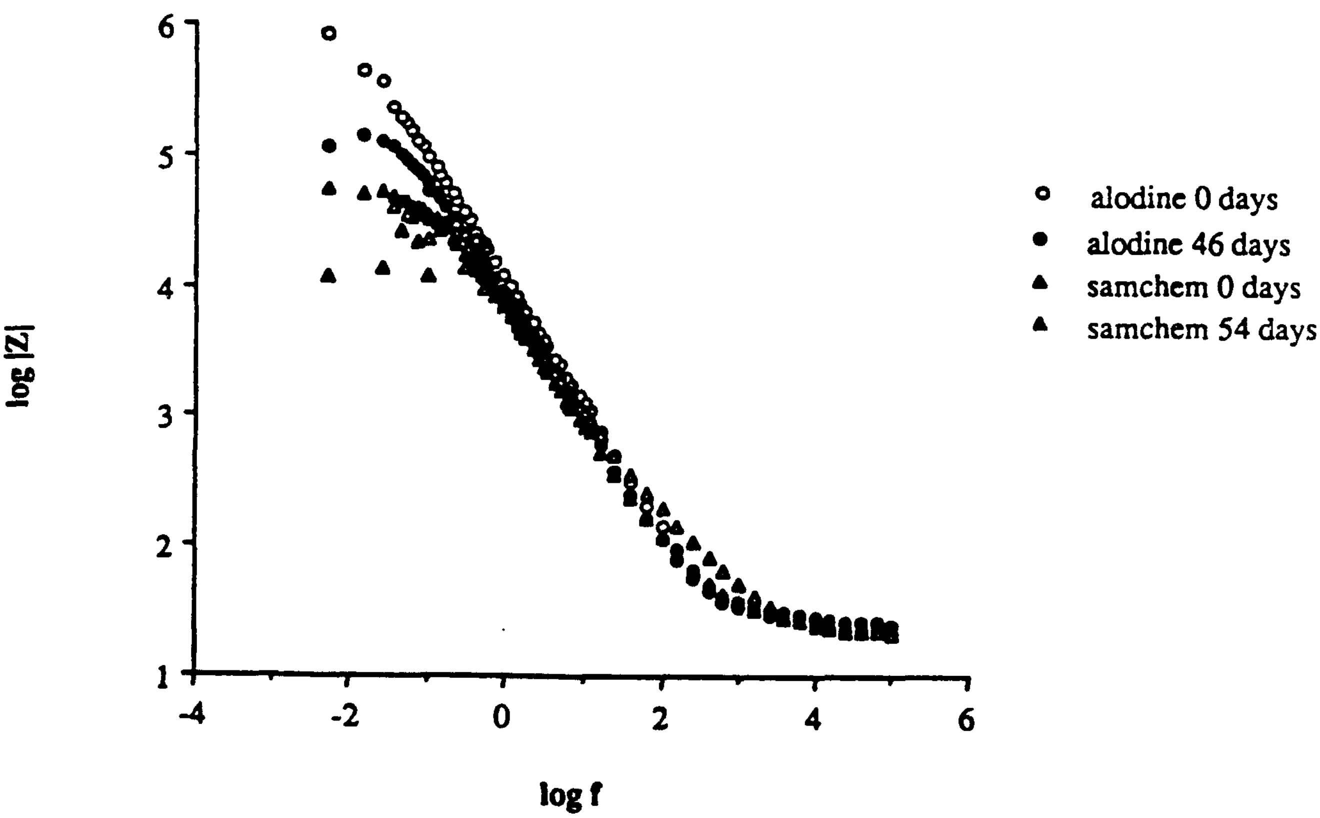
Alodine Vs. Samchem on 7075-T6



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