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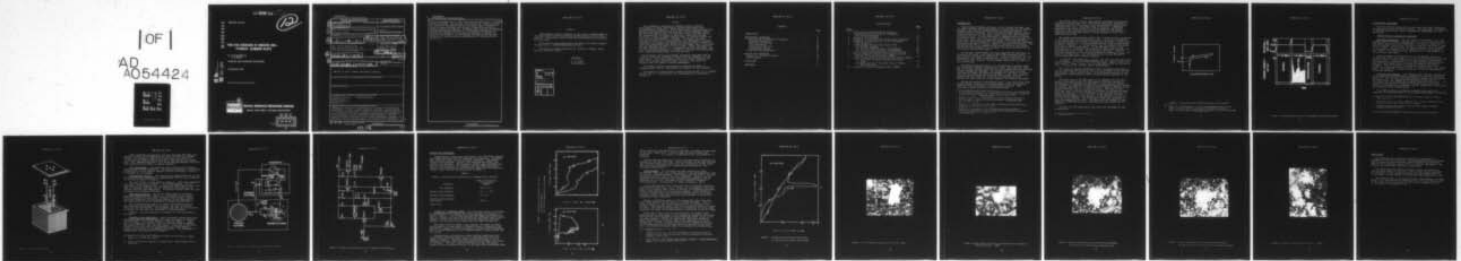
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**THIN FILM CORROSION OF ANODIZED HIGH-STRENGTH ALUMINUM ALLOYS**

BY H. M. DeJARNETTE  
C. R. CROWE

RESEARCH AND TECHNOLOGY DEPARTMENT

23 FEBRUARY 1978

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subject the specimen both to a thin, salty film of moisture and to a controlled electrical potential. The resulting current through the specimen is recorded as a function of this potential and as a function of time. To minimize the effects of random variations, both the order in which tests are run and specimen selection are randomized. Preliminary times-to-failure from aluminum alloy 7075-T73 specimens with sulfuric acid anodizing, with sulfuric acid anodizing plus sealing, and with chromic acid anodizing, corroborate the validity of the procedure. As some specimens survived more than 200 hours before onset of pitting corrosion, planned methods for shortening the test are given.

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SUMMARY

This document reports progress in the study and measurement of the atmospheric corrosion affecting aluminum alloys used in Naval aircraft. Preliminary results from the potentiostatic polarization work are given.

This work is being supported by the Naval Air Systems Command through Task No. A3200000/004A/8R022-00-000.

This work is being reviewed by Dr. Steven G. Fishman, Head, Metallic Materials Branch.

*J.R. Dixon*

J. R. DIXON  
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PREFACE

The atmospheric corrosion of aluminum alloys in marine environments is being investigated using potentiostatic and potentiodynamic techniques. This interim report both describes the apparatus and procedures being used and gives preliminary results. The work was begun to meet the need for a rapid, reliable method for measuring the resistance to corrosion, especially pitting corrosion, of high strength aluminum alloys in various tempers and with various anti-corrosion coatings. At present, the only alternative method for testing materials for atmospheric corrosion resistance is to expose test panels of selected alloys to marine or industrial atmospheres while monitoring the weight loss of the panels and the frequency and depth of pits in them. While this method is reliable and quantitative, it is expensive and time consuming. The test method being developed is designed to preserve these advantages while lowering significantly the time and cost.

Potentiostatic and potentiodynamic techniques have been used by others to study the corrosion of metals that are immersed in bulk electrolyte. This report details the use of these techniques to study the corrosion of metals under the salty, thin layers of moisture that are common to marine atmospheres.

Preliminary results from variously anodized specimens of aluminum alloy 7075-T73 corroborate the validity of the procedure.

The method is centered upon a method devised by Drs. S. G. Fishman (CR-32) and C. R. Crowe (CR-32) for investigation of atmospheric corrosion.

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## INTRODUCTION

High-strength aluminum alloys are vital to naval aircraft, yet the choice of both alloys and tempers is limited because the highest strength (7xxx series) aluminum alloys are generally subject to stress corrosion cracking. Stress corrosion cracks begin at stress concentrations such as the small, sharp cracks that are formed by pitting corrosion. Pitting corrosion of aluminum alloys is abated by chlorine ions, and pits will form on aluminum alloy surfaces even under thin, moist, salt-contaminated films. These circumstances necessitate the development of a procedure for testing the atmospheric corrosion resistance of alloys, tempers, and protection methods. The new method must be as reliable and as quantitative as the present method of on-site exposure of specimens, but the new method must be less costly and far more rapid.

In the past, many methods have been suggested for measuring the resistance of alloys to pitting in short-time laboratory tests. Cyclic anodic polarization techniques<sup>1</sup>, scratch techniques<sup>2</sup>, and pit propagation rate (PPR)<sup>3</sup> techniques are currently being studied. In all of this work, however, the environment chosen for the tests is bulk electrolyte.

The method described herein is a continuation of earlier work<sup>4</sup> that subjected a metallic specimen to a thin, salty moisture layer and to a slowly varying electric field while monitoring the resulting current through the specimen. The earlier work found a strong correlation between the corrosion imposed in a few hours on Armco iron that was polarized while it was under a thin, salty layer of moisture and the corrosion suffered by a specimen of like composition after years of on-site exposure to a marine atmosphere.

During the course of the previous work, it was noted that large current pulses occurred in the polarization curves if localized corrosion such as pitting or crevice attack occurred. This observation forms the basis of the present work.

1. Broli, Hasmund and Holtan, "Use of Potentiokinetic Methods for the Determination of Characteristic Potentials for Pitting Corrosion of Aluminum in a Deaerated Solution of 3% NaCl," Corrosion Science, Vol. 13, 1973, P. 237.
2. Pessall, N. and Liu C., "Determination of Critical Pitting Potentials of Stainless Steels in Aqueous Chloride Environments," Electrochem. Acta, Vol. 16, 1971, p. 1987
3. Syrett, B. C., "PPR Curves - A New Method of Assessing Pitting Resistance," Corrosion, Vol. 33, No. 6, 1977, p. 221.
4. Fishman, S. G. and Crowe, C. R., "The Application of Potentiostatic Polarization Techniques to Corrosion Under Thin Condensed Moisture Layers," Corrosion Science, Vol. 17, 1977, p. 27.

The present report details experimental procedures and presents preliminary results from work with various anodizing treatments of aluminum alloy 7075-T73 using a cyclic potentiostatic polarization technique under thin, salt-laden films of moisture. Additional experiments are being conducted at fixed potential for various lengths of time to assess the validity of using the PPR method to characterize pitting resistance of alloys subject to thin, salt-laden moisture films.

One common electrochemical method that is used to study resistance to pitting is the cyclic polarization test. The alloy is immersed in the environment of interest and anodically polarized to more noble potentials followed by a reverse depolarization using a potentiostat as shown in Figure 1. At the potential,  $E_b$ , the passive film breaks down locally and pitting ensues. If the potential scan is then reversed so as to decrease the potential to more active values, old pits continue to be active, but no new pits initiate<sup>5</sup>. When the potential decreases to the so-called protection potential,  $E_{prot}$ , the active pits repassivate, and the polarization current returns to values consistent with a passive surface.

In general, the higher  $E_{prot}$  and  $E_b$ , the more resistant the alloy is to pitting. If the corrosion potential  $E_{corr} < E_{prot}$ , there is no pitting, and if  $E_{prot} < E_{corr} < E_b$ , pitting occurs after an incubation time. If  $E_{corr} > E_b$ , pitting occurs spontaneously.

The tests are usually performed in bulk, deaerated electrolyte so that on reversing the potential scan, the pits remain active and the polarization current remains high. This produces hysteresis in the polarization curve which is one of the more salient features of the method.

The basic idea of the PPR method is to separate the pitting or crevice corrosion current from the general corrosion current by performing an electrochemical experiment as described in Figure 2. The specimen is anodically scanned to a preselected potential between  $E_b$  and  $E_{prot}$  and held for a time interval to obtain a measure of the general corrosion rate in the passive state. The scan is then continued until a current density of 10 mA/cm<sup>2</sup> is obtained at which point the potential is step decreased to the preselected potential between  $E_b$  and  $E_{prot}$ . It is held for a time interval, and the current density measured now consists of the general rate plus the rate due to pit growth. The potential is then step decreased to  $E_{corr}$  to repassivate the pits. The PPR corrosion current is then obtained by subtraction and is represented by the shaded area in Figure 2.

To date, all PPR experiments have also been performed in bulk electrolytes.

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5. Syrett, op. cit.

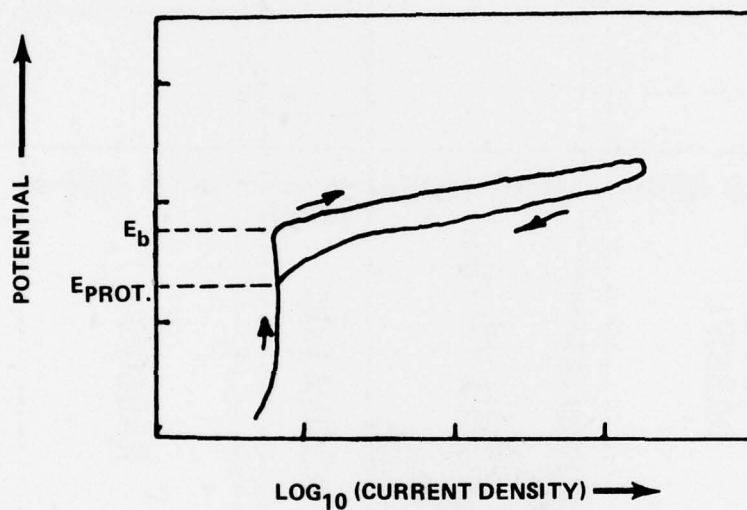


FIGURE 1. Polarization Curve Showing  $E_b$  and  $E_{prot}$  (after Wilde<sup>14</sup>)

14. Wilde, B. E. and Williams, E., "Use of Current/Voltage Curves for the Study of Localized Corrosion and Passivity Breakdown on Stainless Steels in Chloride Media," Electrochem. Acta, Vol. 16, 1971, pp. 1971-1985.

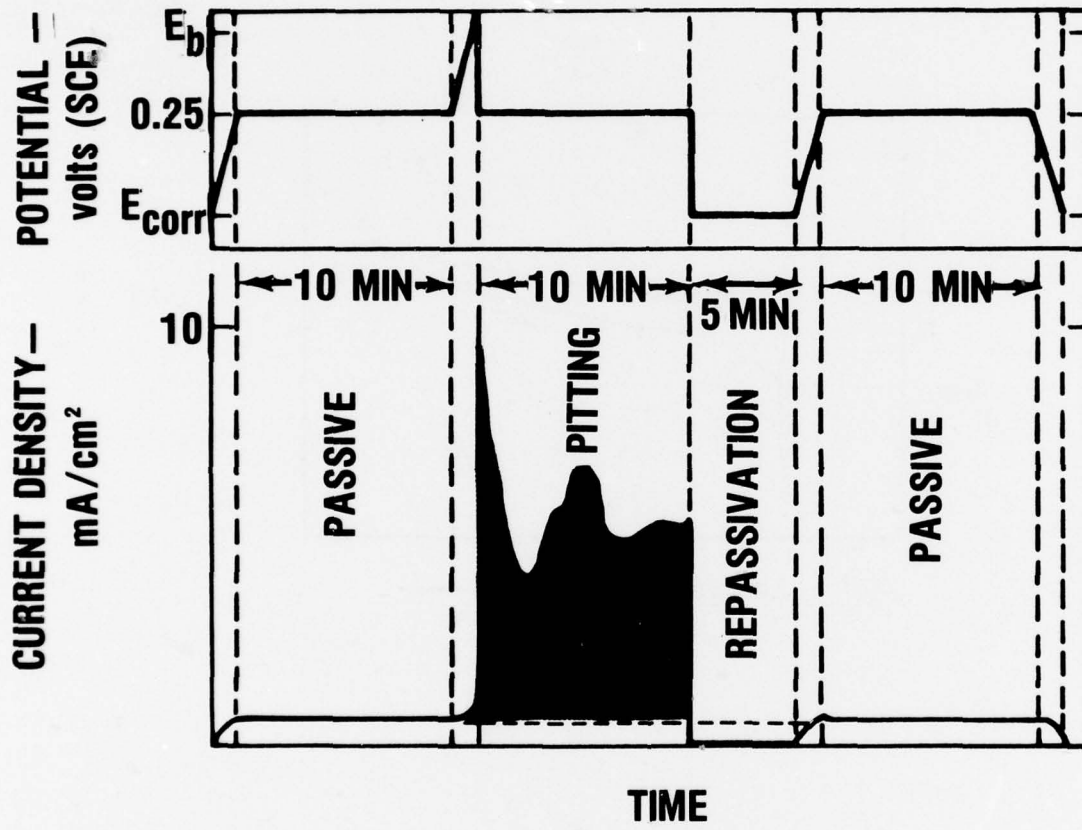


FIGURE 2. Potential-Time Curve from Pit Propagation Rate (PPR) Experiment

## EXPERIMENTAL PROCEDURES

Both the potentiostatic and the potential-time (PPR) techniques are described in the following sections. The paragraphs on anodizing, on specimen preparation, and on experimental design apply to both techniques.

### POTENTIOSTATIC POLARIZATION TECHNIQUE

Specimen Preparation. For each block of experiments, a 9.4 mm diameter dowel of 7075-T6 aluminum alloy is cut in 30 cm long pieces, and sample resistivities are measured\*. The pieces, except for a control specimen, are held at 170-182°C in air for 6-8 hours to gain the T73 temper. Resistivities are again measured, and an increase of six percent in conductivity is taken as proof of achievement of T73 temper<sup>6</sup>. The dowels are then machined to a 6.5 mm diameter to form corrosion specimens.

These specimens are secured into 6.55 mm diameter holes in a 25 mm square plate (See Figure 3), using an acid resistant epoxy that reliably forms a crevice-free bond to the specimen circumference. The dowel face is ground until it is flush with the glass plate by using #600 grit paper with a lubricant of paraffin dissolved in kerosene. This is followed by ultrasonic cleaning for thirty seconds in a detergent-water solution, a tap water rinse, an alcohol rinse, and drying in a stream of warm air. Then the specimens are etched and anodized.

Anodizing and Sealing. For aluminum alloy 7075-T73, samples are being tested in the non-anodized, anodized, or anodized and sealed conditions. Both sulfuric acid anodizing and chromic acid anodizing are being used. All anodizing and sealing is being done in accordance with MIL-A-8625C and standard industrial practice<sup>7,8</sup>. To verify that the proper thicknesses of anodic layers were being achieved, specimens were sectioned, and the thicknesses of the layers were measured using a metallograph.

The aluminum-epoxy interface is examined before and after anodizing at a magnification of 40x to assure the absence of crevices.

6. Mehr, P., et. al., "Alcoa Alloy 7075-T73," Aluminum Company of America, 1971, (Rev. ed.) p. 6.
7. Lyman, Taylor, et. al., Metals Handbook, Vol. 2, 8th ed., American Society for Metals, Metals Park, Ohio, 1964, pp. 620-625.
8. Aluminum Finishes Process Manual, Reynolds Metals Company, Richmond, Virginia, 1973, pp. 112-113

\* Dr. M. A. Mitchell, NSWC/CR-32, performed all resistivity measurements.

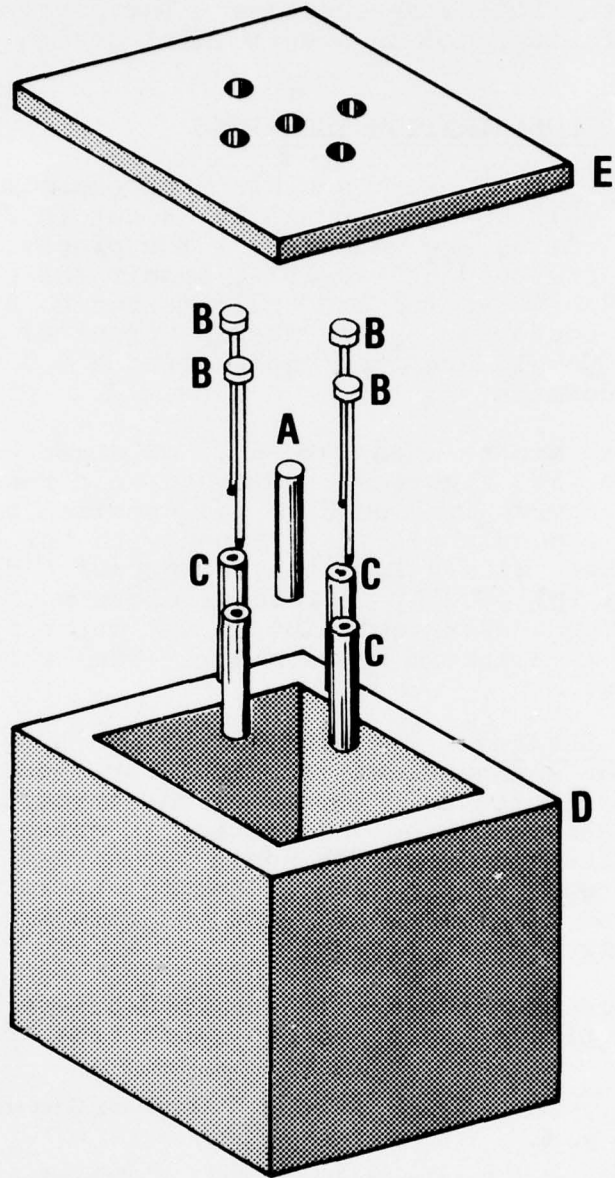


FIGURE 3. Corrosion Cell Assembly

After anodizing the specimen, the four platinum electrodes are mounted in the 6.55 mm diameter holes which surround the specimen. Finally, the assembly is heated by a heat lamp, and a mist of 3.5 percent (by weight) sodium chloride is sprayed over the warmed face. The treatment deposits a salt layer that approximates the daily salt deposition on surfaces in coastal regions<sup>9</sup>.

Test Environment. The corrosion cell is kept at 95% relative humidity and at room temperature. The relative humidity is controlled by placing an 11% (by weight) aqueous solution of sulfuric acid in a container in the chamber.

Potentiostat and Cell. The potentiostat (Weking Model No. HP 10) and stepping motor potentiometer (Wenking Model SMP 69) were connected to the cell as shown in Figure 4.

The cell design is shown above in Figure 3. Tests were conducted in a glove-bag in order to permit: (1) pH measurements, (2) fine adjustment of the Luggin probe, (3) magnified (40x) observation of the face of corroding specimen, and (4) continuous monitoring of the relative humidity and temperature. A saturated calomel (SCE) was used throughout for reference electrode.

Experimental Design. Due to the large number of variables, corrosion measurements are subject to a relatively large scatter. While experimental design cannot eliminate these errors, readily available techniques can provide an estimate of the magnitude of the random errors that are introduced by uncontrolled variables.

The technique being used is to select the anodizing and the anodizing and sealing methods to be tested, and then to schedule three runs for each method. The specimens for each run are selected at random, and the tests are performed in random sequence.

Finally, a one way analysis of variance is to be performed on the data<sup>10</sup>.

POTENTIAL-TIME EXPERIMENTS. Small electronic potentiostats are being fabricated to perform potential-time experiments to evaluate the PPR method. The circuit design is shown in Figure 5. These units are expected to deliver 250 ma at constant anodic potential and should allow for long time PPR studies. Plans are to fabricate several of the units and run simultaneous tests which measure current-time relations at programmed potentials. The experiment is schematically described above in Figure 2.

9. Ambler, H. R. et. al., "Corrosion of Metals in the Tropics," J. Appl. Chem., Vol. 5, 1955, pp. 437-467.

10. "Applying Statistics to Analysis of Corrosion Data," ASTM Standard G 16-71, Section 10.

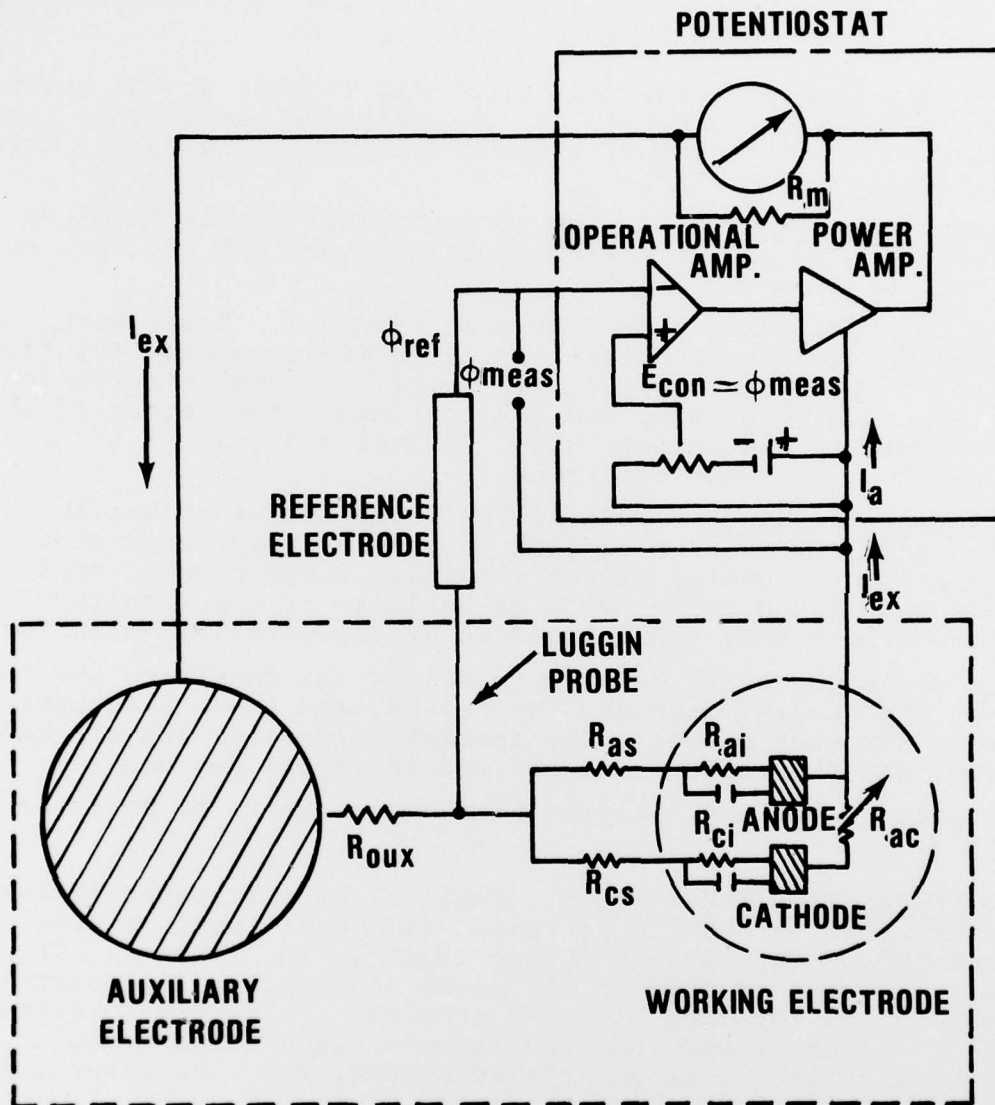


FIGURE 4. Schematic of Potentiostatic Polarization Technique

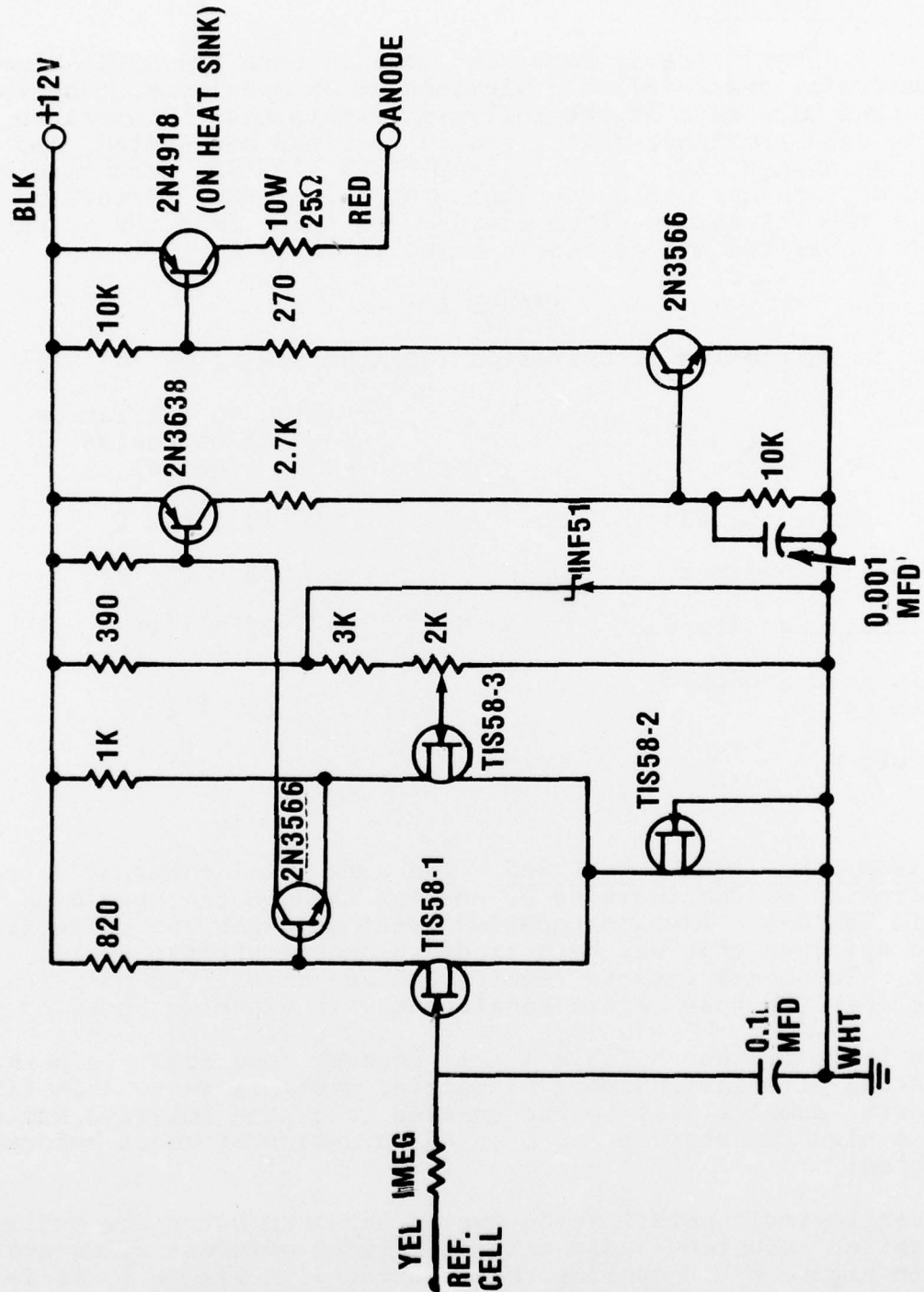


FIGURE 5. Schematic of Potentiostats for Pit Propagation Rate Technique

RESULTS AND DISCUSSION

Potentiostatic traces have been made on pure (99.999%) aluminum and commercial grade 7075-T73 aluminum alloy specimens. Specimens were tested with each of the following treatments: unanodized, sulfuric acid anodized, sulfuric acid anodized and sealed, and chromic acid anodized. Metallographic analysis was performed with the aid of both optical and scanning electron (SEM) microscopy. Times to the initiation of corrosion were taken from the potential current traces and are presented below in Table 1.

TABLE 1

Times to Initiation of Corrosion for Aluminum Alloy 7075-T73

Treatment	Time to Initiation of Corrosion (hours)
not anodized	13.5 ± 1.5
Chromic acid anodized	22 < t < 64*
Sulfuric acid anodized	27.7 ± 1
Sulfuric acid anodized and sealed	262 ± 1

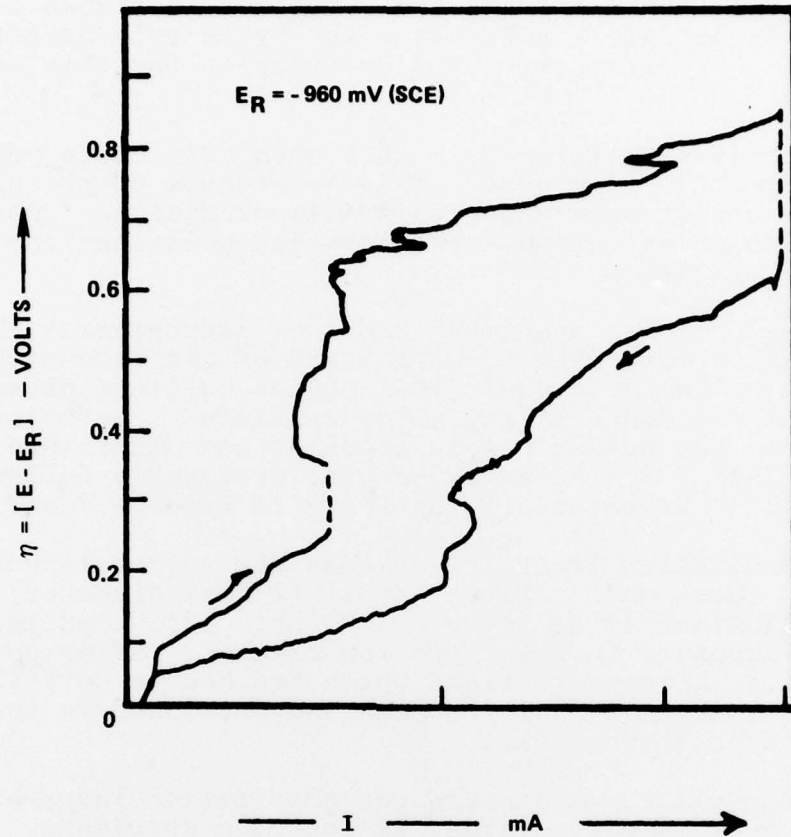
\*power off

CURRENT VS. POTENTIAL CURVES. The time until onset of corrosion, as determined by the increase of current through the specimens, is given in Table 1. The non-anodized specimen began corroding first, and the specimen that was both anodized and dichromate sealed lasted longest. These are interim results and are drawn from only four samples, but the results are consistent with expected behavior.

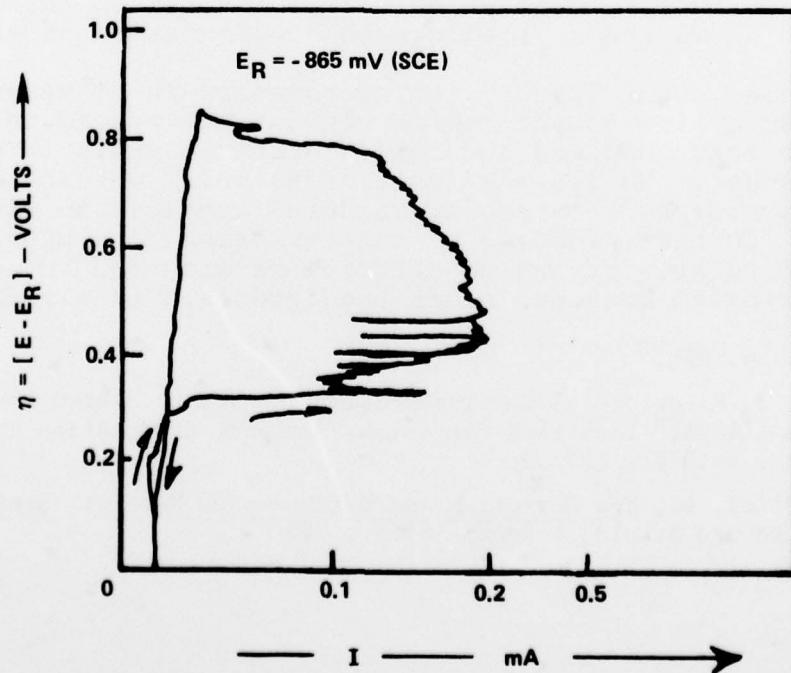
The times listed in Table 1 are, however, too great to permit the testing of a large number of samples within a reasonable time. Henceforth, samples will be pre-exposed to a high relative humidity and to a high temperature for a standard number of hours before they are tested.

Potentiostatic polarization curves obtained using the cyclic polarization technique under thin salt laden moisture films are shown in Figure 6. Comparing these curves with Figure 1, it is immediately obvious that the polarization behavior under thin films is different. The most striking difference is that the anodic current at first increases (on-set of pitting) with increasingly more

FIGURE 6. Potentiostatic Polarization Curves from:  
(a) 99.999% Aluminum  
(b) 7075-T73 Aluminum Alloy, Non-anodized



(a)



(b)

noble potentials then decreases and sometimes increases a second time. In one case, Figure 6(c), a figure eight hysteresis effect was obtained. At present, no explanation for this behavior is proposed.

Another obvious difference is that much more noble potentials are required in the thin film case. This is because of the high solution and interface resistances under thin film conditions<sup>11</sup> which result from activation polarization, concentration polarization, and resistance polarization.

METALLOGRAPHY. All specimens had some corrosion at their periferies. Pits were also found inboard of the circumferential zone. In the pure aluminum specimen, (100) planes outlined pits<sup>12</sup>, but this phenomenon was not found in any alloy specimens. Both in the non-anodized and in the sulfuric acid anodized and dichromate sealed specimens of 7075-T73, the edges of pits frequently followed crystallographic orientations, as shown in Figures 7 and 9.

In the anodization layer, flaws that led to localized corrosion originated in three ways. First, small (2.5  $\mu\text{m}$  diameter) pores were seen in the aluminum oxide layer of sulfuric acid anodized specimens before their exposure in the corrosion chamber. After corrosion testing, similar holes were found which had become so filled with corrosion products that radial cracks had developed in the brittle anodization layer (Figure 8).

Second, curvilinear cracks in the anodization layer have been found above severely pitted areas in corroded specimens. This is shown in Figure 9. The top micrograph (9(a)) is focused on the outside of the aluminum oxide layer; the curvilinear crack is clearly outlined. The lower micrograph (9(b)) is focused on the metal-oxide interface and shows the crystallographic orientation of the pit edges.

Third, holes about five to ten micrometers in diameter were generated when a fine copper<sup>13</sup> wire segment was placed in the center of a sulfuric acid anodized and sealed specimen while it was in the corrosion chamber. Unlike the small holes which were mentioned above and which were isolated, these holes appeared in clusters (Figure 10). Further, instead of the corrosion remaining confined to the immediate area of the penetration as occurred in the first case, the corrosion branched along the interface in all directions.

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11. Fishman, op. cit.

12. Galvele, J. R. et. al., "Critical Potentials for Localized Corrosion of Aluminum Alloys," Localized Corrosion, National Association of Corrosion Engineers, 6-10 Dec 1971.

13. Evans, Ulick, R., The Corrosion and Oxidation of Metals: Second Supplementary Volume, Edward Arnold, London, 1976, p. 76.

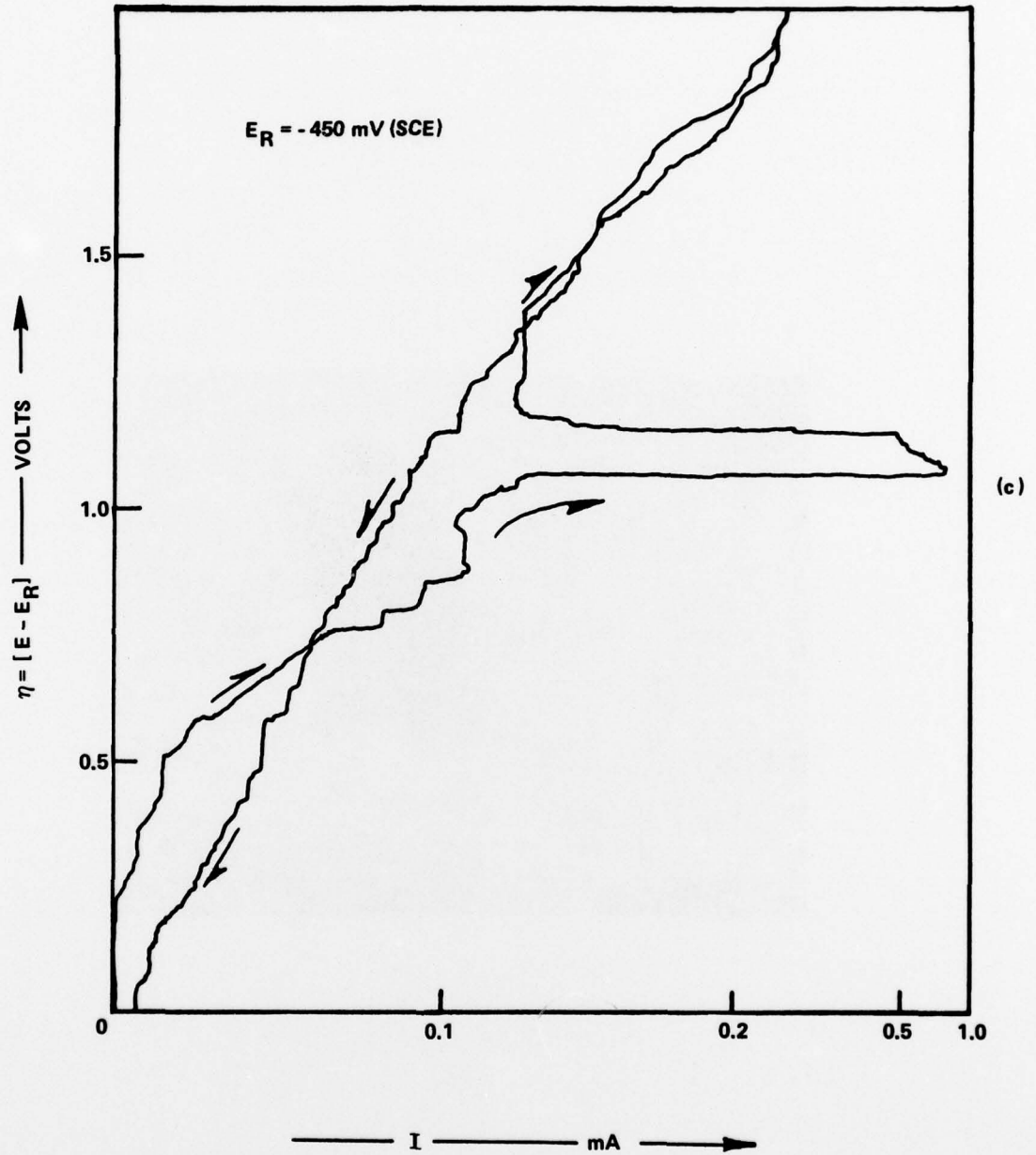


FIGURE 6. Potentiostatic Polarization Curves from:  
(c) 7075-T73 Alloy, chromic acid anodized



FIGURE 7. Pit in Unanodized Aluminum Alloy 7075-T73 (800X)

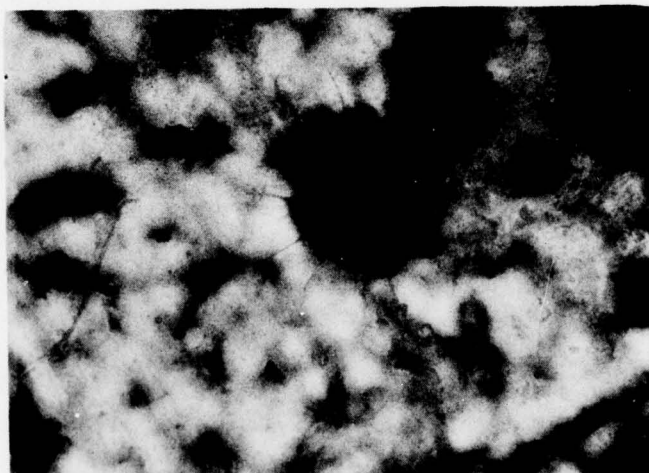


FIGURE 8. Cracks Formed in Sulfuric Acid Anodized Layer by Buildup of Corrosion Products. (800X)

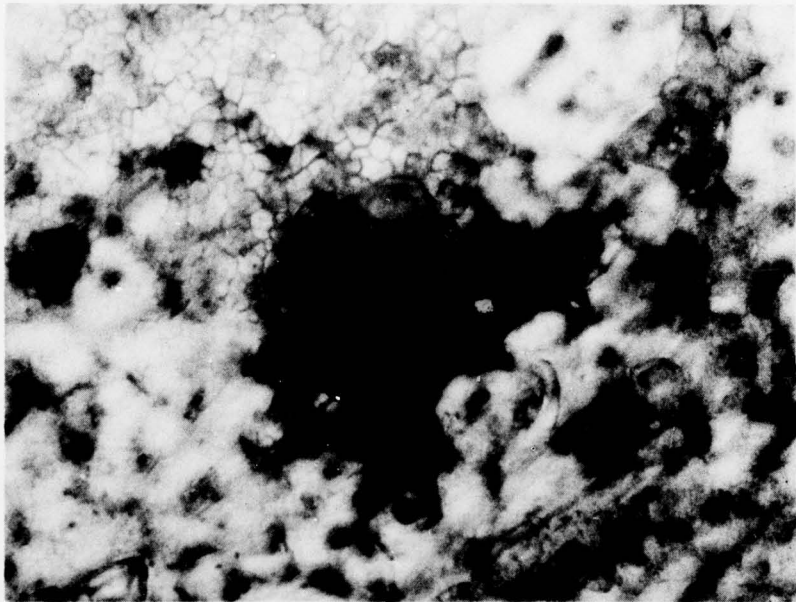


FIGURE 9. Crack in Anodization Layer and Related Pitting (800X):  
(a) Outer Face of Oxide Showing Curvilinear Crack

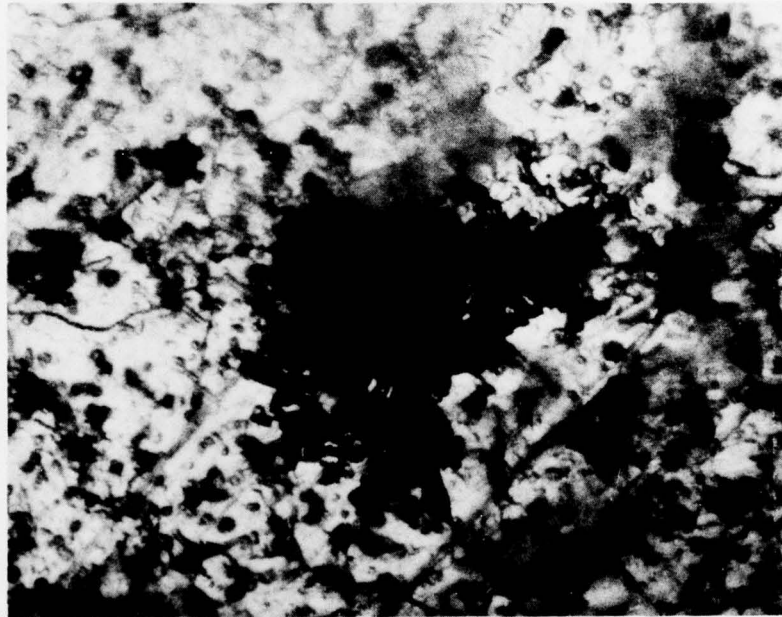


FIGURE 9. Crack in Anodization Layer and Related Pitting (800X):  
(b) Same area as 9(a) but with Focus on Oxide-Metal Interface

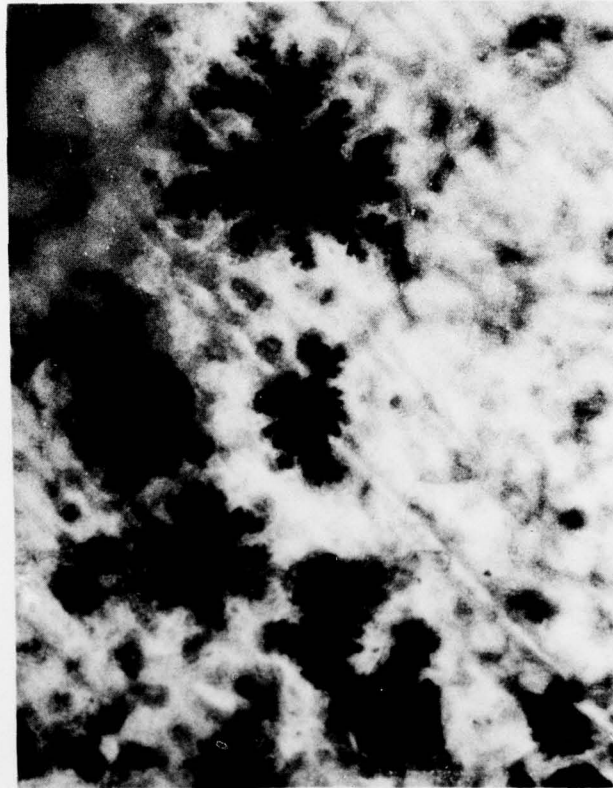


FIGURE 10. Clusters of Holes in Anodized Layer. (880X)

CONCLUSIONS

The potentiostatic method for testing the resistance to atmospheric corrosion of several anodizing methods yields preliminary results which are consistent with those expected from long-term exposure of a similarly treated alloy to a marine atmosphere. The results are preliminary and must be replicated.

The method must be slightly altered in order to shorten the time interval needed for the test. This will be done by exposing the specimens to high relative humidity and high temperature for a set number of hours before the conducting the potentiostatic test.

The anodized layer is breached by at least three methods: (a) small pores in the as-anodized but unsealed layer, (b) curvilinear cracks in the layer, and (c) clusters of small holes through the layer in the vicinity of aggressive ions.

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6. Mehr, P. L. et. al., "Alcoa Alloy 7075-T73," Aluminum Company of America, 1971, (Rev. ed.), p. 6.
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11. Fishman, op. cit.
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