Corrosion of Bare and Coated Al 5052-H3 and Al 6061-T6 in Seawater

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1 INTRODUCTION

1.1 NATURE AND SCOPE OF RESEARCH

The effectiveness of novel hybrid ceramic-polymer or ceramer coatings (i.e., HCLCoat11 and HCLCoat13) developed in the Hawaii Corrosion Laboratory (HCL) on aluminum substrates will be examined. The coating was developed on an Army funded project, and is currently being examined on other DoD projects for applications in the Air Force, Navy, and Army. This study will elucidate the corrosion initiation mechanisms on coated aluminum alloy substrates, as well as determine the corrosion performance of the coated substrates in marine environments. If the coatings are effective, they may be of utility for aluminum heat exchanges in Ocean Thermal Energy Conversion (OTEC) power plants. The coatings are approximately 2 – 10 microns thick, and have shown promise as corrosion barriers in atmospheric and accelerated corrosion tests (i.e., ASTM B117). The corrosion protection results of the ceramer coatings were comparable to that of Alodine 1201-treated coupons (considered an industrial standard). The utility of the ceramer coatings under immersion conditions will be explored in this program. Preliminary tests have shown that the dense ceramic-polymer coatings also have anti-fouling characteristics and resists de-lamination due to covalent bonding with the aluminum substrate. The thinness of the ceramic-polymer coatings may make them suitable for heat-exchanger applications.

Both aluminum alloys Al 5052 and Al 6061 will be examined under the following conditions: 1) Bare; 2) Coated with HCLCoat11 or HCLCoat13; 3) Coated with HCLCoat13 and top coated with epoxy; 4) Coated with HCLCoat13 and top coated with polyurethane; 5) Treated with Alodine 1201 and top coated with epoxy; and 6) Treated with Alodine 1201 and top coated with polyurethane. The coating combinations above will help to determine 1) the performance of HCLCoat11 or HCLCoat13 as stand-alone coatings, 2) the utility of HCLCoat13 as a pretreatment for thicker top coatings for other ocean applications, and 3) the comparison of HCLCoat13 against the industry standard Alodine 1201 type pretreatments for top coats.

Electrochemical, and corrosion exposure tests will be conducted to determine the corrosion mechanisms and corrosion protection properties of the ceramer coatings. Anodic polarization test can provide information on the barrier properties of the coatings. Cathodic polarization test can provide information on the ability of the coatings to support cathodic reactions and sustain corrosion at open-circuit conditions. Immersion testing will be conducted in ASTM simulated seawater in the laboratory; in natural warm surface seawater and in the splash-spray zone at Coconut Island, Oahu; and in cold deep-ocean water at the Natural Energy Laboratory of Hawaii Authority (NELHA), Big Island. Immersion tests in ASTM simulated seawater will provide information on corrosion associated electrochemical corrosion without biological effects. Immersion in natural warm surface water and cold deep-ocean water will provide information on corrosion affected by microbial effects as well as information on biofouling. Exposure to the splash-spray zone will provide corrosion information under severe exposure conditions. Sunlight can deteriorate organic coatings, and thin films of seawater in the splash-spray
zone can result in very high corrosion rates. The evaluation of corrosion degradation will be assessed by weight-loss measurements, profilometry, image analyses, and electron microscopy.

The ceramer coatings will also be examined on larger components for OTEC applications in another program at the OTEC Heat Exchangers (HXs) Facility operated by Makai Ocean Engineering and the Hawaii National Marine Renewable Energy Center (HINMREC). The HCL will be coating specimens for testing at this facility.

1.2 JUSTIFICATION AND BACKGROUND

The required life of the OTEC power plant is approximately 30 years, and therefore materials with sufficient corrosion resistance are needed to meet expected service lives. For example, the longer duration before heat exchangers need to be replaced will reduce overall life-cycle costs. Thus, engineers and designers must consider corrosion resistance in their materials-selection process. Prior research regarding the OTEC project[1] has identified some candidate aluminum alloys based on strength, corrosion resistance and cost. Although some aluminum alloys have good resistance to atmospheric corrosion, pitting corrosion can be more severe in seawater, and therefore coatings that impart corrosion resistance may be needed for immersion conditions.

As a conventional surface treatment for aluminum alloys, chromate conversion coatings are effective to some degree in suppressing corrosion. Chromate conversion coatings, however, may be banned by the Environmental Protection Agency (EPA) in the near future due to the toxicity of hexavalent chromium salts.

Other coatings that may enhance corrosion resistance include organic coatings (e.g., epoxy). A potential concern, however, is that the thickness of organic coatings may hinder their use in heat exchanger applications if they impede heat transfer. Inorganic ceramic coatings are generally thinner, but are brittle and more difficult and expensive to apply.

Relatively novel ceramer coatings have been developed and may have some positive attributes for coatings on aluminum heat exchangers. The incorporation of organic groups into inorganic networks makes it possible to increase ductility and to reduce micro-cracking. Ceramer coatings can also be applied relatively thin (i.e., 2 – 5 µm) by spraying, dipping, or brushing, resulting in lower application costs.
2 MATERIAL SELECTION

2.1 ALUMINUM ALLOY SELECTION

Both Al 5052 and Al 6061 were selected for this study.

It was previously reported by Panchal et al[1] that wrought Al 5052 had good corrosion resistance in seawater. This wrought aluminum alloy of the 5000 series is a strain-hardenable alloy. The composition of Al 5052 is 2.2wt% magnesium, 0.15wt% chromium, and a balance of aluminum [2]. The H3 temper was selected and is generally used for these alloys. The H3 temper produces stable properties with higher elongation levels and improved forming characteristics [3].

Al 6061 was selected as the second substrate alloy due to its good corrosion resistance, availability, and frequency of use. Al 6061 has also showed good adhesion with the ceramer coating [4]. This wrought aluminum alloy of the 6000 series is age hardenable. The composition of Al 6061-T6 is 0.8wt% magnesium, 0.4wt% silicon, 0.15wt% copper, 0.04wt% chromium with a balance of Al [2]. The T6 temper indicates solution heat treated and artificially aged to peak hardness.

2.2 CHROMATE CONVERSION COATING

To compare the performance of the ceramer coatings to the industry standard, specimens treated with a chromate conversion coating were also examined. ALODINE 1201 was selected due to its high performance having good adhesion to aluminum substrates and excellent corrosion resistance [5, 6, 7]. Alodine 1201 is a nonflammable, chromic acid-based treatment that reacts with the aluminum substrate leaving a finish in gold or tan color.

2.3 CERAMIC-POLYMER COATING

The HCL has developed and evaluated novel ceramer coatings [4, 8]. This study uses two variations HCLCoat11 and HCLCoat13. HCLCoat11 is a quasi ceramic coating, having little to no hydrocarbons in the structure. HCLCoat13 has more hydrocarbons in the coating structure, providing better adhesion to topcoats.

2.4 ORGANIC COATING SELECTION

For immersion and outdoor exposure test, two organic top coatings were selected: A polyamide cured epoxy coating and a polyurethane coating.
2.4.1 EPOXY

The epoxy coating was a polymer of epichlorohydrin and bisphenol A designated as D.E.R. 331 (Dow Chemical, USA) with the epoxy curing agent Ancamide 2325 (Air Products, USA). This epoxy resin and curing agent are widely used for aluminum alloy and are discussed in a few publications [9, 10].

2.4.2 POLYURETHANE

Polyurethane coatings generally provide good resistance to sunlight. In this study, two polyurethane coatings were used. The first coating was a two-part, water-borne linear polyurethane enamel designated as WR-LPU Urethane Topcoat (System Three, USA), and is widely used in marine environments. The other coating was a two-component, solvent-based polyurethane designated as CONATHNE CE-1155 (CYTEC, USA) that provides outstanding degradation and abrasion resistance and is qualified to MIL-I-46058.

3 METHODS AND PROCEDURES

3.1 IMMERSION AND OUTDOOR EXPOSURE TEST

3.1.1 COUPONS

3.1.1.1 Manufacturing of Coupons

Test coupons were manufactured in the College of Engineering machine shop. A total of 624 coupons were processed for testing. Figure 1 shows the shape and size of the coupon.

![Figure 1: Coupon dimensions](image)

Manufacturing of the aluminum coupons was performed using the following procedure: Coupons were cut into 2.125” by 48” strips with hydraulic shear, and ground with 180 grit aluminum oxide paper. The strips were then sheared to produce 2.125” by
1.125” coupons. The coupons were then milled to a final size of 2” x 1” to remove the sheared edges.

3.1.1.2 Surface Preparation of Coupons

Surface preparation is required to improve coating adhesion. To remove oils, greases, salts, and other contaminants, a non-flammable phosphoric acid based cleaner designated as ALUMIPREP 33 (Henkel, USA) was used. The surface preparation was done as following steps: (1) ALUMIPREP 33 was diluted with purified water in 25 parts of ALUMIPREP 33 and 75 parts of water. (2) Coupons were dipped in the dilute ALUMIPREP 33 solution for 2 minutes (1 minute per side). (3) The coupons were then rinsed with water. (4) The coupons were cleaned in a sonic cleaner for 5 minutes. (5) The coupons were then rinsed with purified water. (6) The coupons were dried. (7) The coupons were finally wiped with ethanol. The procedure is shown in Figure 2.

![Surface preparation steps](image)

(a) Dipped in dilute solution  (b) Rinsed with tapped water  (c) Cleaned by ultra-sonic cleaner

(d) Rinsed with purified water  (e) Dried with paper towel  (f) Wiped with ethanol

Figure 2: Surface preparation

3.1.1.3 Chromate Conversion Coating

After surface preparation by ALUMIPREP 33, 198 of the 624 coupons were treated with Alodine 1201: (1) The ALODINE 1201 solution was prepared by mixing 25 parts of ALODINE 1201 and 75 parts of purified water. (2) Coupons were dipped for three minutes. (3) The coupons were rinsed with purified water. (4) The coupons were then dried. The procedure is shown in Figure 3.
3.1.1.4 Ceramer Coating: HCLCoat11 and HCLCoat13

A total of 198 coupons were coated with HCLCoat11 and another 198 coupons were coated with HCLCoat13. Both coatings procedures were similar: (1) The coupons were dipped in HCLCoat11 or HCLCoat13 for 10 seconds. (2) The coupons were then cured at room temperature ($\approx 25^\circ C$) for two days then at $37^\circ C$ for four days. The procedure is shown in Figure 4. Complete curing can also be conducted at room temperature for one week.
3.1.1.5 Epoxy Coating

The epoxy coating was composed of epoxy resin (D.E.R. 331)/curing agent (Ancamide 2325)/solvent (MEK) of 5/3/5 by weight. After prior coating with Alodine 1201 or HCLCoat13, the epoxy coating was applied using the following steps: (1) The coupon was dipped in epoxy for 10 seconds. (2) The coupon was dried at 35°C for 8 hrs.

3.1.1.6 Polyurethane Coating

For chromate conversion coating coupons, the water-borne polyurethane: WR-LPU polyurethane topcoat was used. A solvent-based polyurethane: CONATHNE CE-1155 was used for the HCLCoat13 coupons due to the hydrophobicity of HCLCoat13.

Both coating procedures were as follows: (1) A coupon was dipped in polyurethane batch for 10 seconds. (2) The coupon was dried at room temperature of 25°C.

3.1.2 IMMERSION AND OUTDOOR EXPOSURE TEST

To evaluate the effectiveness of the ceramer coatings against corrosion in marine environments, six coating schemes and bare coupons were tested. The six coating schemes are HCLCoat11; HCLCoat13; HCLCoat13 as under coating with epoxy as topcoat; HCLCoat13 as undercoat with polyurethane as topcoat; chromate conversion coating as undercoat with epoxy as topcoat; and chromate conversion coating as undercoat with polyurethane as topcoat.

The coated and bare coupons were prepared as shown in the following sections. The coupons were immersed in artificial ASTM seawater in the laboratory, and will be exposed outdoor at 3 field test sites for two test periods (4 month and 8 month). The number of coupons per substrate and per test period is shown in Table 1. Thus, the total number of coupons is 624 coupons (i.e., 156 x two substrates x two test periods).

<table>
<thead>
<tr>
<th>Table 1 the number of coupons at each test site</th>
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<tbody>
<tr>
<td></td>
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<tr>
<td>ASTM artificial seawater</td>
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<tr>
<td>Splash Spray in Coconuts Island</td>
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<tr>
<td>Warm seawater in Coconuts Island</td>
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<tr>
<td>Cold seawater in Big Island</td>
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To compare corrosion damage after immersion and outdoor exposure, the weight loss and quantitative damage analysis by a Video Image Enhanced Evaluation
Weathering (VIEEW) system will be conducted. The VIEEW system uses image analyses to assess corrosion damage. All of the specimens have been digitally scanned prior to exposure, and will be scanned again after the immersion period. Moreover, corrosion sites on the surface of coupons will be analyzed by SEM, optical microscopy, and Raman spectroscopy.

3.1.2.1 PROCEDURE OF IMMERSION AND OUTDOOR EXPOSURE

All coupons excluding bare coupons were scribed on one side as shown in Figure 5 to assess undercutting of the coating and localized corrosion.

![Figure 5: Dimensions of the scratch on the coupon](image_url)

3.1.2.1.1 ASTM Artificial Seawater

The setup for testing in artificial ASTM seawater is shown in Figure 6. The temperature of the artificial seawater is sustained at 30°C using a heater and water circulator (pump). The artificial seawater and specimens are contained in beakers with water circulation on the outside to maintain the exposure temperature of 30°C.
3.1.2.1.2 Splash Spray at Coconuts Island

The coupon rack will be mounted as shown in Figure 7. The racks have been fabricated and deployment is scheduled for the week of 9/18/2011.

Figure 7: Method of mounting coupons for splash spray test

3.1.2.1.3 Warm Surface Seawater at Coconuts Island

The specimen racks are being fabricated, and deployment is being scheduled for the week of 9/25/2011.
3.1.2.1.4 Cold Seawater at Big Island

The specimen racks are being designed, and will be encased in large diameter clear PVC tubing through which cold, deep-ocean water will be pumped through at NELHA.

3.2 POLARIZATION TEST

3.2.1 ELECTRODES

The following sections describe the procedure for fabricating the aluminum electrodes Figure 8.

Figure 8: Three electrodes; the metallic area is 1cm²

3.2.1.1 Bare Electrode

Electrodes 1/8” thick, and 1.0 cm x 1.0 cm were cut from aluminum sheets. The electrodes were cut using a Buehler Isomet low speed saw with a Buehler diamond wafering blade (series 15 LC diamond). The corners of the electrode coupon were filleted by wet-grinding on a Buehler Ecomet 6 variable speed grinder-polisher using Buehler-Met II silicon carbide 180-grit grinding paper. The electrode coupon was attached to copper wire with silver conductive epoxy (MG Chemicals) and cured at 70°C for 20 minutes. The copper wire was shielded by using approximately 20 cm length of borosilicate glass tubing. A clear epoxy patch adhesive was used to seal the edges of the electrode, and attach the electrode to the glass tubing. Care was taken to ensure that the tubing end was sealed with epoxy, and that no copper wire was exposed. The epoxy was cured in the oven at 70°C for 1.5 hour, and then removed and allowed to cool to room temperature. The exposed face of the aluminum electrode coupon was wet-ground using Buehler Ecomet 6 variable speed grinder-polisher. The grinding sequence was done sequentially as follows: 180-grit, 320-grit, and 600-grit. All of the grinding papers used were Buehler-Met II silicon carbide grinding paper. After grinding with 600-grit paper, the electrode coupon surface was rinsed with purified water. Polishing was done on the Buehler Ecomet 6 variable speed grinder-polisher in the following sequence: 1µm and 0.3µm Buehler de-agglomerated alpha alumina.
3.2.1.2 Coated Electrode

To investigate the electrochemical properties of the aluminum alloy coated with the ceramer coating, the electrodes were coated with either HCLCoat11 or HCLCoat13. Bare electrodes were dipped into HCLCoat11 or HCLCoat13 for 10 seconds. The coated electrodes were then dried at room temperature of 25°C for 2 days, and then placed in a dry box.

3.2.2 POLARIZATION TEST

3.2.2.1 Electrolyte Preparation

Solutions of 0.5M sodium sulfate and 3.15 wt% NaCl were prepared with ultrapure water (18 MΩ•cm) and Fisher Scientific Certified A.C.S sodium sulfate and NaCl, respectively. The initial solution pH was approximately 7.

3.2.2.2 Polarization Procedure

Deaerated electrolytes were sparged with compressed nitrogen and aerated electrolytes were sparged with compressed air. Prior to starting the polarization experiments, the uncoated electrodes were re-polished and soaked in ultrapure water for approximately 10 minutes to ensure that the structure of the electrode surface was consistent for the set of experiments. During experiments, the electrolytes were kept at a constant temperature of 25 °C. A three-electrode electrolytic cell with a platinum counter electrode and saturated Calomel reference electrode was used. The open-circuit potential of the electrodes was stabilized and measured for 1 hour using a Potentiostat/Galvanostat Model 273 (Princeton Applied Research) prior to starting the potentiodynamic polarization tests at a scan rate of 1mV/s. Potential and current were recorded using the Electrochemistry PowerCORR software (Princeton Applied Research) on a Windows system. The polarization curves shown in this report were the average of three polarization curves conducted with the same test conditions. The error bars on each side of the curve are one standard deviation.

4 STATUS AND CURRENT RESULTS

4.1 IMMERSION AND OUTDOOR EXPOSURE TEST

The ASTM artificial seawater immersion test has been in progress for approximately 3 months. The first set of specimens will be removed and analyzed after 4 months exposure, and the second set will be removed and analyzed after 8 months of exposure. The warm surface water, splash-spray zone, and deep-cold water tests will be initiated shortly. The scheduled times for the tests are 1) week of 9/18/2011 for the warm surface water test, 2) week of 9/25/2011 for the splash-spray zone test; 3) and the month of 10/2011 for the deep-cold water tests.
4.1.1 **WEIGHT LOSS MEASUREMENT**
All of the specimens have been weighed prior to exposure.

4.1.2 **QUANTITATIVE DAMAGE ANALYSIS**
Both surfaces of all coupons were scanned using the VIEEW analyzer prior to immersion and outdoor exposure.

4.2 **POLARIZATION TEST**

Anodic polarization tests were conducted for bare, HCLCoat11 and HCLCoat13 coated electrodes in deaerated and aerated chloride-free solution (0.5 M sodium sulfate solution) and in deaerated and aerated chloride-containing solution (3.15 wt% NaCl solution). Anodic polarization tests of all test conditions have been complete, and cathodic polarization tests are in progress.

4.2.1 **ANODIC POLARIZATION**

4.2.1.1 **0.5 M Sodium Sulfate Solution**

Anodic polarization diagrams for bare, HCLCoat11-coated, and HCLCoat13-coated Al 5052-H3 electrodes are compared in Figures 9 and 10.

Both figures illustrate how anodic polarization diagram of the Al 5052-H3 alloy varies with the influence of the coatings.

Both HCLCoat11 and HCLCoat13 generally suppressed the dissolution rate of the Al 5052 substrate from the open-circuit potential to 0 V_{SCE} (deaerated) and 0.5 V_{SCE} (aerated). However, there was significant scatter in the data for the coated specimens, and addition polarization tests are planned.

Anodic polarization diagrams for bare, HCLCoat11 coated, and HCLCoat13 coated Al 6061-T6 electrodes are compared in Figures 11 and 12.
Figure 9: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 5052-H3 in deaerated 0.5M sodium sulfate solution at 25.5°C

Figure 10: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 5052-H3 in aerated 0.5M sodium sulfate solution at 25.5°C
Both HCLCoat11 and HCLCoat13 generally suppressed the dissolution rate of the Al 6061 substrate from the open-circuit potential to 0.25 V_{SCE} (deaerated and aerated). However, there was significant scatter in the data for the coated specimens, and additional polarization tests are planned.

The comparison of anodic polarization diagrams of bare Al 5052-H3 and Al 6061-T6 are shown in Figure 13 and 14. Al 5052-H3 alloy has slightly lower passive current densities compared to Al 6061-T6.
Figure 12: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 6061-T6 in aerated 0.5M sodium sulfate solution at 25.5°C

Figure 13: Comparison of mean anodic polarization diagrams of bare Al 5052-H3 and Al 6061-T6 in deaerated 0.5M sodium sulfate solution at 25.5°C
4.2.1.2 3.15wt% NaCl Solution

Anodic polarization diagrams for bare, HCLCoat11-coated, and HCLCoat13-coated Al 5052-H3 electrodes are compared in deaerated (Figure 15) and aerated (Figure 16) 3.15 wt% NaCl.

On bare Al 5052 in deaerated 3.15wt% NaCl solution, the pitting potential was approximately -0.73V_{SCE}. In aerated 3.15wt% NaCl solution, oxygen reduction polarizes the Al 5052 to its pitting potential, which is coincident with the open-circuit potential.

For electrodes coated with HCLCoat11 and HCLCoat13, their pitting potentials are almost same as the bare electrodes; however, the current densities are less by 1 to 2 orders of magnitude.
Figure 15: Comparison of mean anodic polarization diagrams of bare, HCLCoat11-coated, and HCLCoat13-coated Al 5052-H3 in deaerated 3.15wt% NaCl solution at 25.5°C
Figure 16: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 5052-H3 in aerated 3.15 wt% NaCl solution at 25.5°C

The anodic polarization diagrams of bare, HCLCoat11-coated and HCLCoat13-coated Al 6061-T6 are compared in deaerated (Figure 17) and aerated (Figure 18) 3.15 wt% NaCl.

The pitting potentials were approximately -0.7 V_{SCE} in deaerated 3.15 wt% NaCl, and coincident with the open-circuit potentials in aerated 3.15 wt% NaCl. HCLCoat11 and HCLCoat13 also reduced the current densities compared to bare Al 6061-T6 by 2 or 3 orders of magnitude.

The comparisons of the anodic polarization diagrams of Al 5052-H3 and Al 6061-T6 in deaerated (Figure 19) and aerated (Figure 20) 3.15 wt% NaCl indicated that Al 5052-H3 has slightly better corrosion resistance compared to Al 6061-T6, as gauged by anodic polarization diagrams, but this does not necessarily indicated that Al 5051-H3 will out perform Al 6061-T6 in the field.
Figure 17: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 6061-T6 in deaerated 3.15wt% NaCl solution at 25.5°C.

Figure 18: Comparison of mean anodic polarization diagrams of bare, HCLCoat11, HCLCoat13 coated Al 6061-T6 in aerated 3.15wt% NaCl solution at 25.5°C.
Figure 19: Comparison of mean anodic polarization diagrams of bare Al 5052-H3 and Al 6061-T6 in deaerated 3.15wt% NaCl solution at 25.5°C

Figure 20: Comparison of mean anodic polarization diagrams of bare Al 5052-H3 and Al 6061-T6 in aerated 3.15wt% NaCl solution at 25.5°C
5 SUMMARY AND FUTURE WORK

5.1 IMMERSION AND OUTDOOR EXPOSURE

The short term (4 months) ASTM artificial seawater immersion test will be completed in October 2011. Analyses by optical microscopy, SEM, Raman spectroscopy and FTIR spectroscopy will conducted to help elucidate corrosion initiation sites, and degradation of the ceramer coatings. The second batch of specimens will be removed and analyzed after 8 months of exposure.

Field studies in warm surface waters and in the splash-spray zone at Coconut Island, and in the cold-deep ocean water at NELHA will shortly be in progress.

5.2 POLARIZATION

The pitting potentials for the HCLCoat11-coated and HCLCoat13-coated Al 5052-H3 and Al 6061-T6 range from approximately -0.6 and -0.7V_{SCE}. Two plausible explanations for the pitting corrosion on the coated electrodes are presented here. First, the coating might breakdown when the applied potential of the polarization scan imparts an electric field on the electrode and exceed the dielectric strength of the coatings. Second, anodic currents emanate from preexisting breaches on the ceramer-coated substrates caused by mechanical damage or by the presence of intermetallics in the alloy. Metallographic investigations will elucidate the pitting corrosion mechanisms on the coated electrodes.

Cathodic polarization experiments are currently in progress. These results will help to determine corrosion rates of the coupons in the open-circuit condition.
6 REFERENCE


