ELECTRIC POWER
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Accelerated Corrosion Testing of BORAL®

LICENSED MATERIAL

Accelerated Corrosion Testing of BORAL®

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REPORT SUMMARY

BORAL® sheets consist of a core of uniformly mixed and distributed boron carbide and alloy 1100 aluminum particles with the core clad on both sides by thin aluminum sheaths serving as solid barriers. BORAL has been used widely for reactivity control in high-density spent nuclear fuel storage racks. This report presents the results of accelerated cyclic corrosion testing of BORAL specimens under nominally low, moderate, and high borated water flow conditions in order to determine the impact of such cyclic exposure on corrosion.

Background

Blister formation and corrosion pits in the cladding of BORAL surveillance coupons have been observed at a U.S. nuclear plant. Blister formation occurs when the BORAL cladding separates from the Al/B_4C core region of the material. Accelerated corrosion could occur in the dryout period when the coupons are out of the pool. BORAL has significant open porosity in the core region that can retain water while the coupons are out of the pool. This moisture is exposed to an oxygen-rich atmosphere (air), which would raise the dissolved oxygen in the internal moisture to ~9 ppm at 20°C, compared with dissolved oxygen levels in the spent fuel pool that are typically in the range of 3 – 5 ppm. Given the importance of BORAL in ensuring criticality safety in spent fuel pools, the EPRI Neutron Absorber Users Group (NAUG) sponsored this phase of cyclic corrosion testing.

Objective

To design and carry out accelerated corrosion testing in order to investigate the effects of cyclic exposure of BORAL to boric acid solution on its corrosion rate relative to continuous exposure.

Approach

The research team designed and custom fabricated two sets of three capsules containing a BORAL specimen. These capsules replicated the geometry of the capsules containing the surveillance coupons on which blister formation and corrosion pits had been originally observed. One capsule in each set had a tight fitting that limited flow between the internal capsule volume and the bulk corrosion bath volume. The other two capsules in each set had a flow path through the capsules formed by stainless steel disks simulating blisters in the BORAL cladding.

The team subjected one set of capsules to continuous exposure to boric acid (2500 ppm) at 195°F for four months. They subjected the other set to one-month exposure in this environment followed by drying in a desiccator for two days and exposure to the laboratory atmosphere for 28 days. This cyclic exposure was repeated four times so that each set was exposed to the corrosion bath for a total of four months. Before and after testing, the BORAL coupons were subjected to visual inspection, high-resolution digital photography, thickness measurements, and weight measurements.

Results

The test data conclusively demonstrated that capsules with some flow permitted result in higher corrosion rates on BORAL coupons than tight fitting capsules with limited flow. This phenomenon is attributed to a constant source of oxygen-rich water from the corrosion bath around the BORAL in the capsule. Such a result confirms the correlation between blister volume and the extent of corrosion observed on the actual surveillance coupons.

The coupons subjected to cyclic exposure experienced greater corrosion rates than those subjected to continuous exposure. However, the observed acceleration of corrosion was small.

A significant result of this work relates to an understanding of the mechanism of blister formation in BORAL. A total of three blisters on two coupons developed during the course of these tests. Since the tests were conducted under isothermal conditions and the temperatures were always less than boiling (195°F), the blisters could not have been caused by steam formation in the BORAL core. This suggests hydrogen formation—a by-product of corrosion of the internal pore surfaces in the BORAL core—as the likely cause of blisters.

EPRI Perspective

Potential causes for the observed accelerated corrosion in some BORAL surveillance coupons in a U.S. PWR could include, among others, gamma heating and the atypical cyclic exposure conditions to which the surveillance coupons have been subjected. Gamma heating was investigated, and the results—documented in EPRI report 1016641, *Thermal Analysis of BORAL® in Storage Racks*—show that gamma heating cannot explain the acceleration in corrosion. The exposure conditions tested in the current project, although clearly observable, did not produce an effect commensurate with the magnitude of the acceleration observed on the surveillance coupons. One potential cause could involve a synergism between cyclic exposure and specific material characteristics of some surveillance coupons. Further work will be required for a clear understanding of the observed phenomena.

Keywords

BORAL®
Spent fuel pool
Accelerated corrosion
Blistering
Pitting

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1 INTRODUCTION

BORAL® has been used extensively for reactivity control in high-density spent nuclear fuel storage racks. Once installed during the fabrication process, the BORAL is generally sealed within the rack structure and cannot readily be subjected to in-service inspection. Accordingly, when new fuel storage racks are installed, a series of surveillance coupons of the neutron absorber material are placed in the racks at that time. The coupons are housed in a specially designed assembly and placed in a storage cell surrounded by recently discharged spent fuel assemblies. Periodically, one or two coupons are removed from the assembly and sent to a laboratory for testing and inspection. Normally the coupons are not returned to the spent fuel pool after testing.

In August of 1998, NextEra Energy Resources installed additional spent fuel storage racks in the Seabrook spent fuel pool to increase its storage capacity. The racks were supplied by Westinghouse. The racks' design makes use of the neutron absorber BORAL. In order to track the in-pool performance of the BORAL, a coupon surveillance program was initiated in February of 1999. The coupon program consists of sixteen large coupons (6.5-inch wide x 13-inch long x 0.075-inch thick) that have been encapsulated in stainless steel capsules and placed in the pool. The method of coupon encapsulation replicates the manner in which the BORAL is housed in the racks. In addition, eight coupons have been retained as archive coupons.

Starting in January of 2003, and at intervals of approximately one fuel cycle (18 months), some of the coupons have been subjected to inspection. In each inspection, coupons are removed from their capsules at Seabrook, the surface cleaned of transferable contamination, dried in a desiccator using silica gel and shipped to an outside laboratory. After testing, the coupons are returned to Seabrook for re-encapsulation and placement back into the spent fuel pool. The last two inspections (third and fourth inspections in 12/04 and 6/06) were conducted by NETCO in the NETCO laboratory at the Pennsylvania State University's Radiation Science and Engineering Center. In the 12/04 and 6/06 inspections, nine of the coupons from the spent fuel pool were inspected as well as an archive coupon. The results of these inspections and tests have been presented previously.[1]

During the first two inspections, blisters and corrosion pits were noted in the cladding of the BORAL. Blister formation occurs when the cladding of BORAL separates from the Al/B_4C core region of the material. Figure 1-1 shows the trimmed edge of a BORAL coupon showing the Al-1100 cladding on both sides of the central core region.

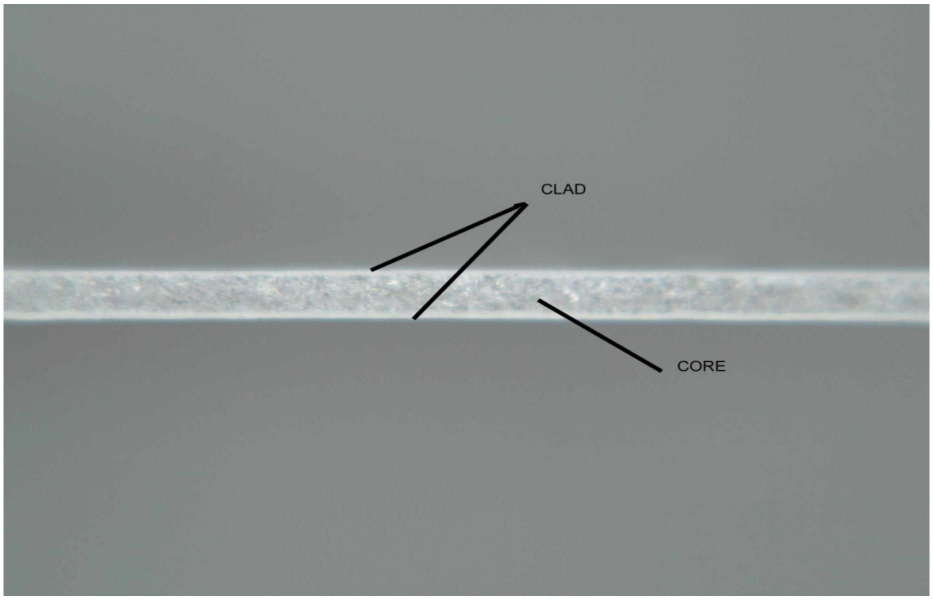


Figure 1-1
Microphotograph of the Trimmed Edge of BORAL

According to NextEra Energy Resources personnel, areas of deep clad corrosion and thick oxide were observed during the first examination before the coupons were initially dried. Accordingly, the inspections in 12/04 and 6/06 were expanded to include more coupons and further characterization. The scope of the last two inspections included:

- Visual inspection and high resolution digital photography
- Blister size characterization
- High resolution neutron radiography
- Neutron attenuation measurements
- Optical microscopy.

During inspections three and four, further corrosion and blister growth were observed. The corrosion on two of the coupons had advanced to a condition where the corrosion products were spalling off as relatively large flakes, resembling exfoliation corrosion (See Figures 1-2 and 1-3).

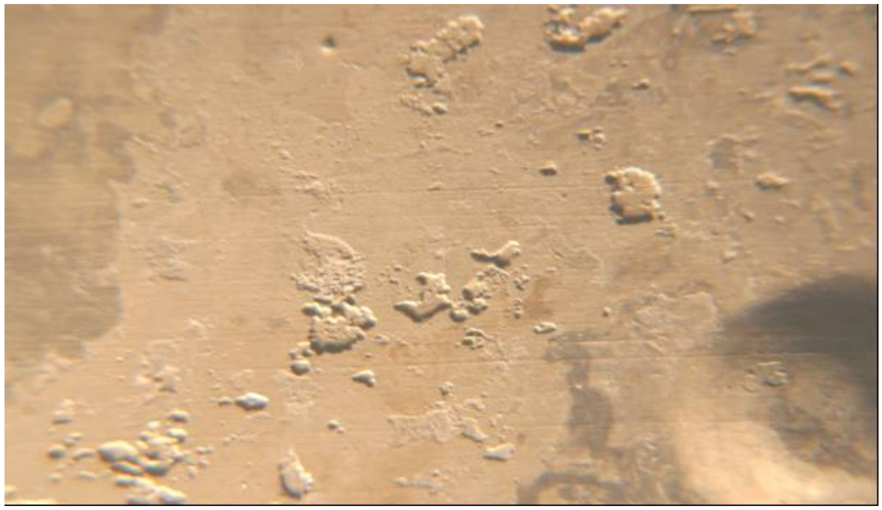


Figure 1-2
Microphotograph (8X) of Corrosion Field

Figure 1-3
Photomicrograph (40X) of Through-Cladding Corrosion Pit

The extent of corrosion on these coupons is not typically observed in BORAL surveillance coupons. While significant pitting corrosion and heavy corrosion along the edges of coupons resulting in separation of the cladding and core have been observed[2], the surface area of the corrosion fields and their extent on some of the Seabrook coupons is atypical.

In an evaluation of the data from the third and fourth inspections[1], a correlation of blister volume and extent of corrosion was noted, that is the greater the volume of blisters on a coupon, the greater the extent of corrosion. It was postulated that the blisters deform the thin gage wrapper plate on the capsules causing gaps in the capsule edges. The gaps form a flow path in and out of the capsule whereby oxygen-rich water from the bulk pool volume can continuously replace oxygen-depleted water (from corrosion of the aluminum surfaces of the coupons) in the capsule. The continuous presence of oxygen-rich water around the coupon is thought to accelerate corrosion.

The one feature of the Seabrook program that potentially could contribute to the observed corrosion rates is the periodic cycling of the Seabrook coupons. That is, every 18 months, the surveillance assembly is removed from the pool and nine coupons are removed from the assembly and removed from their capsules. The coupons are then dried and sent to a laboratory for testing. The period that the coupons are out of the pool is some 30 days or more.

It is well known that cyclic exposure of metals to a corrosive environment can result in accelerated corrosion relative to continuous exposure. This has led to the development of cyclic corrosion tests that are used extensively in the automotive and other industries for evaluation of coatings and finishes of metals. During cyclic exposure, accelerated corrosion occurs primarily in the transition from immersion-to-dry-out and dry-out-to-immersion.

As related to BORAL, accelerated corrosion could occur in the dry-out period when the coupons are out of the pool. BORAL has significant open porosity in the core region that can retain water while the coupons are out of the pool. This moisture is exposed to an oxygen-rich atmosphere (air), which would raise the dissolved oxygen in the internal moisture to ~9 ppm at 20°C. This compares with dissolved oxygen levels in the spent fuel pool that are typically in the range of 3 - 5 ppm. Further, any dissolved oxygen in the internal moisture consumed by corrosion would be replaced by the oxygen-rich air as the dissolved oxygen comes back to saturation.

This report describes accelerated corrosion testing to determine the influence of cyclic exposure of BORAL to boric acid solution on its corrosion rate relative to continuous exposure.

2
ACCELERATED CORROSION TESTING

2.1 Test Objectives

The objectives of this project are to design and conduct an accelerated corrosion test to investigate the corrosion phenomenon observed on some of the NextEra Energy Resources Seabrook surveillance coupons. This accelerated corrosion test investigates the effect of cyclic exposure to a corrosive environment versus continuous immersion in a corrosive environment. Additionally, the test investigates the effect of flow inside the capsule on corrosion rates.

2.2 Test Description

Two sets of three capsules each were custom fabricated to house half-scale BORAL coupons replicating the Seabrook capsules and coupons. Each set of three BORAL-containing capsules consisted of:

- Capsule 1: Characterized by a "tight" fitting capsule and cover plate
- Capsule 2: Capsule with a small gap between the cover plate and capsule frame
- Capsule 3: Capsule with a large gap between the cover plate and capsule frame

One set of three capsules was subjected to boric acid (2500 ppm as boron) at 195°F for 4 months continuous exposure. At 195°F, 1 month exposure is equivalent to approximately 18 months at 90°F, a temperature more representative of a spent fuel pool.[3] The other set of three capsules was exposed to the same corrosion bath for 1 month followed by drying in a desiccator for 2 days and 28 days exposure to the laboratory atmosphere. This cycle was repeated four times. Each time, the coupons were removed from their capsules at the beginning of the drying cycle and re-encapsulated prior to re-immersion, replicating the history of the Seabrook coupons.

Coupon Description

Figure 2-1 shows three BORAL coupons before testing. The coupons are half length and width scales, i.e., 3.25" x 6.5" and full thickness scale, i.e., 0.075", compared to the coupons in the Seabrook program. All six coupons used in the test were cut from the same BORAL plate (YK320412-1). Other material tested from this plate by NETCO was previously determined to be blister-resistant BORAL under simulated dry cask vacuum-drying conditions.[4]

Figure 2-1
Three BORAL Coupons Prior to Testing

Capsule Description

Six capsules were fabricated from 304L stainless steel as shown in Figures 2-2 and 2-3. The capsules consist of a heavy gage backer plate 0.0625-inch thick, a 0.090-inch thick picture frame that forms the enclosure for the coupon and a thin gage (0.020-inch thick) wrapper plate. The hole in the wrapper plate replicates, in scale, the inspection port in the wrapper plate of the Seabrook coupons and fuel racks.

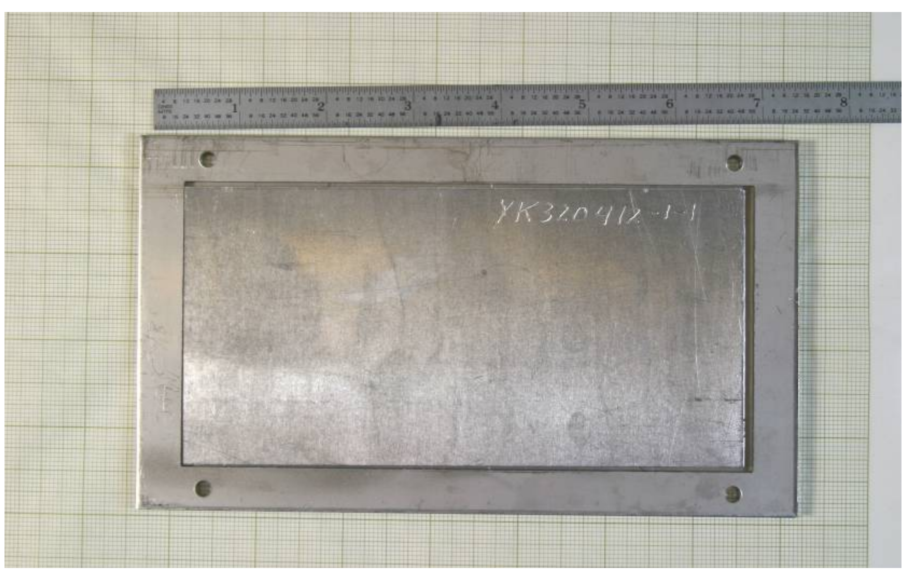


Figure 2-2
Capsule with Coupon in Place

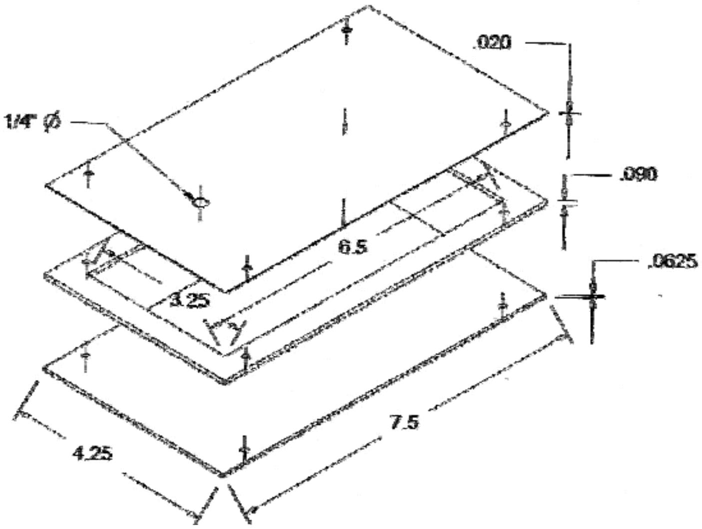


Figure 2-3
Capsule Details

Figure 2-4 shows a capsule fully assembled and secured with four stainless steel machine screws. The spacing of the machine screws replicates the spacing of the capturing devices on the Seabrook coupons and the welds in the racks.

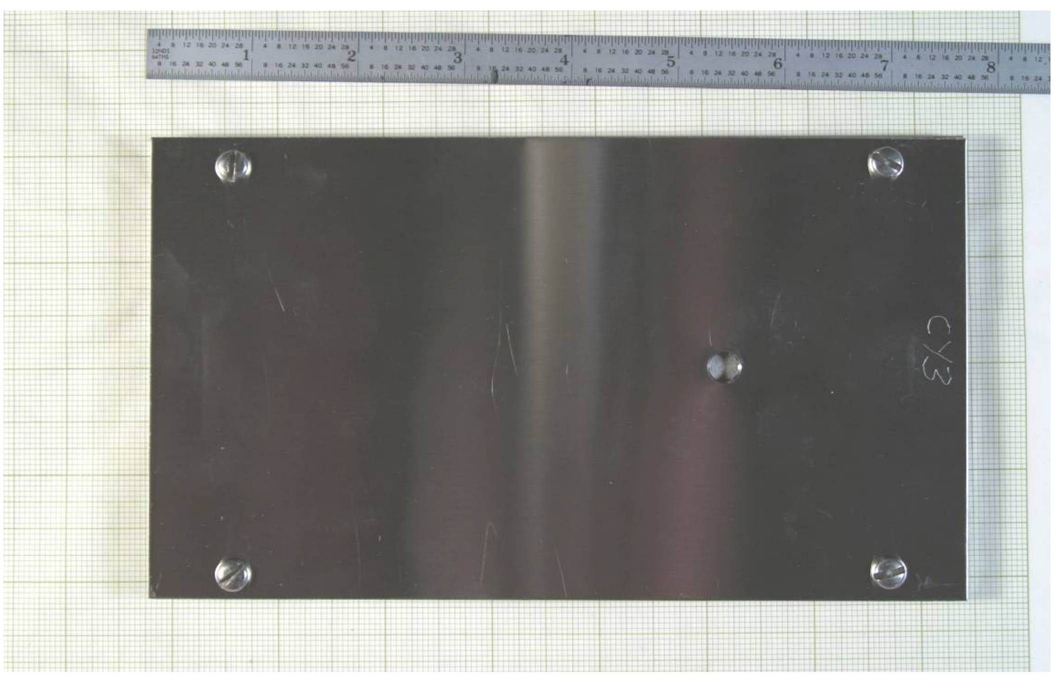


Figure 2-4
Fully Assembled Capsule

Stainless steel spacer disks 0.030" and 0.060" thick were used between the coupon and thin gage wrapper plate to simulate blisters that deform the wrapper plate, creating flow path. Figure 2-5 shows the placement of the spacer disks on the coupon. Figure 2-6 shows one set of low, intermediate and high flow capsules viewed from the edge. The high flow capsule utilizes two 0.060-inch thick spacer disks to create gaps in the two long edges of the capsule that are nominally 0.045-inch wide at their widest point. The intermediate capsule uses two 0.030-inch thick disks to create gaps that are nominally 0.015-inch wide in the two long edges of the capsule at their widest point. The low flow capsule does not incorporate spacer disks and the three components of the capsule are held tightly together by the four machine screws.

Figure 2-5
Spacer Disks That Form a Flow Path

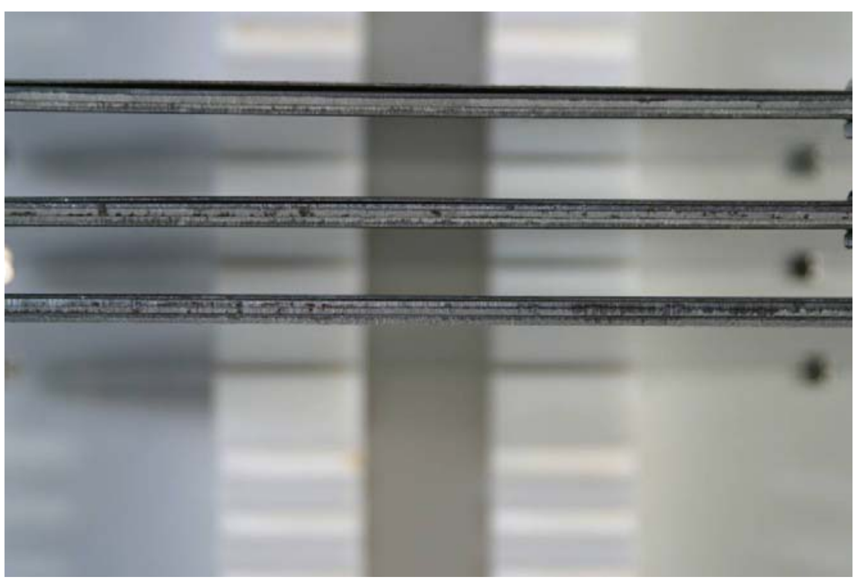


Figure 2-6
Capsule Edge Configuration

Accelerated Corrosion Bath

The coupons were maintained in the corrosion bath in a special Teflon rack, as shown in Figure 2-7. Aeration was provided to assure that oxygen concentration in the bath was always at saturation at 195°F. At a bath temperature of 195°F, one month is equivalent to approximately 18 months at 90°F, a temperature representative of the Seabrook spent fuel pool water.

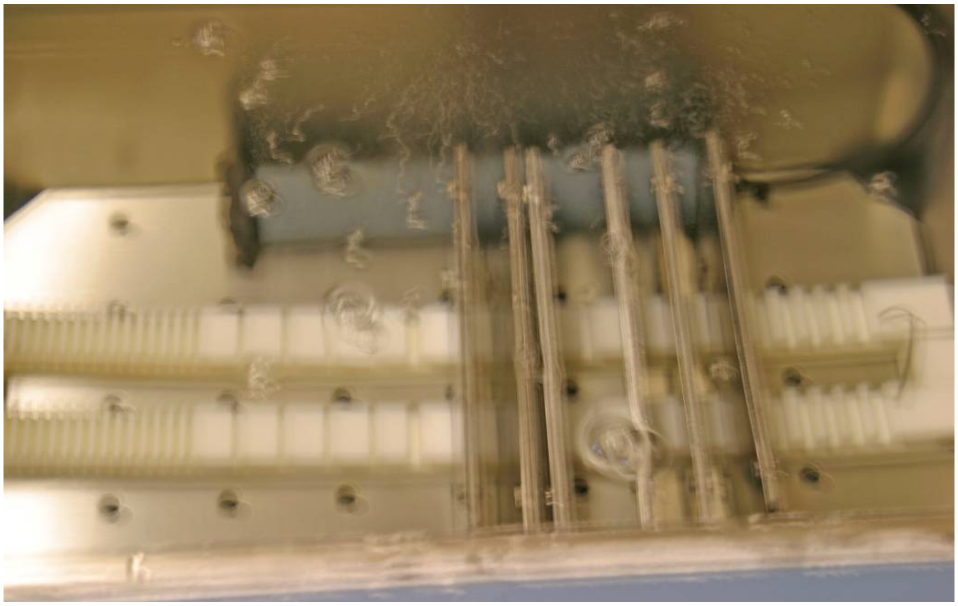


Figure 2-7
Capsules in the Corrosion Bath

The corrosion test solution was prepared as follows: Laboratory tap water was processed by first passing it through two universal ion exchange columns and then through two research grade ion exchange columns. The typical quality of the deionized water used for the initial bath solution and make-up water is shown in Table 2-1.

Table 2-1
Water Specification for Corrosion Test

pH	5.75 @ 20°C
Conductivity	0.5 µS/cm
Resistivity	2.0 Ω/cm
Aluminum	<0.01 ppm
SiO_2	<0.1 ppm
Cl	<0.01 ppm
Na	<0.03 ppm

Sufficient reagent grade boric acid was added to bring the boron concentration to ~2500 ppm. This increased the initial conductivity from <1.0 µS/cm to ~40 µS/cm at 20°C, and decreased the initial pH from 5.75 at 20°C to 4.76 at 20°C. The boron concentration in the initial bath water was verified by chemical analysis and periodically checked throughout the test.

The conductivity and pH of the bath was measured at an approximate frequency of once per week. Figures 2-8 and 2-9 are plots of the measured conductivity and pH of the bath versus time. During the third exposure to the laboratory atmosphere, there were no coupons in the corrosion bath and no conductivity and pH data were taken.

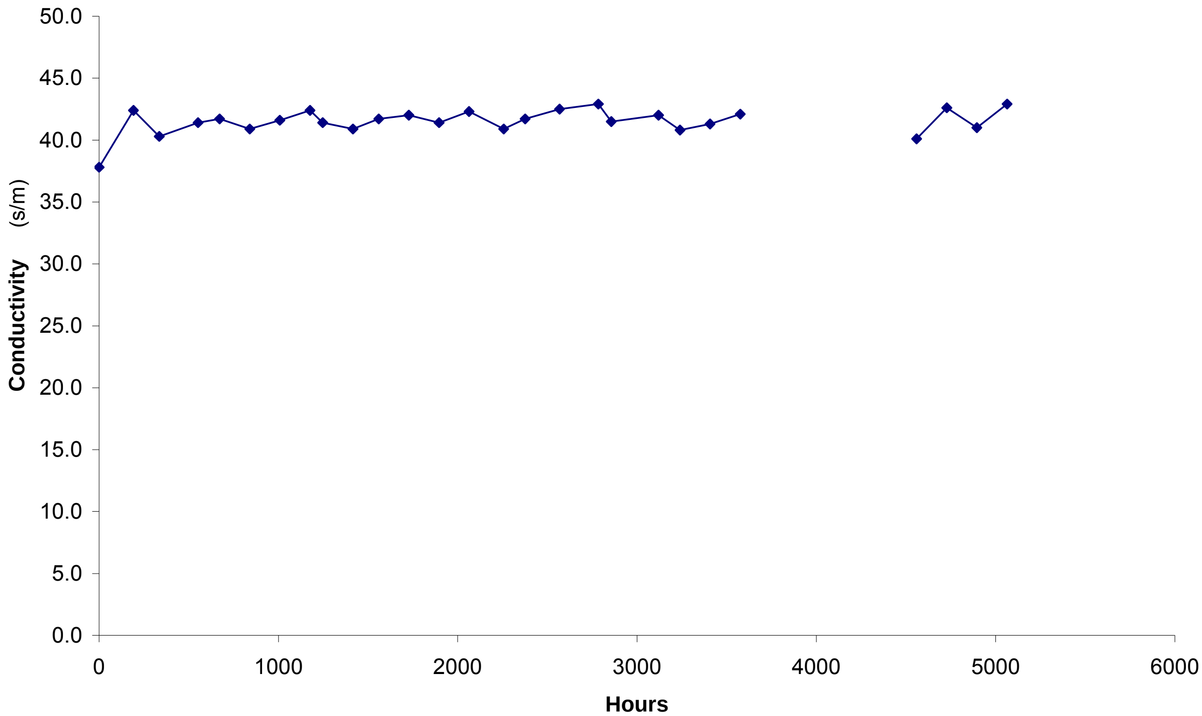


Figure 2-8
Conductivity of the Corrosion Bath vs. Time

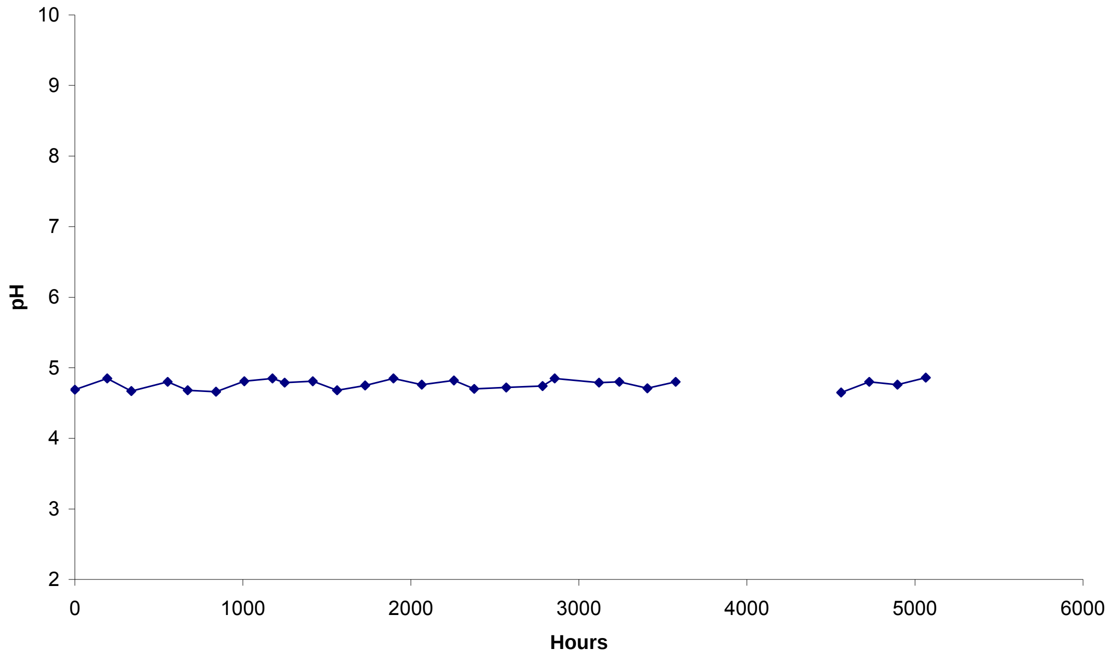


Figure 2-9
pH of the Corrosion Bath vs. Time

Figure 2-10 shows the thermal exposure history of the two sets of capsules.

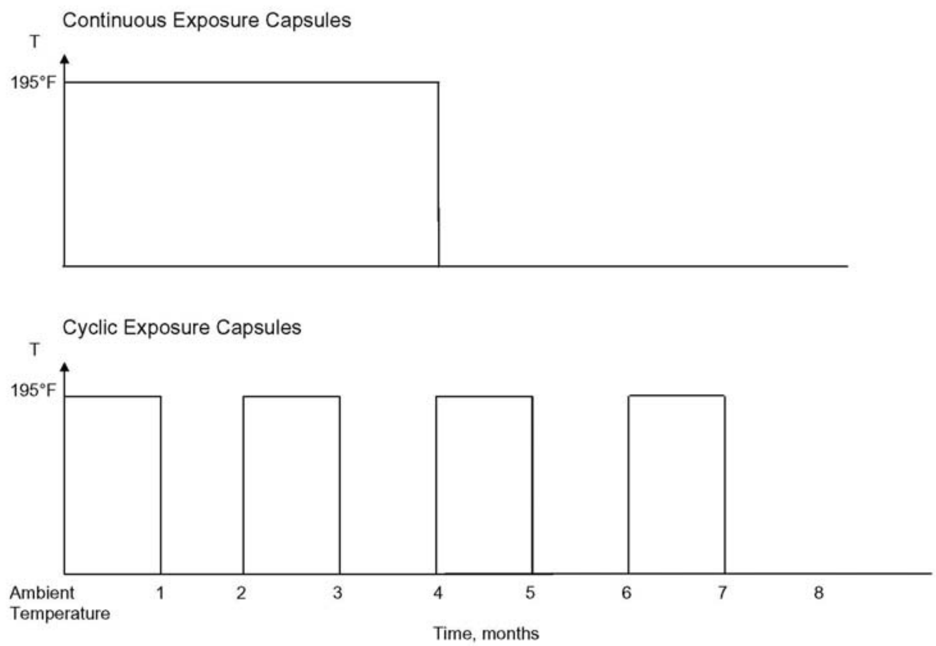


Figure 2-10
Capsule Exposure History

3
TEST RESULTS

Prior to testing, each of the coupons was characterized with respect to the following physical attributes:

- Visual appearance and high resolution digital photography
- Thickness at 10 locations
- Weight (after 48 hours in a desiccator)

The continuously exposed coupons were subjected to the same characterization after 4 months in the corrosion bath. The cyclically exposed coupons were subjected to characterization after each 30-day exposure.

Visual Appearance

Figures 3-1 and 3-2 show the front and back sides of the continuous exposure coupons after 4 months in the corrosion bath. Qualitatively, the low flow coupon has less surface corrosion than either the moderate or high flow coupon. A blister had developed on the back side of the coupon in the moderate flow capsule as shown in Figure 3-3. The blister was 0.43 inch in diameter and 0.038-inch high.

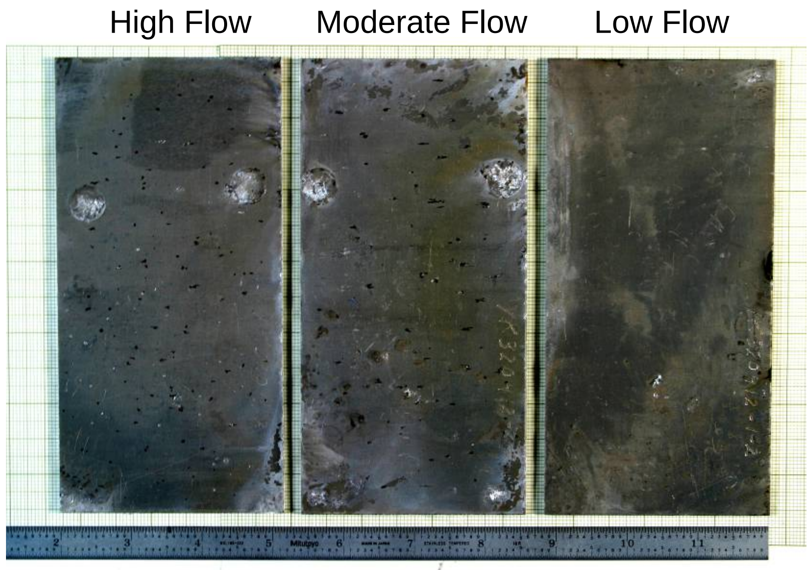


Figure 3-1
Continuous Exposure for Four Months – Front Side

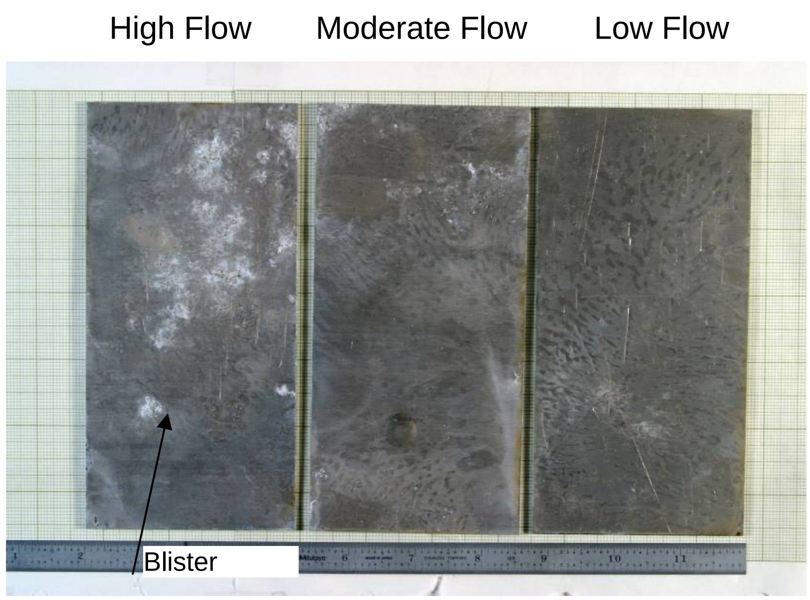
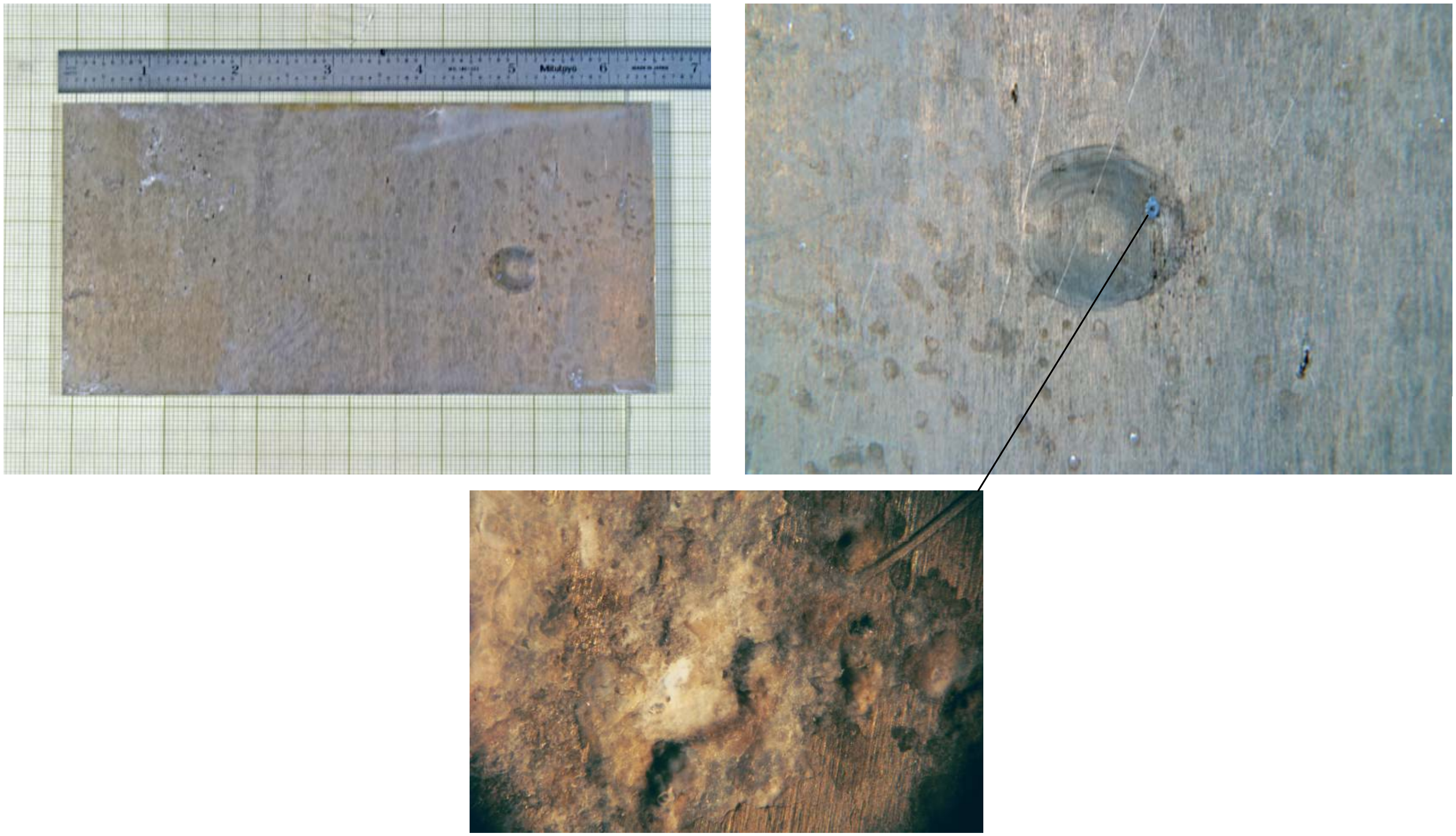


Figure 3-2
Continuous Exposure for Four Months – Back Side

Macrophotograph

Microphotograph (8x mag)



Photomicrograph: Through-cladding pit on cap

Figure 3-3
Blister in Back Side of Coupon from the Moderate Flow Capsule

Figure 3-3 shows a through-cladding corrosion pit had developed in the blister cap. It is believed that water entered the core material through the pit and the blister formed due to pressure buildup of hydrogen from subsequent corrosion. This type of blister formation phenomenon has been observed before.[4]

Figures 3-4 and 3-5 contain macrophotographs of the front and back sides of the coupon subjected to cyclic exposure. Appearance-wise, the cyclically exposed coupons are not substantially different from the continuously exposed coupons. The coupon in the low flow capsule has less surface oxide than either the coupon in the moderate or high flow capsule.

During the fourth exposure cycle, two small blisters developed on the front side of the coupon in the moderate flow capsule. One blister was in the upper left corner; a second in the lower left corner. Figures 3-6 and 3-7 are microphotographs of these two blisters at 8X magnification. The blister in the upper left corner was 0.35 inch in diameter and 0.020-inch high. The blister in the lower left corner was 0.30 inch in diameter and 0.015-inch high. Neither blister had a corrosion pit in the cap.

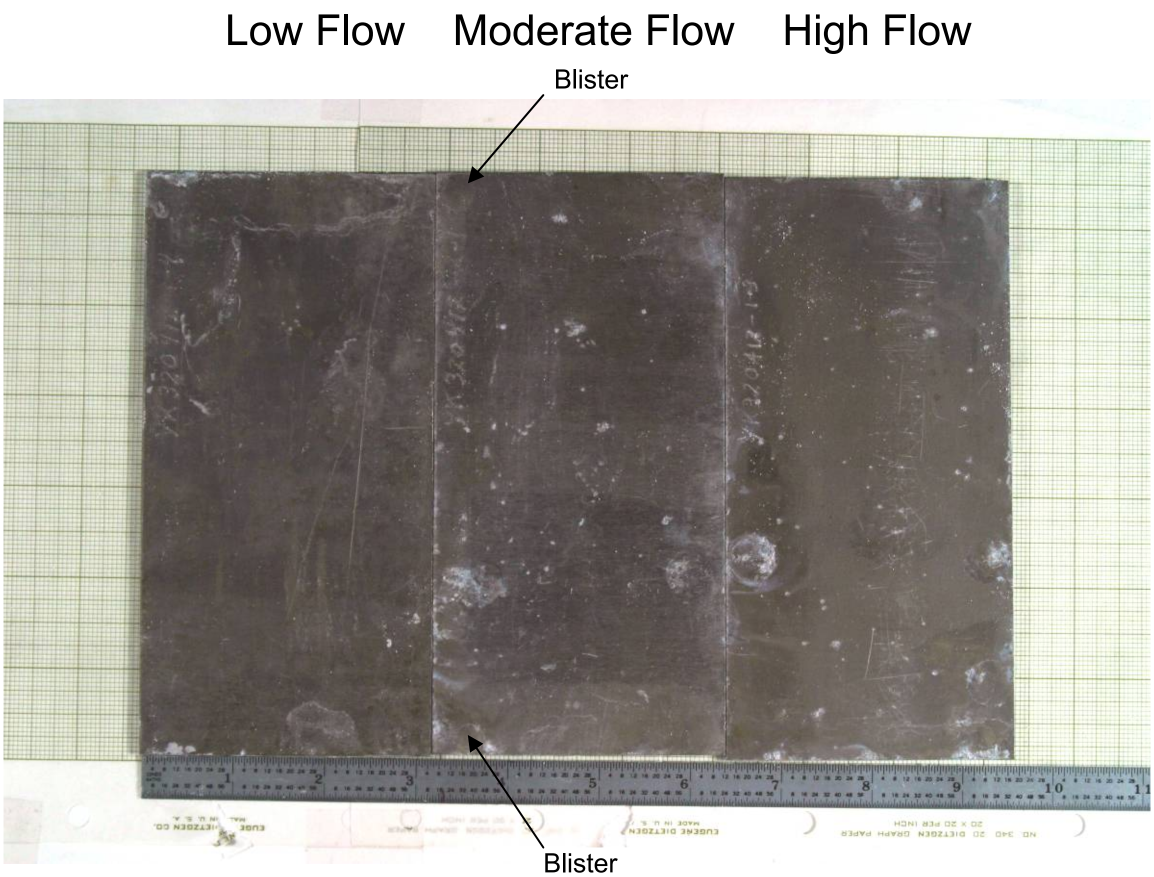


Figure 3-4
Front Side of Cyclically Exposed Coupons

High Flow Moderate Flow Low Flow

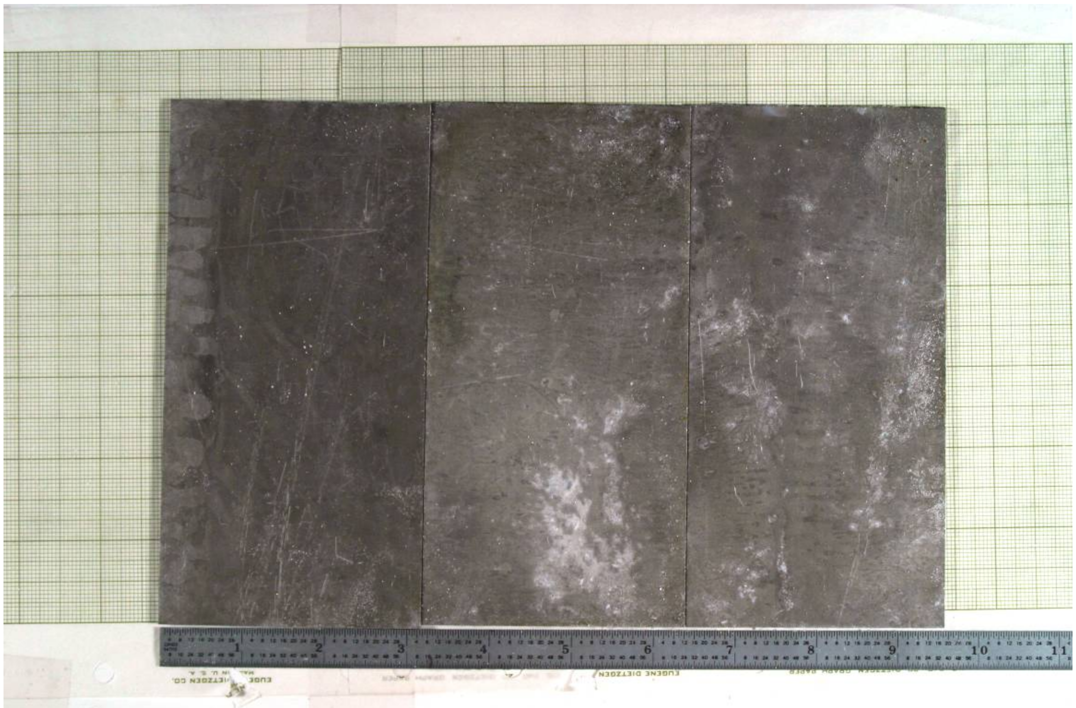


Figure 3-5
Back Side of Cyclically Exposed Coupons

Figure 3-6
Microphotograph (8x) of Blister in Upper Left Corner of Coupon from Moderate Flow Capsule

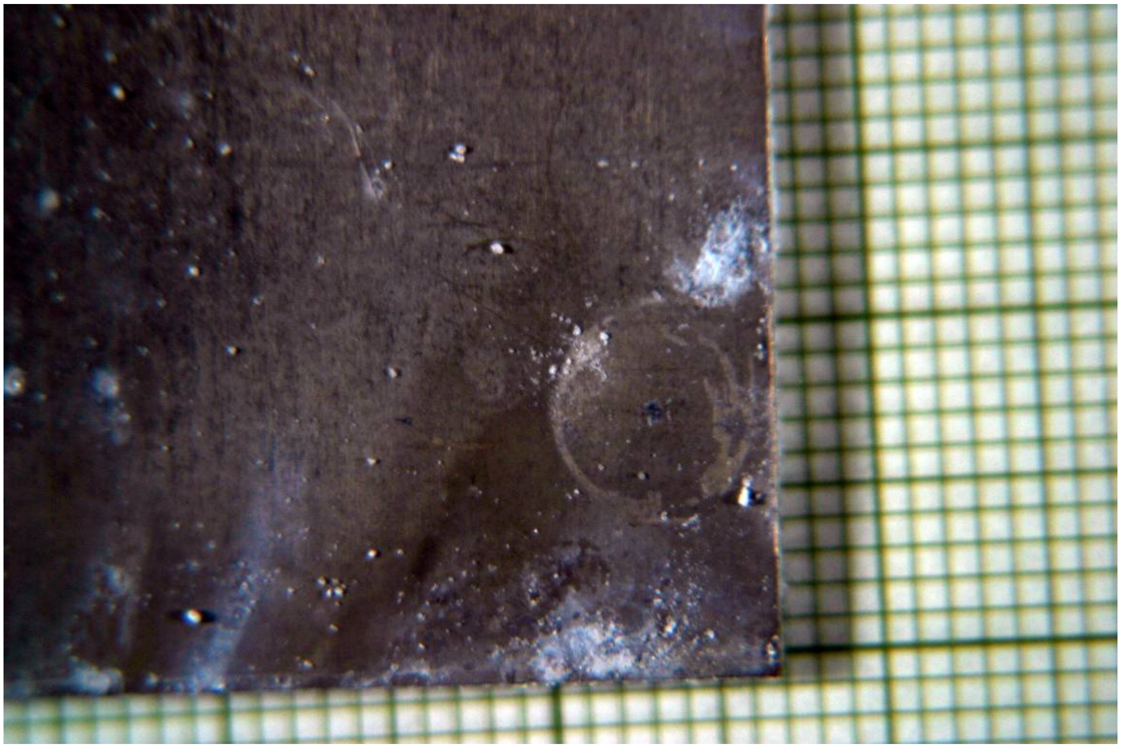


Figure 3-7
Microphotograph (8x) of Blister in Lower Left Corner of Coupon from Moderate Flow Capsule

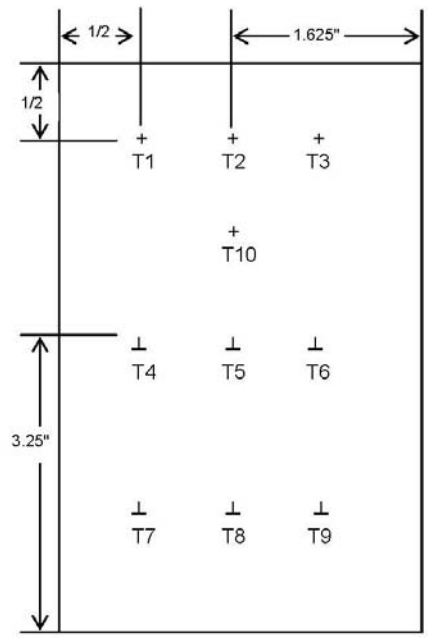
It is therefore presumed that moisture entered the core region through the edge of the coupon and subsequent hydrogen generation caused the cladding to delaminate.

Coupon Thickness

The thickness of each coupon was measured before and after testing at ten locations as shown in Figure 3-8. Within the precision of the measurements, no significant change in thickness was observed.

Coupon Weight

The initial weight of all coupons was measured after drying in a desiccator. The coupons subjected to continuous exposure were removed from the boric acid solution after 4 months, dried in a desiccator for 48 hours, and reweighed. After each exposure cycle, the cyclic coupons were dried in a desiccator for 48 hours and reweighed. Figure 3-9 contains a plot of percent weight gain.



Note: Location T10 is under the inspection port

Figure 3-8
Locations for Coupon Thickness Measurements

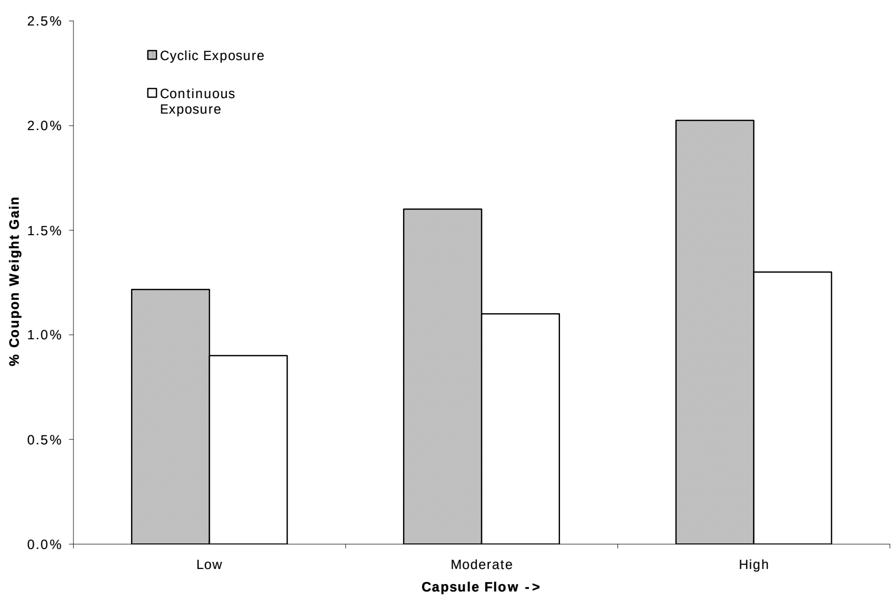


Figure 3-9
Coupon Weight Gain

The data in this figure are quantitative confirmation of the qualitative conclusion based on visual appearance that moderate flow and high flow capsules result in greater coupon weight gain from corrosion products than the coupons in the low flow capsule. Further, all coupons subjected to cyclic exposure sustained greater weight gain than the corresponding coupons subjected to continuous exposure. This confirms the effect of cyclic immersion followed by dryout and re-immersion although the extent of corrosion observed in these tests was considerably less than that sustained by some of the coupons in the Seabrook surveillance program.

4 SUMMARY AND CONCLUSIONS

An accelerated corrosion test for BORAL was designed and carried out to investigate:

- The effect of cyclic immersion on corrosion rates.
- The effect of capsule flow on corrosion rates.

The accelerated corrosion tests were prompted by the observed heavy corrosion on some previously examined BORAL coupons from the NextEra Seabrook surveillance program.

The tests did confirm the effect of capsule flow on corrosion rates postulated previously.[1] Capsules that had moderate and high flow rates had coupons that experienced greater corrosion than coupons in capsules with low flow rates. Further, coupons subjected to cyclic exposure to boric acid experienced more corrosion than coupons subjected to continuous exposure. However, the accelerated corrosion test did not replicate the extent of corrosion experienced by some of the Seabrook coupons. The reason for this could be attributable to differences in water chemistry between the test and the Seabrook pool, or to differences in production lots of BORAL, or possibly, to a still unidentified cause or mechanism. It is noted that some of the coupons in the Seabrook program experienced very limited corrosion, which may be keyed to the production lot.

A significant finding from these tests relates to an understanding of the mechanism of blister formation in the cladding of BORAL. A total of three blisters on two coupons developed during the course of testing. Since the coupons were subject to isothermal conditions at a temperature below the boiling point of water (195°F), steam formation in the water entrained in the internal porosity in the core of BORAL could not have caused the cladding to delaminate and form a blister. This suggests hydrogen formation, a byproduct of corrosion of the internal pore surfaces in the core of BORAL, as the likely cause of blisters.

This is further confirmation of the analysis of the thermal conditions in BORAL used in nuclear fuel storage racks conducted previously.[5] These calculations showed that the temperatures in the BORAL were insufficient to result in boiling conditions. This leads to the conclusion that blister formation in BORAL used for storage racks is likely due to hydrogen generation and not boiling of moisture entrained in the core of BORAL.

In one coupon, a blister formed in the central region of the coupon. Inspection of the blister cap revealed what had been a through-cladding corrosion pit. It is believed that moisture entered the core region of the BORAL through the corrosion pit. Subsequent corrosion around the pit sealed the pit, preventing a path for the escape of hydrogen from the core.

In another coupon, two small blisters formed on two corners of one side of the coupon near the coupon edges. In this case, no corrosion pits were observed on the blister caps and it is believed water entered the core through porosity in the cut edges of the BORAL.

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