



L-Basin Corrosion Surveillance Program Plan

J. I. Mickalonis

September 2014

SRNL-TR-2009-00140, Revision 3



DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *Spent fuel storage,
aluminum alloys, pitting
corrosion*

Retention: *Permanent*

L-Basin Corrosion Surveillance Program Plan

J. I. Mickalonis

September 2014

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



EXECUTIVE SUMMARY

This report is the revised plan for corrosion surveillance of the aluminum-clad fuel and aluminum storage systems (racks, tube bundles, and oversized storage containers) in the Savannah River Site L Basin. Corrosion surveillance supports the demonstration of continued safe storage of spent or used nuclear fuel in L Basin.

The corrosion surveillance program plan contains the following:

- Description of the coupon materials;
- Immersion locations in L Basin;
- Coupon withdrawal and evaluation schedule;
- Corrosion evaluation procedure and protocols; and
- Corrosion surveillance reporting

Corrosion evaluation is performed in the laboratory on corrosion coupons of surrogate materials of the fuel cladding and the storage systems following their exposure in L Basin.

The purpose of the L Basin corrosion surveillance program is to provide early detection and characterization of corrosion attack to the fuel and storage system materials as a result of prolonged exposure to the L Basin water environment. The early detection of corrosion allows for adjustment of the water quality and fuel storage configurations to mitigate excessive corrosion attack. The corrosion surveillance program is to be continued for the duration of the storage period of fuel in L Basin.

This revision of the corrosion surveillance program plan includes a description of the new corrosion coupon assembly for exposing furniture rack coupons (i.e. storage system material) and a new protocol for analyzing the coupons in the laboratory with a laser confocal microscope.

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF FIGURES	vii
LIST OF ABBREVIATIONS	viii
1.0 Introduction.....	1
2.0 Corrosion Coupon Surveillance – Historical Overview	1
3.0 Surveillance Coupon Description	2
3.1 Ray Guns.....	3
3.2 Junior Ray Gun	4
3.3 Chime Rack.....	5
3.4 New Furniture Rack Assembly.....	6
3.5 Coupon Location.....	8
3.6 Coupon Withdrawal and Evaluation Schedule	9
4.0 Corrosion Evaluation Protocols	10
4.1 Coupon Removal	10
4.2 Corrosion Evaluation	11
5.0 Reporting and Documentation	13
6.0 Special Inspection	14
7.0 References.....	14

LIST OF TABLES

Table 1	Ray Gun Coupon Configurations.....	3
Table 2	Nominal Compositions of Aluminum Alloys for Corrosion Coupons	4
Table 3	Junior Ray Gun Coupon Configurations.....	5
Table 4	Coupon Assembly Location and Installation Details.....	7
Table 5	New EBS Coupon Positions on Basin Assembly	8
Table 6	Coupon Assembly Location and Installation Details	9

LIST OF FIGURES

Figure 1	Ray gun removed from L Basin in 2004 showing the coupon configurations .	4
Figure 2	Junior ray gun after exposure, the gun hung in L Basin with the left side being the top.....	4
Figure 3	Sketch of coupons showing the isolation of coupons from the rod (washer a) and other coupons (washer a and b): A) galvanic or crevice pair and B) individual coupon.....	5
Figure 4	Chime rack with strings of furniture rack coupons made of rectangular plates and U-channels.....	6
Figure 5	As-received condition of the furniture rack coupons removed from L Basin in 2011.....	6
Figure 6	Schematic drawings of the new EBS tubing and U-channel samples showing the cut lines and additional welds for making the corrosion coupons for the new L Basin furniture rack assembly (blue – vendor weld, gray site weld	7
Figure 7	Schematic of proposed EBS coupon assembly for exposure in L Basin	8
Figure 8	L Basin map showing location of remaining coupon assemblies (ovals – surveillance coupons, triangles – furniture rack coupons).....	9
Figure 9	Laser confocal microscope images of the 2010 surveillance coupon Al6061 #044: (A) laser and optical scan, which highlights surface topography and (B) height scan, which highlights differences in elevation across the coupon (data on bottom of photograph shows the height profile for the line scan)	12
Figure 10	Pictures of pits taken during pit depth measurements using both a standard white light microscope (A – focus on top of pit, B – focus on bottom of pit, 100x) and a laser confocal microscope (C – focused optical image, D – laser height scan showing pit depths, 100x).....	13

LIST OF ABBREVIATIONS

DOE	Department of Energy
EBS	Environmental Biotechnology Section
GTA	Gas Tungsten Arc
HTS	Horizontal Tube Storage
IAEA	International Atomic Energy Agency
LCM	Laser Confocal Microscope
MB	Machine Basin
PI	Principal Investigator
SFP	Spent Fuel Project
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SS	Stainless Steel
SWM	Standard White Light Microscope
TB	Transfer Bay
TFE	Tetrafluoroethylene
VTS	Vertical Tube Storage

1.0 Introduction

The L Basin corrosion surveillance program provides for early detection of corrosion of the materials of construction of the aluminum-clad fuel and aluminum storage systems due to prolonged exposure in the L Basin water environment. Early detection of corrosion allows for adjustments, as necessary, of the basin water quality and fuel storage configuration to mitigate excessive corrosion.

The use of corrosion surveillance in management of research reactor spent nuclear fuel in basin storage is promoted by the International Atomic Energy Agency (IAEA) [1, 2]. The design of a corrosion surveillance program has been published [1, 2]. The IAEA corrosion surveillance program for research reactor fuel evolved primarily from the comprehensive response by Savannah River Site (SRS) to corrosion in reactor basins as they were transitioned from temporary storage to long-term storage [3].

Corrosion surveillance involves the immersion, periodic removal and evaluation of a set of test coupons exposed to the basin water for the purpose of detecting and characterizing corrosion processes. Water quality parameters are also determined at periodic intervals and documented with the corrosion results, so the impact on corrosion of the prevailing water chemistry and any chemistry transients can be evaluated.

The effects of water quality on an aluminum storage system in L Basin can also be assessed from inspections using non-destructive techniques. A special inspection of the oversize storage canisters was performed using remote underwater visual and ultrasonic inspection methods [4]. The objectives of the inspection were to evaluate the condition of the canister and the condition of the canister internals and are a component of surveillance activities. Additionally, activities are in progress to perform inspection of the fuel using remote underwater visual inspection.

This corrosion surveillance program plan discusses the coupon configurations and materials of construction, basin location and current inventory as well as the protocol for evaluation, documentation and reporting. The current revision supersedes the last plan and discusses the design for a corrosion assembly of new furniture rack coupons as well as a new protocol for evaluating surveillance coupons with a laser confocal microscopy (LCM).

2.0 Corrosion Coupon Surveillance – Historical Overview

The L-Reactor basin at the SRS was refurbished in the mid 1990's to receive a large number of the aluminum-clad research reactor assemblies from both domestic and foreign sources through 2035. With this refurbishment and as a part of the response to observed corrosion in aluminum materials in basin storage, a comprehensive corrosion surveillance program was developed to directly support the new extended fuel storage activity at SRS.

L Basin corrosion surveillance activities began in 1992 and, in 1995, with a Research Reactor Corrosion Surveillance Program that included fuel storage at SRS in P, K, L, and

RBOF basins. This program concentrated on aluminum alloys that were typical of cladding materials for foreign spent reactor fuel. Other alloy-clad fuels like Zircaloy and stainless steel (SS) are more corrosion resistant and are not included in this program. At present, L Basin is the single basin maintained and in use at SRS for basin storage of fuel. Maintenance of the systems for continued safe storage until retrieval and disposition of the fuel are led through the engineering organization under the Spent Fuel Project (SFP) organization.

Data from this program and laboratory corrosion testing activities provided a technical basis for maintaining and improving basin management practices, establishing operational limits for L Basin water chemistry, and supporting the prediction of fuel storage performance in the basin. In addition to providing data to support the new spent fuel storage mission at SRS, original results from this program fed directly into the IAEA sponsored corrosion surveillance activities for research reactor aluminum-clad spent fuels.

The L Basin Corrosion Surveillance Program continues to provide information to evaluate the resistance of spent fuel cladding to corrosion in the basin water and to provide assurance of continued safe, interim storage for the aluminum-clad fuels and the aluminum storage systems. The corrosion surveillance program is planned for continuation throughout the duration of fuel storage in L Basin.

3.0 Surveillance Coupon Description

Surveillance coupon assemblies currently are of three types, ray guns and junior ray guns with disk coupons of surrogate aluminum materials, and chimes of actual furniture rack components. The coupons were not pretreated to produce a protective oxide layer prior to exposure. These non-protected coupons are more susceptible to corrosion than most of the spent nuclear fuel cladding which has developed a protective oxide layer from high temperature radioactive operations. Some of the fuel cladding oxide may not be in pristine condition due to spallation or mechanical damage prior to arrival at SRS. These damaged areas on the fuel cladding are considered to have similar susceptibility to corrosion as the coupons. Data from the corrosion surveillance program has shown that general corrosion on aluminum-clad, irradiated elements with the high temperature protective oxide^a coating is negligible unless there was a breach in the coating [5].

A fourth assembly for L Basin has a new design for exposing furniture rack coupons. These coupons will extend the coverage period of the current coupon assemblies and set up a removal schedule for improving estimates of the degradation rate of aluminum alloys in basin water.

^a Corrosion oxides formed on fresh, aluminum coupons in water at low temperatures (< 70 °C) is significantly different from that of irradiated aluminum-clad fuel. The structure of the low temperature oxide is of the form Bayerite ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$) which is loose, flocculent, and non-protective. A high temperature, compact, and protective Boehmite oxide coating ($\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) is formed on the surface of the irradiated aluminum-clad fuel.

3.1 Ray Guns

The ray gun, which was first placed into L Basin, has multiple disk shaped coupons sleeved on a rod. The complete listing of coupons is shown in Table 1. Some coupons were also welded and are annotated in the table.

Table 1 Ray Gun Coupon Configuration

Coupon Type	Number	Alloy 1	Alloy2	Total Coupons
Galvanic*	2	304SS	1100	36
	2	304SS	6061-T6 ^a	
	2	304SS	6063-T5	
	2	1100	6061-T6	
	2	1100	6063-T5	
	2	6061-T6	1100	
	2	6061-T6	6063-T5	
	2	6063-T5	1100	
	2	6063-T5	6061-T6	
Crevice**	2	1100	1100	12
	1	1100w	1100w	
	2	6061-T6	6061-T6	
	1	6061-T6w	6061-T6w	
Crevice (TFE serrated washer)	2		1100	8
	2		1100w	
	2		6061-T6	
	2		6061-T6w	
Single Coupon	2		1100	14
	2		1100w	
	2		6061-T5	
	2		6061-T6w	
	2		6063-T5	
	2		5086	
	2		304SS	

* Alloy 1 has 70 mm diameter

** Alloy 2 has 32 mm diameter

Figure 1 shows a picture of the ray gun that was removed in 2004. These coupons are separated by Teflon® washers to keep them isolated from other coupons and the rod as shown in Figure 3 for the junior ray gun. The ray gun has 70 coupons which are configured either individually, in galvanic pairs with 304SS coupons or a dissimilar aluminum alloy, and in creviced pairs with either a like coupon of the same alloy or a serrated washer.

^a T5 indicates a heat treatment which involves cooling from the shaping process and artificially aged for precipitation; T6 indicated a solution heat treatment with artificially aging.



Figure 1 Ray gun removed from L Basin in 2004 showing the array coupon configurations

The nominal compositions of the aluminum alloys are shown in Table 2.

Table 2 Composition of Aluminum Alloys for Corrosion Coupons

Alloy	Elemental Concentration (wt%)				
	Si	Cu	Mg	Cr	Fe
6061	0.6	0.28	1	0.35	
6063	0.4		0.7	0.1	0.35
5086**			4	0.15	
1100	*	0.05 -0.2			*

* Si + Fe < 1%

** Mn – 0.4

3.2 Junior Ray Gun

The junior ray gun, which is pictured in Figure 2, has multiple disk-shaped coupons sleeved on a rod. These coupons are separated by Teflon® washers to keep them isolated from other coupons and the rod as shown in Figure 3.

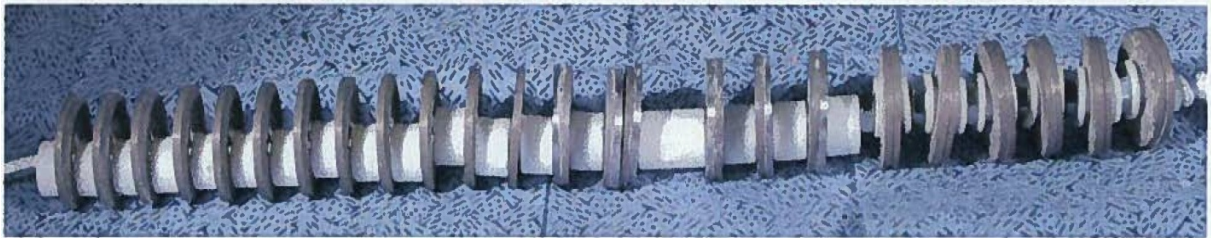


Figure 2 Junior ray gun after exposure, the gun hung in L Basin with the left side being the top

The coupons nominal dimensions are 1.25 inch OD, 0.375 inch ID, 0.12 inch thick. These coupons have three configurations: individual and galvanic and crevice pairs. The galvanic pair couples an aluminum coupon with a similar sized coupon fabricated of 304SS. The crevice pair couples two aluminum coupons of the same alloy. The aluminum alloys are 1100, 6061-T6, and 6063-T5; nominal compositions are given in Table 2. A total of 36 coupons are in a junior ray gun with the distribution of the coupon configurations shown in Table 3.

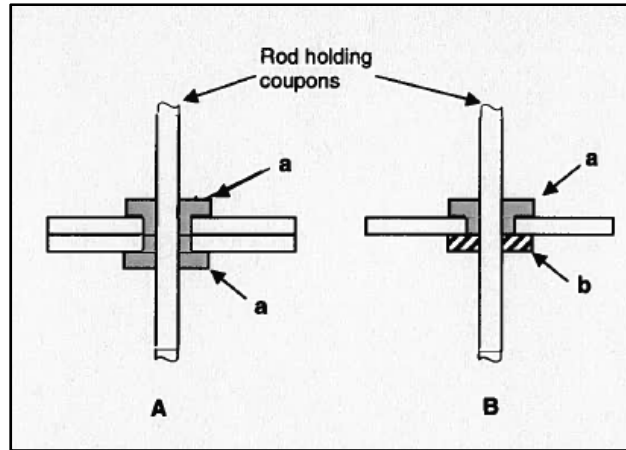


Figure 3 Sketch of coupons showing the isolation of coupons from the rod (washer a) and other coupons (washer a and b): A) galvanic or crevice pair and B) individual coupon

Table 3 Junior Ray Gun Coupon Configurations

Type	Number	Alloys	Total Coupons
Galvanic	2	304/1100	12
	2	304/6061-T6	
	2	304/6063-T5	
Crevice	2	1100/1100	12
	2	6061-T6/6061-T6	
	2	6063-T5/6063-T5	
Individual	4	1100	12
	4	6061-T6	
	4	6063-T5	

3.3 Chime Rack

The chime rack, which is shown in Figure 4, contains strings of furniture rack coupons^a with a string of five being removed at one time.

These coupons are of two types and aluminum alloys: three rectangular plates fabricated from 6061-T6 and two U-channels fabricated from 6063-T5 (See Figure 5). The rectangular plate has dimensions of 4 inch by 2 inch by 0.25 inch. The extruded U-channels have dimensions of 4 inch by 0.625 inch by 0.125 inch. One type of each coupon has a single weld bead, approximately 0.25 inch wide. Coupon welding was performed using gas tungsten arc (GTA) welding by the fabricator of the furniture racks and a R4043 weld rod was used.

^a Furniture rack coupons are made of storage rack structures of alloys from the Horizontal Tube Storage system, which was removed from the basin.



Figure 4 Chime rack with strings of furniture rack coupons made of rectangular plates and U-channels

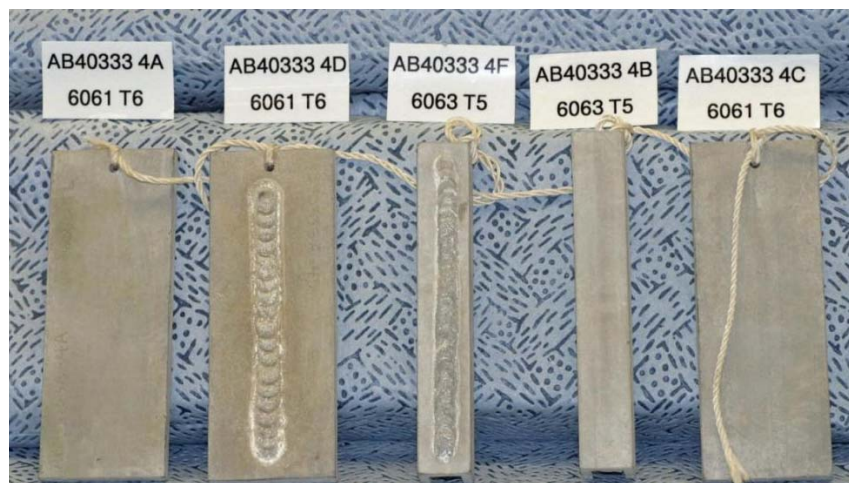


Figure 5 As-received condition of the furniture rack coupons removed from L Basin in 2011

3.4 New Furniture Rack Assembly

When new Expanded Basin Storage (EBS) racks were recently manufactured, furniture rack samples were also procured and made from the same heat of material as the EBS racks. The details about the samples are given in Table 4. The samples were cut from the 6-inch by 6-inch square tubes (fuel bundling tubes) or 6-inch by 2.375-inch U-channels (cross support braces) used in fabricating the EBS racks. Some coupons have a GTA weld placed in one corner. The tubing and U channel are specified as 6061-T6 and the weld filler metal is ER4043.

Table 4 Details of Furniture Rack Samples from New EBS Racks

Sample	Dimensions (inch)	Shape	Heat #	Quantity
Tubing	6 × 6 × 2.375	Square	283376-01	10
Cross brace	6 × 2.375 × 2	U-Channel	HC 534074	10
Tubing – Welded	6 × 6 × 2.375	Square	283376-01	10
Cross brace -welded	6 × 2.375 × 2	U-Channel	HC534074	10

Corrosion coupons for the new assembly will be fabricated from the new EBS furniture rack samples. To maximize the number of coupons for exposure, the square tube samples will be cut into four sections and the U-channel into two sections as shown in Figure 6 below. For each welded sample, the vendor put a GTA weld (blue welds in figure below) in one of the corners. An additional GTA weld will be made by a site machine shop to increase the number of welded samples.

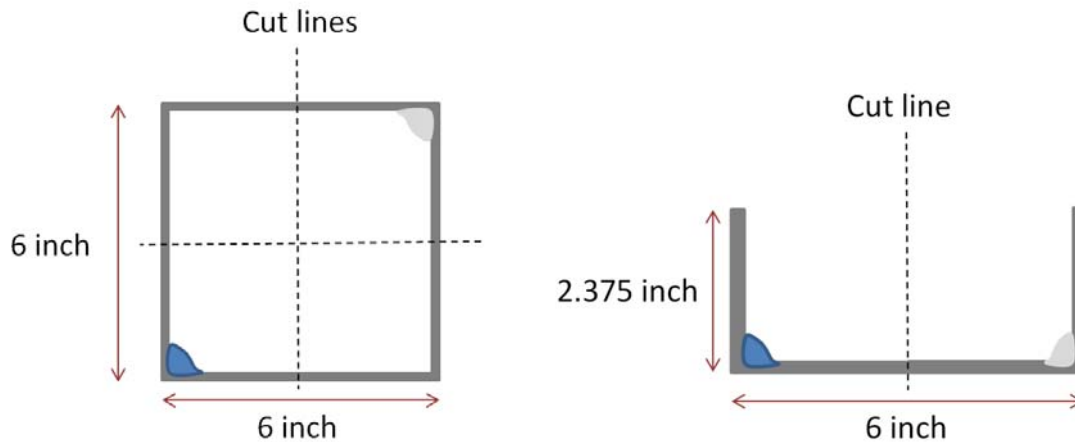


Figure 6 Schematic drawings of the new EBS tubing and U-channel samples showing the cut lines and additional welds for making the corrosion coupons for the new L Basin furniture rack assembly (blue – vendor weld, gray site weld)

Additional coupon preparation will include drilling a hole for mounting to the assembly structure, etching with a unique identifying number, and sanding to remove any sharp edges. Dimensional measurements will be made with M&TE micrometer and recorded to the thousandth place. Coupons will be weighed and weights recorded to the nearest hundredth of a gram. Coupons will be photographed to document initial surface conditions. All data will be recorded in an electronic notebook dedicated to the L Basin Corrosion Surveillance Program and stored on the winsan1 server in the following folder Mat_tech/L Basin Corrosion. Several new EBS coupons will be archived for comparison to exposed coupons.

A characteristic assembly is shown in Figure 7. The assembly structure will be made of aluminum to minimize the assembly weight as well as galvanic interactions with the coupons. The coupons would be attached individually with non-reactive polymeric washers to prevent metal to metal contacts. At this time attachment mechanisms are

being investigated so that coupons can be removed one at a time. Both vertical and horizontal positioning of the assembly may be used depending on location of assembly in the basin.

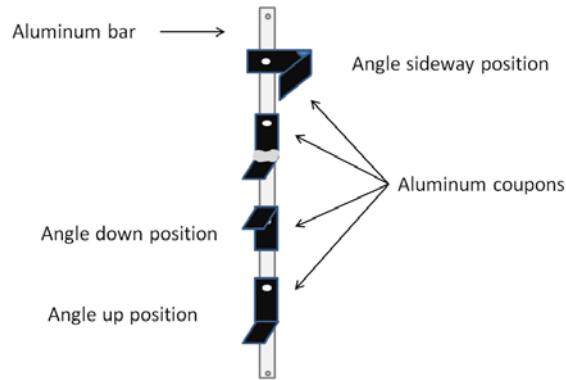


Figure 7 Schematic of proposed EBS coupon assembly for exposure in L Basin

Different coupon orientations will also be used to have both vertical and horizontally oriented surfaces, including weld surfaces. The exposure orientations of the coupons are summarized in Table 5 and shown schematically in Figure 7.

Table 5 New EBS Coupon Positions on Basin Assembly

Sample Type	Position
Tubing – non-welded	Angle up, angle down, angle sideways
Tubing – welded	Angle up, angle, down, angle sideways
Cross brace – non-welded	Angle up, angle down
Cross brace – welded	Angle up, angle down

3.5 Coupon Location

The initial coupon assemblies were placed around L Basin and the installation was performed over several years. Table 6 shows the location and installation dates and details of the assemblies. The location of the new EBS assemblies will be chosen based in part on the location of the current assemblies. The new assemblies will be located in areas where the basin water has nominal flow (closer to surface) and stagnant flow (between racks). These locations will be chosen in conjunction with both SFP operations and engineering personnel. The number of locations for the new EBS assemblies as well as the total number of coupons will determine the number of assemblies fabricated.

The initial assemblies are located in both the vertical tube storage (VTS) and horizontal tube storage (HTS). One ray gun, four junior ray guns, and four strings from chime racks remain in L Basin. The junior ray guns are approximately five feet below the water surface and the ray gun is at approximately twice the depth of the junior ray gun. A map is shown in Figure 8 of the remaining assembly locations. Small pin coupons for investigating microbiological influenced corrosion and microbial activity are located on

the northern ends of Lanes 15 and 22 and removed on annual basis for biofilm analysis [11].

Table 6 Coupon Assembly Location and Installation Details

Installation Date	Assembly	Location	Detail
7/10/95	Chime	HTS (41)	TB side, 1 string
		HTS (43)	MB side 3 strings
1/11/96	Ray gun	VTS Lane 1	10 feet deep
5/3/99	Junior	VTS Lane 18	5 feet deep
		VTS Lane 24	5 feet deep
		HTS (1)	Rust staining from steel chain
		HTS (68)	Next to empty chime rack
1/14/04	MIC	VTS Lane 15	SRNL EBS responsible
		VTS Lane 22	SRNL EBS responsible

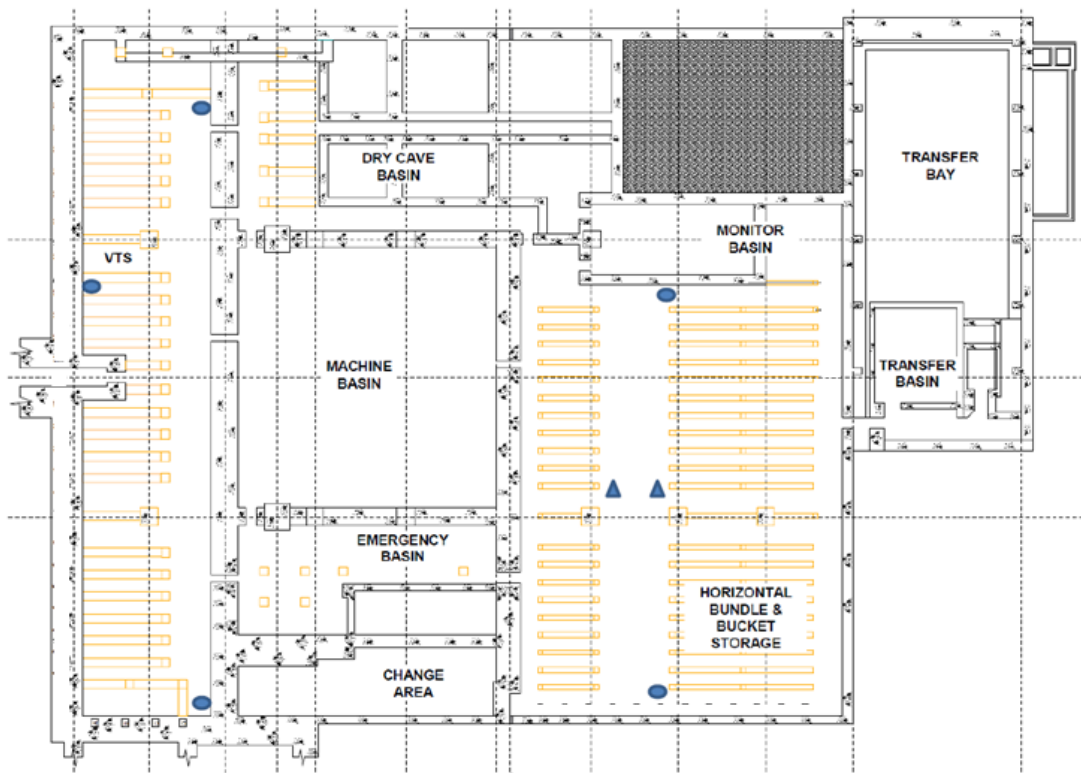


Figure 8 L Basin map showing location of remaining coupon assemblies (ovals – surveillance coupons, triangles – furniture rack coupons)

3.6 Coupon Withdrawal and Evaluation Schedule

A coupon assembly was removed from L Basin annually starting in 1997 (some years had multiple assemblies removed). Maintaining this frequency the coupon assemblies would have been depleted prior to basin closure. The frequency of removal was changed to

extend the existing coupon inventory until approximately 2020 [6]. The frequency was biennially, alternating between chimes and ray guns. The coupon assemblies would cover the time period of authorized receipt of foreign fuel until 2019. The coupon retrieval period was adjusted again in 2012 to every other year to cover a longer period of the expected life of the basin (2035). The frequency withdrawal was extended again during 2013 from two to three years [7] which changes the last removal from 2029 to 2038. The L Basin is currently planned to be used as a SNF storage facility with domestic receipts of fuel through the year 2035. The new EBS assemblies will provide for further extension of corrosion surveillance program if needed to cover further extension of the basin lifetime as a storage facility.

The technical bases for enabling an extended withdrawal schedule were the results from the corrosion surveillance program. The maintenance of good water quality has resulted in very low corrosion pitting rates and near-negligible general corrosion rates that would not adversely impact the safe storage of the fuel [8].

Coupons may also be removed for a special corrosion evaluation per the direction of SFP Engineering such as with the occurrence of an upset condition in the basin.

4.0 Coupon Evaluation Protocol

Coupons are removed from the basin, packaged and transported to the Savannah River National Laboratory (SRNL) for corrosion evaluation. The physical removal is performed by L Basin operation personnel as directed by SFP Engineering and is a schedule activity. The date of removal is selected in accordance with the withdrawal frequency plan [7]. SFP Engineering arranges for the transport of the coupons to SRNL.

4.1 Coupon Removal

The removal of the coupons from L Basin is managed by the SFP Engineering organization and coordinated with Materials Science & Technology of SRNL. The coupons are removed from the basin and placed into packaging that is agreed on between SRNL and SFP Engineering. Coupons should be either dried to stop the corrosion process or stored in basin water to maintain a wetted surface for analysis.

The previous handling involved placing the coupons in a polyethylene bag in a “drip-dry” only condition. That is, no drying protocol was prescribed or used. This practice causes uncertainty as to whether corrosion processes are mitigated or stopped which is important since up to several months can pass before specimens are received at SRNL and retrieved for corrosion evaluation.

The preferred storage method is in basin water so that surface oxides are maintained close to their exposure condition in the basin. With new SRNL microscopy capabilities, imaging through the oxide to the underlying metal surface is feasible so oxide thickness measurements can be made. Drying of the aluminum oxide will lead to changes in thickness.

4.2 Corrosion Evaluation

The corrosion evaluation for the basin surveillance coupons is a multi-step process for documenting and characterizing the observed corrosion. Upon receipt at SRNL, the coupon alloy and numbers are verified against the accompanying paperwork. Coupons are handled as contaminated materials until cleared by the SRNL Radiological Control Organization.

The coupons are photographed both in the as-received condition and after cleaning. The as-received condition includes if they are sent as an assembly. Each coupon is photographed individually on both sides using a digital camera. These photographs are stored on the winsan1 server in the following folder Mat_tech/L Basin Corrosion, along with other archived pictures and documents.

The coupons are weighed both prior to and after cleaning to assess the degree of oxidation as well as a general corrosion rate. A calculated corrosion rate requires that initial weights prior to coupon exposure are available. These data are recorded in Excel spreadsheets that are stored on the winsan1 server in the following folder Mat_tech/L Basin Corrosion.

The cleaning procedure involves soaking the coupons in 16M nitric acid (stock bottle strength of reagent grade nitric acid) for 5 or 10 minute intervals and rinsing with demineralized or distilled water. This aggressive acid solution is necessary for removing the tenacious aluminum oxide. After cleaning, the coupons are weighed on an analytical balance to track the weight loss and assess the degree of attack. Coupons are inspected for oxide retention and assessed to determine if sufficient oxide has been removed to make pit measurements. Additional nitric acid soaks are used as required if residual oxide interferes with the pitting characterization. Ultrasonic cleaning may also be used to expedite the cleaning process. During cleaning, new aluminum coupons used as control specimens are also cleaned to monitor the degree of attack. Weight loss due to the nitric acid cleaning is small (<0.0005 g versus 5-6 g for a coupon), which agrees with published results [9].

Historically, the cleaned coupons have been examined to characterize the corrosion morphology using a standard white light microscope (SWM, a Nikon MM-400 measuring microscope with E-MAX series software). Pit measurements are performed per ASTM G-46 [10] with the primary emphasis on the deepest pit, i.e. the closest to penetrating. This part of the protocol involves a lot of time for a laboratory technician to choose the pits and make the measurements, which requires adjusting the focal plane between the top and bottom of the pit. Typically, the aluminum surveillance coupons have numerous small pits (<0.001 in).

The reported values for pit characterization include the maximum pit depth, the average of the ten deepest pits, the count or density of pits and pit diameters. If the average is performed with less than 10 pits, data records are annotated. Pit diameters are measured for 10 pits of approximately the same size. Large pits, i.e. greater than twice the diameter of the nominal size are also recorded. Statistical analysis is used to assist in identifying

the areas for pit measurements. Pit growth rates are calculated based on the pit depth measurements as well as the total exposure time.

For future evaluations, a new laser confocal microscope (LCM) will be used in this evaluation, jointly with the SWM initially. During the next surveillance coupon analysis, this joint work will allow the new protocol for coupon analysis with the LCM to be tested and verified. The LCM has the capability of scanning at the external surface of the oxide-covered surveillance coupons and at the aluminum/oxide interface. If this capability proves effective, cleaning of the coupons may not be required during subsequent analysis. The type of data (optical and height) taken with a LCM is shown in Figure 9 for a cleaned 2010 surveillance coupon, which had previously been analyzed with the SWM [11]. This particular coupon had no significant pits (> 0.002 in).

For the next set of surveillance coupons removed from the basin, the protocol consists of scanning the entire uncleaned coupons, followed by scanning of cleaned coupons. Using this protocol will also allow the impact of cleaning on the measured corrosion parameters, such as pit depths to be measured and documented. Literature data, which have agreed with experimental observations in the SRNL laboratory on new unexposed coupons, have shown that pitting is not initiated with nitric acid cleaning solutions [9].

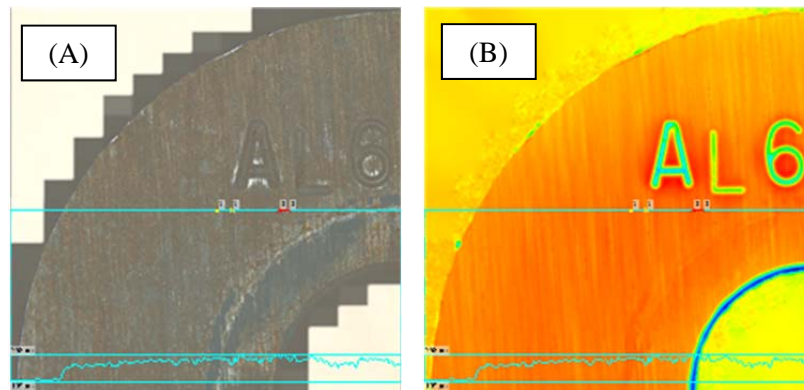


Figure 9 Laser confocal microscope images of the 2010 surveillance coupon Al6061 #044: (A) laser and optical scan, which highlights surface topography and (B) height scan, which highlights differences in elevation across the coupon (data on bottom of photograph shows the height profile for the line scan)

Pit measurement protocol for the SWM and LCM differ. For the SWM, the pit depth is determined by the z-direction translation between focusing on the top and bottom of each pit. Figure 10 (A) and (B) show this change in focus for a pitted aluminum coupon. If oxide remains inside the pit, focusing on the bottom is not possible. The LCM performs a scan of the whole area and automatically focuses at numerous planes. These data are used to generate a height scan from which pit depths can be determined. Figure 10 (C) and (D) show the laser and optical as well as the height scan for a pitted aluminum coupon.

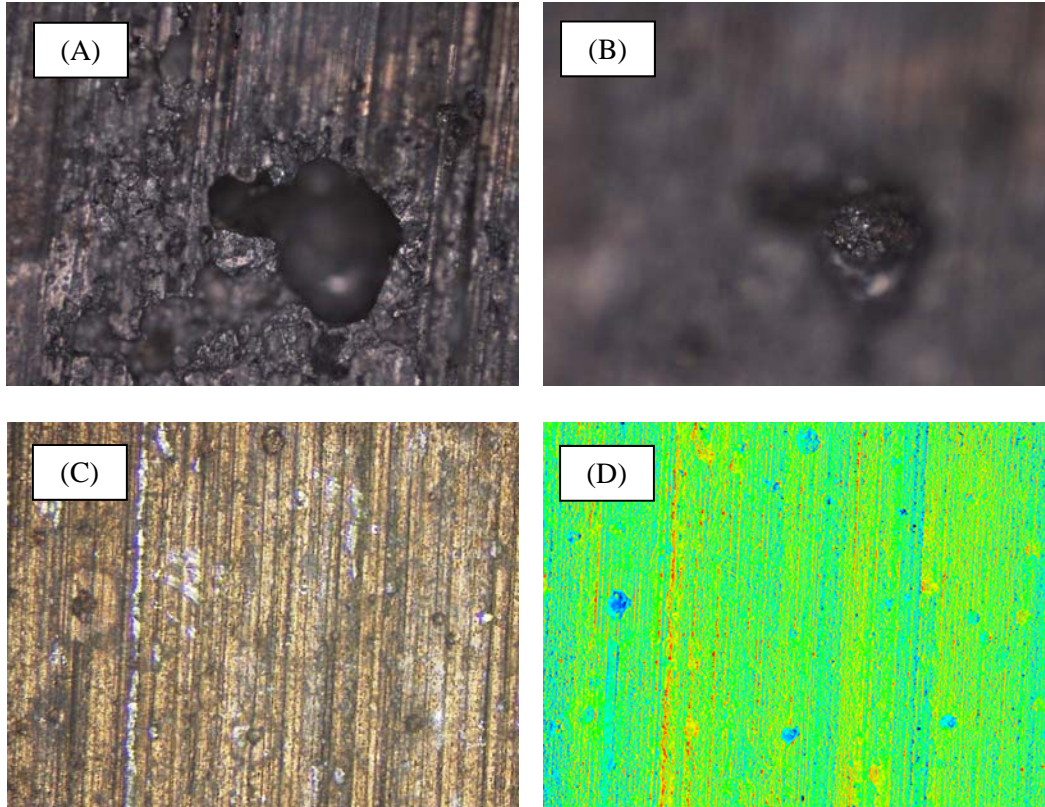


Figure 10 Pictures of pits taken during pit depth measurements using both a standard white light microscope (A – focus on top of pit, B – focus on bottom of pit, 100x) and a laser confocal microscope (C – focused optical image, D – laser height scan showing pit depths, 100x)

Higher magnification pictures on either a stereomicroscope or laser confocal microscope may also be taken for specific areas of corrosion as determined by the Principal Investigator (PI). Additional examinations may be performed with the scanning electron microscope to further assess the corrosion and the state of the aluminum oxide.

5.0 Reporting and Documentation

All data are documented in SRNL controlled laboratory notebooks and stored electronically on the winsan1 server in the Mat_tech/L Basin Corrosion folder. Data include photographs, spreadsheets with pit measurements as well as any corrosion rate calculations.

A technical report is generated to distribute the results of the corrosion surveillance coupon evaluation. This report includes a compilation of the data: coupon photographs, pit depth measurement summary variables, and calculated pit growth rates. Recommendations may be included to adjust L Basin water chemistry if sufficient data is available. These reports are also stored electronically on the winsan1 server in the Mat_tech/L Basin Corrosion folder.

Coupon photographs include before and after cleaning as well as micrographs of degradation at higher magnification. Comparisons with past coupon surveillance photographs are not included unless significant differences are found by the PI.

Coupon weights are reported as percentage weight (individual coupons) and normalized weight (galvanic coupons) gains if original weights are available. Pit depths and growth rates (average and maximum) are reported in tabular or graphical form on individual, creviced, galvanic, and furniture rack coupons. Comparisons with previous data are included to assess changes in corrosion over time.

Results on evidence of corrosion attack from the microbial monitoring program are part of the basin surveillance reporting [12].

6.0 Special Inspection

A special inspection of the OS storage canister [4] provided a direct characterization for a condition assessment of a storage system material. This inspection provides information important to a corrosion evaluation of the L Basin conditions. In this case, the OS cans were in L Basin for approximately 10 years, and no significant corrosion was detected with either remote visual examination or ultrasonic test methods.

Additionally, activities are in progress to perform inspection of the fuel using remote underwater visual inspection. The comparison of fuel inspection results to corrosion surveillance results would be reported in the corrosion surveillance report.

7.0 References

1. 2009 IAEA Nuclear Energy Series No. NP-T-5.2, "Good Practices for Water Quality Management in Research Reactors and Spent Fuel Storage Facilities," International Atomic Energy Agency, Division of Nuclear Fuel Cycle and Waste Technology, Research Reactor Section, 2011
2. International Atomic Energy Agency, "Corrosion of Research Reactor Aluminum Clad Spent Fuel in Water," IAEA-TRS-418, IAEA, Vienna, 2003
3. S. D. Burke and J. P. Howell, "The Impact of Prolonged Wet Storage of DOE Reactor Irradiated Nuclear Materials at the Savannah River Site," Proceedings of the Topical Meeting on DOE Spent Nuclear Fuel – Challenges and Initiatives, Salt Lake City, UT, December 1994, pp 76-82
4. Nondestructive Examination Condition Report, Job # L20130017, Work Request # 1211213, October 25, 2012
5. M. R. Louthan, Jr., R. L. Sindelar, N. C. Iyer, and H. B. Peacock Jr., "Corrosion of Aluminum Clad Fuel and Target Elements: The Importance of Oxide Films and Irradiation History, American Nuclear Society Proceedings of the Embedded Topical Meeting on DOE Spent Nuclear Fuel and Fissile Material Management, Reno, Nevada, (June 16-20, 1996), pp 57-61
6. P. R. Vormelker and R. W. Deible, "L-Basin Corrosion Surveillance Program Plan," SRNL-TR-2009-00140, Revision 1, September 2009

7. J. I. Mickalonis, "Extension of L-Basin Corrosion Coupon Removal Frequency," SRNL-TR-2013-00131, Rev. 0, June 2013
8. R.W. Deible, "L Basin Water Chemistry Control Program," WSRC-TR-97-0239, Revision 5, June 2010
9. K. S. Ferrer and R. G. Kelly, "Comparison of Methods for Removal of Corrosion Products from AA2024-T3," Corrosion, Vol. 57 (2), pp 110-117
10. ASTM G46 – 94 (2013), "Standard Guide for Examination and Evaluation of Pitting Corrosion," ASTM International, West Conshohocken, PA, 2013
11. J. I. Mickalonis and T. H. Murphy, "L Basin Corrosion Surveillance Program – Evaluation of 2010 Surveillance Coupons," SRNL-2014-TR-00004, Revision 0, in draft (expected issue December 2014)
12. C. J. Berry, "Summary of the Microbial Condition of the L-Disassembly Basin Spent Fuel Storage Facility Through July FY08," SRNL-EST-2008-00028, September 2008